Green conversion of red snapper fish scale-derived carbon dots and its absorption properties for solar thermal desalination

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ABSTRACT

Fisheries wastes have been used as precursors for the synthesis of carbon dots (CDs). These wastes are often converted using hydrothermal methods which require high temperature and pressure, leading to high production costs, especially for large-scale production. This study aims to innovate a low-cost synthesis method with the potential for large-scale production. Green conversion of CDs from red snapper fish scale waste was carried out using a combination of immersion method and ultrasonic wave treatment. The results showed that the products had an absorption peak at a wavelength of 404 nm and an energy bandgap of 2.7 eV. Excitation at 404 nm was associated with non-bonding n orbital to antibonding orbital π^* electronic transitions due to the presence of free electron pairs and related with carbon-nitrogen (C-N) bonds. The 2.7 eV energy bandgap was associated with the state of amine groups containing free electron pairs located on the surface of CDs as well as blue light emission at a wavelength of 460 nm when CDs were illuminated with ultraviolet light. Red snapper fish scale-derived CDs showed fluorescence characteristics and the presence of nitrogen elements, making them potential photothermal materials for solar-powered seawater desalination processes.

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1. INTRODUCTION

Carbon dots (CDs) are materials with great potential due to their manifold applications, particularly in heavy metal monitoring (ferric ion) [1], photovoltaic materials [2], biomedical [3], and biosensors [4]. The remarkable fluorescence capability of CDs is closely associated with the quantum effects arising from the size difference, which is an important optical characteristic for both absorption and emission [5]. Fluorescence is a form of photoluminescence where CDs or carbon quantum dots, emit different colors of light with lower energy compared to the absorbed light, depending on their bandgap size [6].

Fish scales are one of the waste products obtained from fishing industry activities, seafood restaurants, and local fish markets. These materials have been reported to have a high potential as a carbon source for the production of CDs [7]. Furthermore, fish scales account for 2-4% of approximately 20-80% of the total waste generated during fish processing [8], [9]. As fish production continues to increase, the amount of fish scale produced is also expected to rise. In 2017, fishery production in Indonesia reached 23.19 million tons, thereby significantly increasing the volume of waste [10]. This increased waste production can cause environmental pollution and negative effects on human health without proper management and optimal use [11], [12]. Fish scales are biocomposite materials that can be extracted into organic components, such as chitin, lecithin, guanine, and collagen fibers, as well as inorganic components, including hydroxyapatite crystals and calcium carbonate [7], [13], [14]. Chitin and collagen are mineral-rich substances containing carbon, oxygen, hydrogen, and nitrogen, making them ideal for use as raw materials for CDs [7], [13], [15].

The synthesis of CDs from fish scales is typically achieved using a bottom-up method. In this process, the raw materials are subjected to hydrothermal synthesis, involving high temperatures of approximately 180-200 °C. This method also requires a long synthesis time of approximately 7-20 hours, followed by a dialysis technique for purification [16]. However, the application of high pressure and temperature in the hydrothermal method poses potential threats and is limited to small-scale production [1]. An alternative technique that offers a shorter duration is the microwave-assisted method combined with the sonication process [17]. Although this technique has the advantage of shorter synthesis time, it consumes high electrical power, uses nitrogen gas, and still yields products on a small scale. The development of an environmentally friendly, safe, cost-effective, and producible method for large-scale synthesis of CDs has promising economic impacts. The synthesis of carbon nano-particles (CNP) using environmentally friendly technology had been successfully performed by immersing *Ctenopharyngodon idella* fish scales in ultrapure water at room temperature for 7 days followed by centrifugation claimed as a new method [1]. However, the effect of soaking time on the optical properties of the resulting CNP is not yet clear. Apart from that, the CNP produced has not been fully claimed as CDs.

Previous research recommended the use of ultrasonic waves for the synthesis of CDs either alone [18] in combination with microwave methods [17] or in combination with hydrothermal methods [16]. Ultrasonic waves can affect the process of converting biomass into CDs through acoustic cavitation [18]. Based on these facts, the novelty proposed in our research is a combination of immersion techniques and the application of ultrasonic waves to synthesize CDs. Implementation of ultrasonic methods in CDs production is related to ultrasonic frequency, ultrasonic power, and sonication time [19]. Some researchers used an ultrasonic frequency of around 20-80 kHz, ultrasonic power ranging from 20-320 W, and sonication time varying between 20 minutes to 3 hours [17], [19], [20]. The frequency of ultrasonic waves influenced cavitation processes, while ultrasonic power and sonication time determined the size and shape of CDs [19]. Sonification time for the combination of hydrothermal and ultrasonic methods is 20 minutes [16], and for the combination of microwave and ultrasonic methods it is 30 minutes [17]. The sonication time required for CDs synthesis using a combination of immersion techniques and ultrasonic treatment is not yet clear so it still requires study. Therefore, this study aims to synthesize CDs from fish scale waste using a combination of immersion and ultrasonic wave treatment methods. The study proposed a sustainable method to convert fish scale biomass as CDs with photothermal properties, intended for seawater desalination applications.

2. RESEARCH METHOD

2.1. Materials

The materials used in this study included the red snapper fish scale in Figure 1 as a carbon precursor and distilled water as the solvent. The red snapper fish (*Lutjanus sp.*) scale was used due to its high chitin yield and collagen content [7]. The raw fish scale was commonly found in local markets in North Sulawesi as waste [15]. Raw fish scales in Figure 1(a) were washed and then dried for 2 days in direct sunlight. After that, they were ground with a Miller Fomac FCT-Z100 machine with a rotation speed of 28,000 rpm to obtain fish scale powder with a size of 75 µm (Figure 1(b)).

(a) (b)

Figure 1. Red snapper fish scales (a) raw scale and (b) powder of fish scale

2.2. Research procedure

A total of 5 grams of red snapper fish scale powder was immersed in 50 mL of distilled water and stirred with a magnetic stirrer for 10 minutes at room temperature [1]. These similar samples were made as 6 namely A0, A1, B0, B1, C0, and C1 samples. The immersed fish scale samples were stored at room temperature. C0 and C1 samples were immersed for 5 days, B0 and B1 samples were immersed for 9 days, and A0 and A1 samples were immersed for 7 days [1]. Subsequently, each immersion solution was centrifuged for 15 minutes at a speed of 3,500 rpm, and the supernatant containing CDs was filtered using Whatman No. 42 paper [16], [21]. A1, B1, and C1 samples were subjected to ultrasonic wave treatment with a frequency of 42 kHz and sonication time of 30 minutes using Krisbow ultrasonic cleaner 50 W, and the other samples served as the control [20], [22]. Next, the 6 samples studied the light absorption properties were characterized using ultraviolet-visible (UV-VIS) spectrophotometry of the Shimadzu UV-1800 series with data recording at a wavelength of 380-700 nm at the Pharmaceutical Laboratory, Faculty of Mathematics and Natural Sciences, Sam Ratulangi University [7], [13]. Based on the results of UV-VIS spectrophotometry, the immersion solution immersed for 7 days (A1 sample) was prepared again using the same method to multiplicate three samples for microwave treatment, and they were treated for 15 minutes (A11 sample), 30 minutes (A12 sample), and 45 minutes (A13 sample) [16], [17], [22]. Also, the optical characteristics, specifically absorption properties of CDs in A11, A12, and A13 samples were characterized using a UV-VIS spectrophotometer at the same place as for previous samples. The fluorescence behavior and Tyndall effect of CDs in samples were assessed using an ultraviolet lamp and a red laser pen [1]. Furthermore, the UV-VIS characterization results were presented in a graph depicting absorbance intensity against wavelength and energy bandgap [16]. The energy bandgap was calculated using the Tauc method [23]. The stages conducted in this study are schematically illustrated in Figure 2.

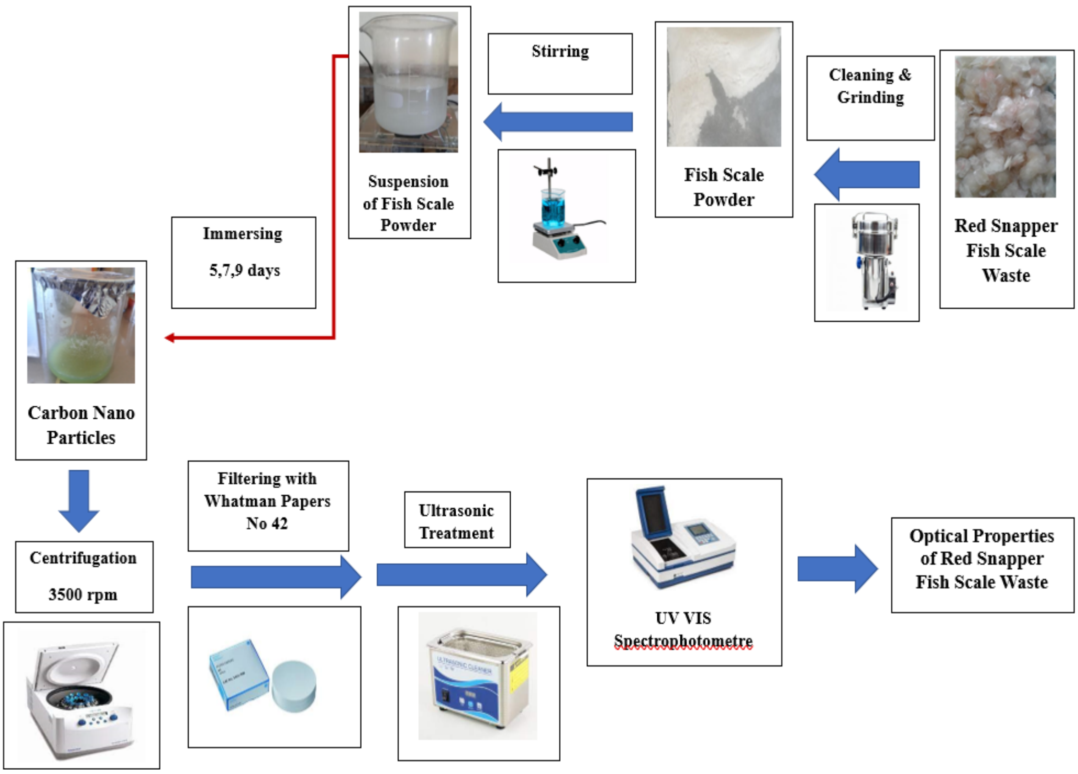

Figure 2. Scheme of the research procedure

3. RESULTS AND DISCUSSION

3.1. Absorption properties

The effect of immersion is a crucial factor that must be investigated as it relates to two key aspects, namely the chemical changes of red snapper fish scale powder mixture in distilled water and the physical characteristics, including light absorption ability, and energy bandgap size within the material. Figure 3 presents the immersion process and results of red snapper fish scale powder for 5, 7, and 9 days (Figures 3(a) to (c)). During the process, structural changes were observed, which led to the dehydration of molecules in fish scales. This process caused a color change in the suspension due to the physical shedding of several components of the samples, such as collagen, hydroxyapatite, and lipids to form nano-scale fluorescent particles that were soluble in water [1].

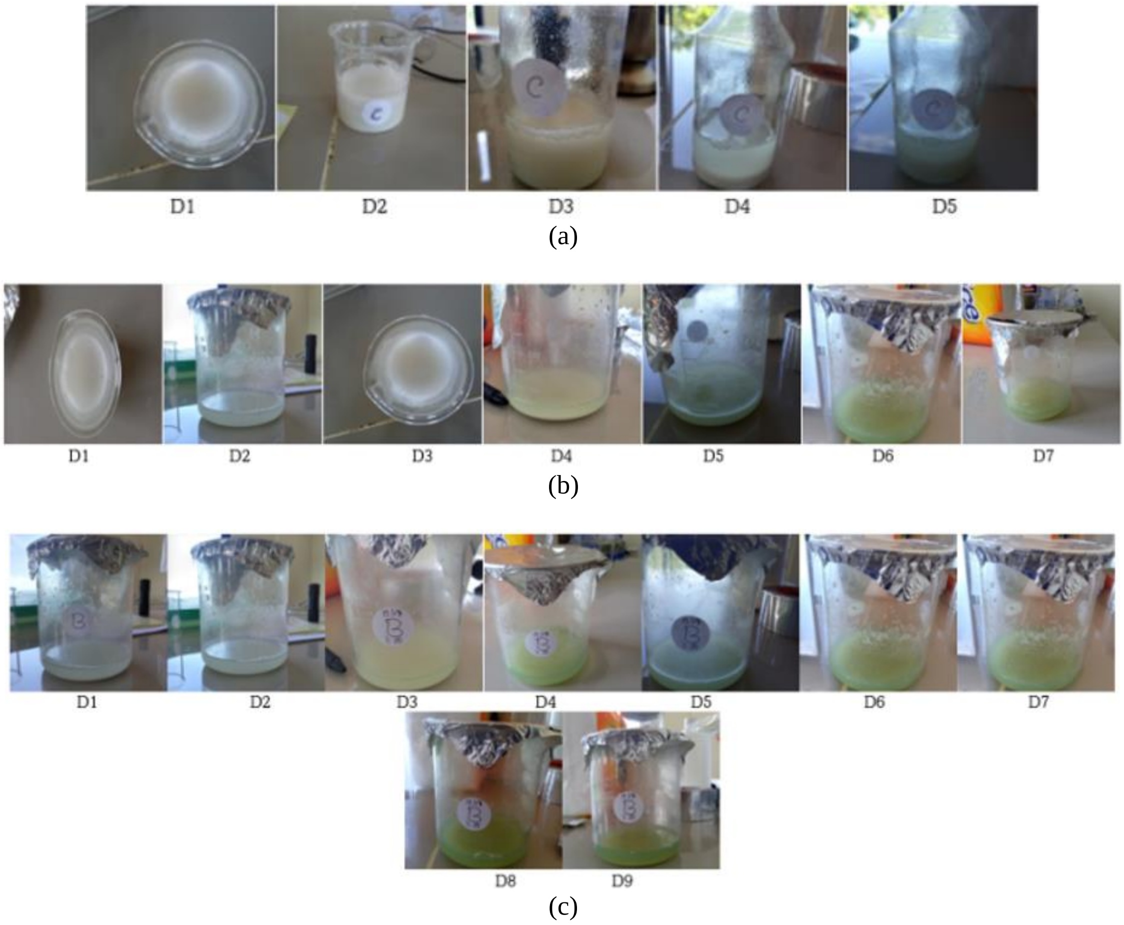

Figure 3. Immersion with time variations at (a) 5, (b) 6, and (c) 7 days

The effect of immersion time on UV-VIS light absorbance revealed that the 9 day-immersion gave a high absorbance, while the 5 day-immersion showed significantly low levels (Figure 4). The 7-day treatment exhibited a spectral absorbance peak at a wavelength of approximately 404 nm, indicating electron excitation from the highest occupied molecular orbital (HOMO) to the lowest unoccupied molecular orbital (LUMO) within the material [21]. Meanwhile, the 9-day treatment did not show a spectral absorbance peak, but there was an increase in intensity, making the 7-day immersion results more significant. Beyond the 7-day treatment, the grinding aspect became less dominant, and decomposition was believed to emerge, accompanied by changes in the odor of suspension. According to Yao *et al*. [1] the treatment produced CNP with absorbance of approximately 270 nm. The occurrence of excitation within the wavelength range of 250-405 nm led to the emission of light at 460 nm or blue light, and the UV-VIS spectrum showed two absorbance peaks at 270 and 405 nm [1]. Ultraviolet light absorbed in the range of 270-405 nm was emitted back at wavelengths of 335 and 460 nm [1]. The emission-excitation behavior could be attributed to the presence of carbonyl and amide chromophores in the material, indicating its fluorescent nature [1], [6].

The influence of ultrasonic wave treatment was applied to the supernatant obtained from the immersion, followed by semi-quantitative analysis using a UV-VIS spectrophotometer, as shown in Figure 5. Ultrasonic waves exerted an effect on carbon material, facilitating its conversion into CDs [24], [25]. Figure 5 illustrates that the combination of the immersion method with variations in ultrasonic wave treatment time at a frequency of 42 kHz led to changes in absorbance intensity. Furthermore, ultrasonic waves generated a mechanical effect, which was similar to the pyrolysis effect for thermal treatment, with the ability to speed up the reaction, mass transfer, and increase the penetration of the solvent into the cells of red snapper fish scale material [1]. Material subjected to 30-minute ultrasonic wave treatment exhibited an absorbance peak at 404 nm and a larger range of intensity absorption increase. The UV-VIS spectral pattern indicated that the 30-minute ultrasonic wave treatment yielded the most effective acoustic cavitation, leading to the highest increase in absorbance at a wavelength of 450-400 nm compared to the 15 and 45-minute treatments (Figure 5). The sonochemical and sonomechanical effects experienced for 15 minutes did not lead to structural changes in the material resembling CDs, as indicated by the lowest absorption level and the absence of clear absorption peaks. However, the ultrasonic wave treatment for 45 minutes caused both sonomechanical and sonochemical effects. This presumably deformed the structure achieved during the 30-minute treatment, thereby preventing the quantum phenomenon at 404 nm.

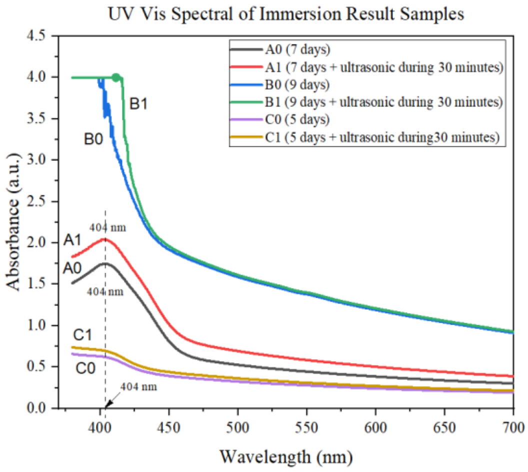

Figure 4. Absorbance spectral versus immersion time

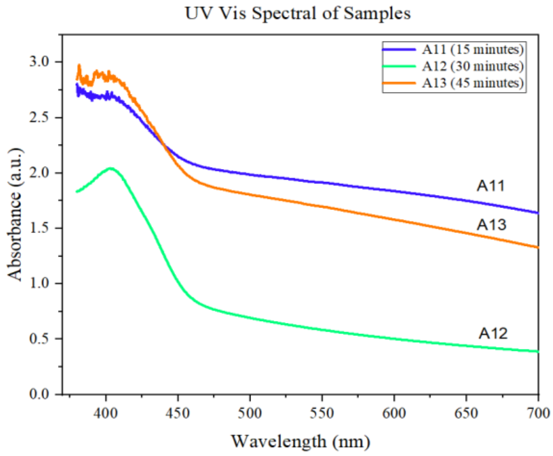

Figure 5. Ultrasonic effect on absorption properties

3.2. Bandgap energy

UV-VIS analysis of samples immersed for 7 days with and without a 30-minute ultrasonic treatment using the Tauc method showed the energy bandgap of CDs, as shown in Figure 6. The energy bandgap of samples without treatment showed a value of 2.72 eV. Furthermore, the application of ultrasonic wave treatment for 30 minutes after 7 days of immersion increased the absorbance intensity and shifted the energy bandgap from 2.72 to 2.7 eV. The combination of the 7-day immersion and 30-minute ultrasonic treatment at a frequency of 42 kHz was the most optimal composition, leading to a supernatant with distinct characteristics of absorption or UV-VIS excitation peaks at a wavelength of 404 nm and an energy bandgap of 2.7 eV. These findings were consistent with previous studies, where the energy gap of CD materials was within the range of 1.5 to 3.5 eV [6], [26].

Figure 7 showed that the 15 and 45 minute ultrasonic wave treatments reduced the energy bandgap to 2.48 and 2.56 eV, respectively. These variations were related to the increase in supernatant temperature due to the sonomechanical and sonochimia effects. Higher reaction temperatures tended to fully carbonize the functional groups on the surface of the amorphous carbon region, leading to larger carbon core sizes, and a shift of photoluminescence towards longer wavelengths or the near-infrared region [27]. Zhang *et al*. [28] showed that the degree of carbonization increased at reaction temperatures between 120-220 °C, while the quantum yield, UV-VIS absorption, and photoluminescence spectra of N-doped CDs initially increased and then decreased with increasing temperature. The supernatant from red snapper fish scales treated with ultrasonic waves showed increased UV-VIS absorption, but then decreased, as observed in Figure 5. The prolonged ultrasonic wave treatment effect was similar to the impact of temperature variation in hydrothermal synthesis. Furthermore, the adjustment and optimization of the ultrasonic wave frequency presented an opportunity to modify the optical properties of CDs. This modification led to the exhibition of photoluminescence properties that shifted towards the near-infrared region. Based on bandgap energy of 2.7 eV, the synthesized CDs were associated with blue light emission with a wavelength of 459.7 nm. A value of 2.7 eV was optically characteristic or related to photoluminescence properties associated with electronic transitions in the amino functional group (-NH_2) located on the surface of the carbon core. Therefore, the absorption peak at a wavelength of 404 nm in the UV-VIS spectrum of CDs based on red snapper fish scales could be attributed to the n–π* electronic transition of the C-N bond [6], [29], [30].

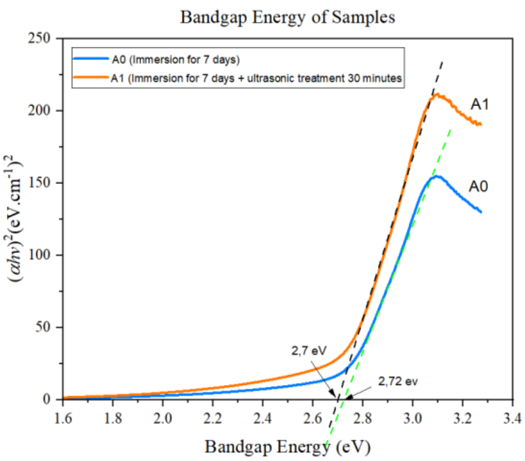

Figure 6. Bandgap energy versus immersion time

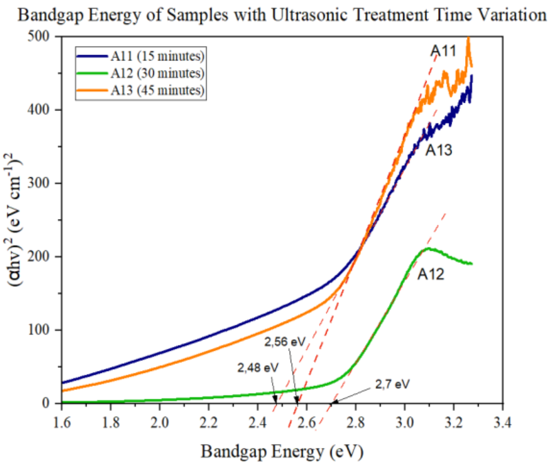

Figure 7. Bandgap energy versus time of ultrasonic treatment

3.3. Fluorescence properties

The liquid material produced through the immersion process exhibited characteristics of CDs, as found by previous studies, such as the ability of CDs to absorb higher-energy light and emit light with longer wavelengths as shown in Figure 8. The liquid material emitted blue light when illuminated with ultraviolet light as seen in Figure 8(a) and appeared light brown-yellow when illuminated with sunlight in the room as shown in Figure 8(b). Figure 9 showed the presence of the Tyndall effect, confirming that the supernatant contained colloid CDs. Based on the results, the red laser light, with a wavelength of 630-650 nm, interacted with the particles in the colloid system. Subsequently, the particles scattered or redirected the laser light in all directions. According to a previous study, the Tyndall effect occurred in colloid dispersion when the nano-particle size ranged from 1 to 1,000 nm, as observed in carbon quantum dots synthesized by Cruz *et al*. [31] with an average particle diameter of 4.1±1.1 nm. The nano-size was also confirmed by the quantum phenomenon, where the supernatant emitted blue light when illuminated with ultraviolet light, as shown in Figure 8. This process was known as photoluminescence, where spontaneous emission occurred after photoexcitation. The light emission was temporary and stopped when the UV light was turned off. This condition was known as fluorescence, indicating that the synthesized material with a combination of both methods behaves as CDs particles, and this was consistent with previous studies.

(a) (b)

Figure 8. CDs solution under (a) sunlight irradiated by UV lamp and (b) sunlight

Figure 9. Tyndall effect on CDs

3.4. Implication of the results in seawater desalination technology

The CDs material synthesized from red snapper fish scales in this study contained amino functional groups on its surface structure. The presence of these functional groups was relevant for use as photothermal materials in solar cell desalination because nitrogen played a role in enhancing the photoluminescence properties of CDs [25]. Furthermore, hydroxyl groups could improve the efficiency of converting solar energy into heat [32]. The presence of nitrogen elements in fish scales provided an opportunity for them to be converted to CDs during the synthesis process. Nitrogen doping occurred automatically in the structure of CDs, making them suitable as photothermal materials in the near-infrared region [33]. The fluorescence properties of fish scale-based CDs could potentially be combined with organic or inorganic materials to enhance the efficiency of converting solar energy into thermal energy [34]. CDs possessed hydrophilic properties, which were crucial for their application as photothermal materials due to the aromatic structure with C=C groups [21]. These properties allowed salt water to penetrate the photothermal material area for heating and evaporation. These materials exhibited high photothermal conversion efficiency, wide light absorption spectrum, low thermal conductivity, low thermal dissipation, and high localized heat generation [35]. Furthermore, they had hydroxyl (-OH) and carboxyl (-COOH) functional groups that contributed to increasing the efficiency of converting solar radiation into heat [32], [36]. They could also be composited or combined with organic and/or inorganic materials to enhance the evaporation rate. The use of CDs in the desalination of seawater has been reported to hold great potential [32], [37]–[44].

4. CONCLUSION

Synthesis of CDs using immersion techniques combined with ultrasonic waves is a simple, cheap, and environmentally friendly synthesis technique compared to hydrothermal techniques. In conclusion, powder from red snapper fish scales was soaked for 7 days, centrifuged for 15 minutes, and ultrasonicated for 30 minutes, producing a supernatant that exhibited the Tyndall effect. This indicated that the supernatant contained colloid CDs. The appearance of one absorption peak in the UV-VIS spectra at a wavelength of 404 nm was consistent with previous studies, where the supernatant exhibited fluorescence. Experiments with a portable ultraviolet lamp on the supernatant showed that it emitted a blue color associated with the presence of amine groups (-NH_2). However, further studies were needed to analyze the functional groups, allowing the energy band gap associated with the electron transition from these functional groups to the molecular orbital structure HOMO to be determined. In the future, modifications and assemblies of CDs based on fish scale waste with other organic and inorganic materials are important to study in-depth enhancing the seawater evaporation rate.

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