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Studying the biochar modified by $AgNO_3$ from coffee grounds to handle organic pollutants and microorganism in the seafood-processing wastewater

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ABSTRACT

The aim of this study is to use four types of biochar modified $AgNO_3$ from coffee grounds including CF5 (600°C/1.5h), CF6 (600°C/3h), CF7 (700°C/1.5h), and CF8 (700°C/3h) for the treatment of organic contaminants and microorganism in the seafood-processing wastewater. The results showed that the materials had the porous structure and the diameter of the pores corresponded to the crystal plane. The carbon content in the four samples reached over 90%. In addition, four samples observed the presence of Ag with the weight percentage varying from 0.06 to 0.09%. The adsorption capacities for pollutants by the four materials at different times were different. After the adsorption time, the COD value was still from 1.6 to 5.2 times higher than the limited value of the Vietnamese Standard QCVN 11:2015/MONRE - National technical regulation on the effluent of aquatic products processing industry. The three remaining parameters: TSS, ammonium (NH_4^+), and microorganism met the standard limit value requirement of QCVN 11:2015. The TSS contents decreased with the lowest adsorption efficiency in CF5 sample (68.87%) and the highest value in CF8 sample (99.21%); NH_4^+ content decreased respectively from 385.14 mg/L to 19.97; 15.55; 21.5 and 19.16 mg/L (CF5-CF8) with the efficiency over 94% after 2h adsorption. The removal capacity of coliform in CF7 sample is the highest value of 640 MPN/100 mL with the efficiency of 98.61% while the lowest one obtained in CF5 sample with 4.300 MPN/100 mL and the efficiency of 90.65%.

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1. Introduction

In recent years, Vietnamese seafood industry has made great progress with the continuous increase in domestic production and exports which led Vietnam to join the top three leading seafood producing and exporting countries in the world (Food and Agriculture United Nations, 2009). However, the seafood processing industry also discharges a large amount of wastewater into the environment, causing serious pollution to the water source. This wastewater contains high concentration of organic compounds (mainly protein and lipids) derived from sea creatures. The water-quality parameters of untreated seafood processing wastewater are often reported many times higher than the National Technical Regulation on seafood processing wastewater - QCVN 11: 2015/MONRE. Therefore, seafood processing wastewater needs to be treated effectively to reduce levels of pollutants content before releasing into the environment (VASEP, 2016). Therein, biochar is an effective material meeting this requirement.

Biochar is a carbon-rich compound produced by pyrolysis under anaerobic conditions using plant-based raw materials or various organic residuals, especially agricultural products (rice husks, corn cobs, fruit bark, poultry manure, etc.) (Chen et al., 2011; Lehmann, 2007; Pan et al., 2013). Biochar is considered as a potential adsorbent for removing pollutants from wastewater due to its high porosity, large pore size and surface area (500 to 2.500 m^2/g) (Chen et al., 2011; Lehmann, 2007; Pan et al., 2013). Biochar exhibited great adsorption ability and ion exchange capacity leading to storing significant amount of pollutants (Chen et al., 2011; Lehmann, 2007; Pan et al., 2013). In particular, biochar synthesized from coffee grounds has many advantages in terms of the source of initiral raw materials and material characteristics.

In Vietnam, researches on applying biochar to remove organic pollution or pathogenic microorganisms in aquaculture wastewater have not received much attention since most studies focus on soil improvement in agriculture (Doan et al., 2015; Mohammadi et al., 2017; Ngo et al., 2013), remove ammonium (Vu Thi Mai et al., 2016), or remove propoxur insecticide (Nguyen Khoi Nghia et al., 2015), Vietnamese coffee ranked second in the world in terms of producing and exporting volume; the total coffee volume for Vietnam's domestic consumption reached 60,000 tons/year (Truong Hong, 2018), so that the annual amount of coffee grounds in Vietnam is very large, however they are often discarded as wasting carbon-based potential materials. Morever, to make biochar more effective, modified biochar has been studied. According to Cui et al (2015), $AgNO_3$ is considered as a strong antibacterial factor that plays a main role in the treatment of microbial contamination. Therefore, the main aim of this study is to utilize coffee grounds (a kind of agriculture by-products) to synthesize biochar modified by $AgNO_3$ in the slow pyrolysis condition. Finally, the modified biochar will be evaluated basing on the ability to remove organic pollution (COD, TSS, ammonium) and pathogens microorganisms from the seafood-processing wastewater.

2. Materials and methods

2.1. Preparation for the biochar material

Materials used in this study include the coffee grounds collected from a company in Dak Lak province, dried in sunlight for 3 days, moved to the laboratory as the raw material. The seafood-processing wastewater sample was collected at the fish powder production factory in Hai Phong, stored at 4°C and transported to the laboratory.

Silver modified biochar material was synthesized according to the procedure reported by Cui et al., (2015). Coffee grounds were crushed and passed through the 0.5 mm sieve and then heated at different temperatures and time duration: 600°C/1.5h (CF5); 600°C/3h (CF6); 700°C/1.5h (CF7) and 700°C/3h (CF8). Next step, 01 mg of each material of the CF5-CF8 synthesized biochars added into a flask containing 100 mL of deionized water and ultrasonic for 30 minutes in order to form a homogeneous suspension before 3 g of Polyvinylpyrrolidone (PVP) dissolved the solution. Afterwards, 01 mL of $AgNO_3$ solution (0.1N) was added and stired into the solution vigorously at 65°C/6h in the dark condition. Samples were collected by centrifuging with deionized water 3 times and drying at 60°C for 3h.

All chemicals in this study were sourced from Merck supplies.

2.2. Methods for characterization of biochar material

The structural characteristics of the biochar samples were determined by a scanning electron microscope (SEM), transmission electron microscopy (TEM), scanning electron microscope (BET), Fourier-transform infrared spectroscopy (FTIR) and Energy Dispersive X-Ray (EDX) at Institute for Tropical Technology, Vietnam Academy of Science and Technology.

2.3. Methods of determining the adsorption capacity of the biochar

The original wastewater will be analyzed using four parameters as COD, TSS, NH_4^+-N and Coliform bacteria. Four types of biochar materials modified by $AgNO_3$ were evaluated for their adsorption capacity of pollutants according to the procedure reported by Trinh Thi Thu Huong et al., (2015). 4 g of biochar was added in the erlenmeyer flask containing 400 mL of seafood processing wastewater, and then stirred at a speed of 150 rpm for 2h. All the samples were filtered through a filter paper and analyzed using the parameters such as COD, TSS, NH_4^+-N and coliform.

The adsorption capacity of qe (mg/g) at equilibrium was determined by the equation suggested by (Abdelkreem, 2013):

$q_e = (Co-Ce).V/m$

Here: Co - initial concentration (mg/L); Ce - equilibrium concentration (mg/L); V - volume of the dye solution (l); m - mass of the sorbent added (g).

The treatment efficiency (H%) at equilibrium was determined by the equation suggested by (Figueiredo et al., 2017):

H = (Co-Ce)/Co.100 (%)

2.4. Statistical data

The collected data in this study were statistics processed by the following softwares such as GraphPad 6, Origin lab 9 and Excel 2010 with statistical significance of $\rho < 0.05$.

3. Results and discussion

3.1. Structural characterization of synthesized biochar materials

Physical and chemical characteristics of 4 types of $AgNO_3$ modified biochar (CF5-CF8) are given in the Table 1. It shows that the samples produced quite high ash content (> 25%), however the BET surface area of the samples was quite low, ranging from 0.7831 to 1,2807 m^2/g. All four samples recorded the presence of Ag element from 0.06 to 0.09% by weight.

Surface morphology and content of elements of different biochar samples were also determined by SEM and EDX. The results illustrated in Figures 1, 2 and 3 have shown that the surface of the controlled sample and the experimental sample indicated significant differences. The surface structure of four materials (CF5-CF8) noted similarity with the published biochar studies (Cui et al., 2015; Liu et al., 2015). The biochar samples all were observed rough surface, clearly fibrous structures, and the pores space corresponded to the lattice plane (Figure 2) which was different from that in Figure 1A (CF0, un-ashed coffee grounds). It had a solid surface with unknown cellulose fibers. Rough and

Table 1. Physical and chemical characteristics of four types of $AgNO_3$ modified biochar.

Samples	Temperature and Time	Ash Content (%)	BET surface area (m2/g)	Elemental composition of biochar surface			
				C (%)	O (%)	N (%)	Ag (%)
CF0	-	-	-	74.15	18.48	7.30	0.00
CF5	600°C/1,5h	27.57	0.8670	90.78	5.32	3.27	0.06
CF6	600°C/3h	27.00	0.7831	92.32	4.25	3.06	0.06
CF7	700°C/1,5h	26.41	1.1602	91.99	4.83	2.42	0.09
CF8	700°C/3h	25.51	1.2807	93.67	4.15	1.69	0.07

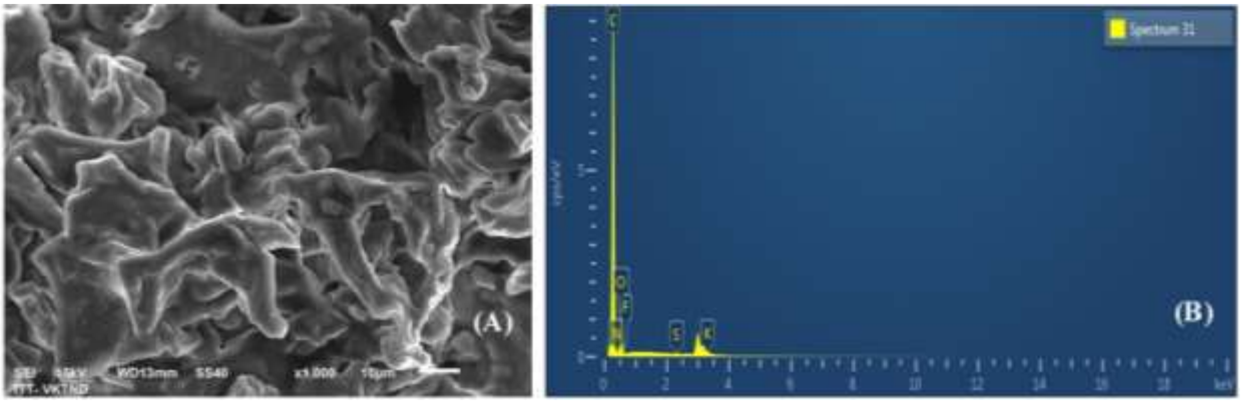


Figure 1. The SEM (A) and EDX (B) images of the control biochar CF0.

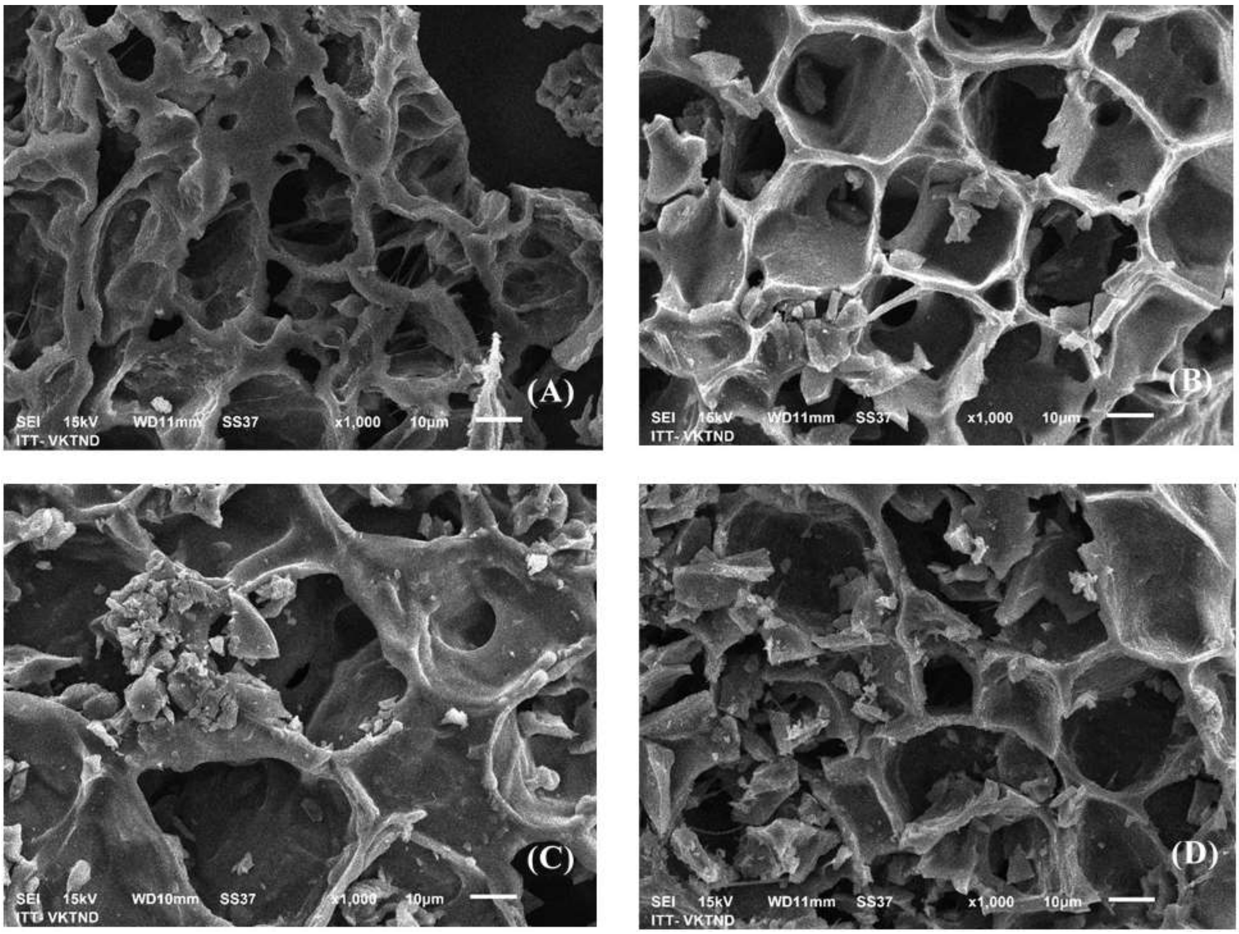


Figure 2. The SEM images of synthesized biochar materials: (A) CF5-600°C/1.5h; (B) CF6-600°C/3h; (C) CF7-700°C/1.5h and (D) CF8-700°C/3h.

patterned surfaces may be present during pyrolysis process or are a part of the properties of the initial raw material (Liu et al., 2015). These properties strongly influence their adsorption properties (Cui et al., 2015; Liu et al., 2015). The results show that the ashesized coffee grounds have better adsorption capacity than the original raw material.

EDX analysis is shown in Figures 1 and 3. It is obvious that the C content in both the raw and ashesized biochar samples was slighly high. However, the biochar samples contained much higher C content, ranging from 90.78% (CF5),

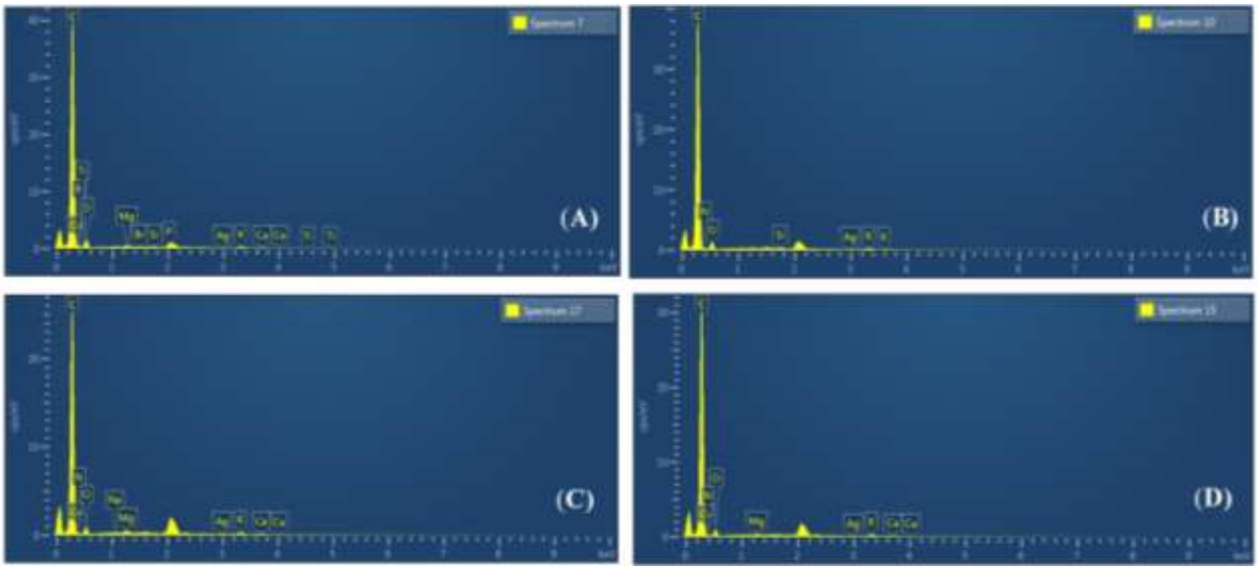
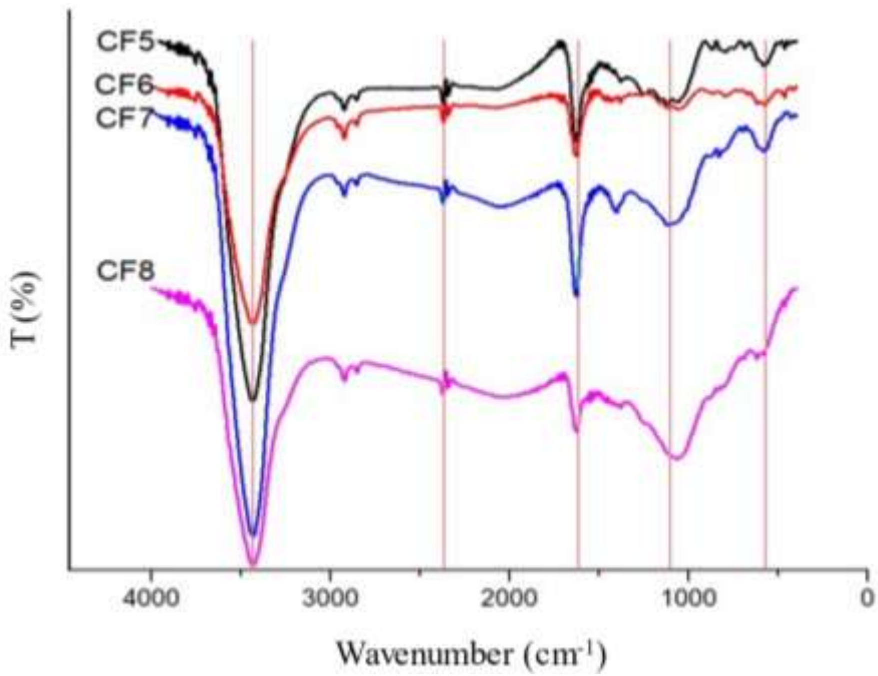


Figure 3. EDX analysis of four synthesized biochars: (A) CF5-600°C/1.5h; (B) CF6-600°C/3h; (C) CF7-700°C/1.5h and (D) CF8-700°C/3h.



Figue 4. FTIR spectrum of four synthesized biochars: (A) CF5-600°C/1.5h; (B) CF6-600°C/3h; (C) CF7-700°C/1.5h and (D) CF8-700°C/3h.

92.32% (CF6), 91.99% (CF7) and 93.67% (CF8), respectively, compared with the original raw material sample with only 74.15% of C. All samples recorded the presence of metallic Ag element with the weight percent range from 0.06 to 0.09%. The Ag element has a great influence on the material's ability to remove coliforms due to its strong antibacterial properties (Cui et al., 2015). The adsorption efficiency of pollutants depends on the polar groups on the surface of the carbonate material. The chemical groups such as hydroxyl, carboxyl groups and functional bonding groups like -OH, CH, C = O affect strongly to the ionic adsorption capacity (Hirata et al., 2002). Therefore, the C content of over 90% after ashing has been demonstrated that biochar samples obtained from coffee grounds material indicate the ability to adsorb pollutants.

Table 2. The result analysis of pollutant parameters in the initial seafood wastewater sample.

Parameters	Unit	Results	QCVN 11:2015/MONRE	Exceedance factor
COD	mg/L	14,400	150	96
TSS	mg/L	151	100	1.51
NH_4^+	mg/L	385.14	20	19.26
Coliform	MPN/100 mL	46,000	5,000	9.2

The bonding characteristics (functional groups and bonds) on the surface of four biochar materials were determined by the FTIR spectrum. The results shown in Figure 4 reported that the adsorption spectrum of four biochar samples ranged from 500 ÷ 4.000 cm^{-1}. All samples have found associated groups such as N-H, C-O, C-H, C≡C, OH. The normal biochar materials showed an increase in intensity of oxygen-containing functional groups, especially C-O and O-H groups, and the position of C-O functional groups had changed significantly. These results proved the strong interactions of biochar materials with functional groups. In addition, no new peaks were observed in the FTIR spectrum, indicating that no new bonds were formed between the biochar materials and functional groups in aqueous solution. These results also demonstrated that the pyrolysis time significantly affected the material properties.

3.2. Evaluation of adsorption capacity of the biochar to organic pollutant and microbiological

To evaluate the removal capacity of organic pollutant and microbiological, the four water quality parameters (COD, TSS, NH_4^+-N and coliform) of the initial seafood wastewater sample were analyzed. The analytical results (Table 2) show that all four parameters exceed many times the allowable Vietnamese standards QCVN 11: 2015/MONRE.

3.2.1. COD removal capacity of the biochar

In this study, the COD removal capacity in wastewater sample was investigated at different intervals of time: at 0h (COD0); 1h (COD1) and 2h (COD2). The COD analysis results in Figure 5 show that, the COD value in the wastewater sample after adsorption by the biochar material significantly reduced comparing to the initial sample. CF7 material achieved the highest removal capacity and recorded COD value of 240 mg/L comparing to the initial sample with the COD value of 14.400 mg/L.

The COD removal efficiency of four different biochar materials was different at the same and different adsorption times (Table 3). The study reported that the highest efficiency of CF7 material sample was 98.3% 2h after the reaction time and the lowest value of CF8 material sample was 94.58% 1h after respectively. The organic treatment efficiency of all CF5-CF8 biochar materials was quite high, however, the COD value after the treatment had not met the output requirements yet according to Vietnamese standard QCVN 11: 2015/MONRE and the COD values were from 1.6 to 5.2 times higher than the standard limit values.

Table 3. The COD removal efficiency of four biochar materials after 1h and 2h.

Time (Hour)	COD (%)			
	CF5	CF6	CF7	CF8
1h	96.67	96.67	97.08	94.58
2h	97.08	97.92	98.3	96.25

3.2.2. TSS removal capacity

Similarly, the four biochar materials were evaluated for their adsorption capacity with total suspended solids (TSS) 1h and 2h after the reaction time . The results are illustrated in Figure 6 and Table 4. The analyzed results (Figure 6) indicate that the TSS content significantly decreased compared to the initial wastewater

Table 4. The TSS treatment efficiency of four biochar materials after 1h and 2h.

Time (hour)	TSS (%)			
	CF5	CF6	CF7	CF8
1h	61.19	45.30	52.32	82.25
2h	68.87	88.45	80.40	99.21

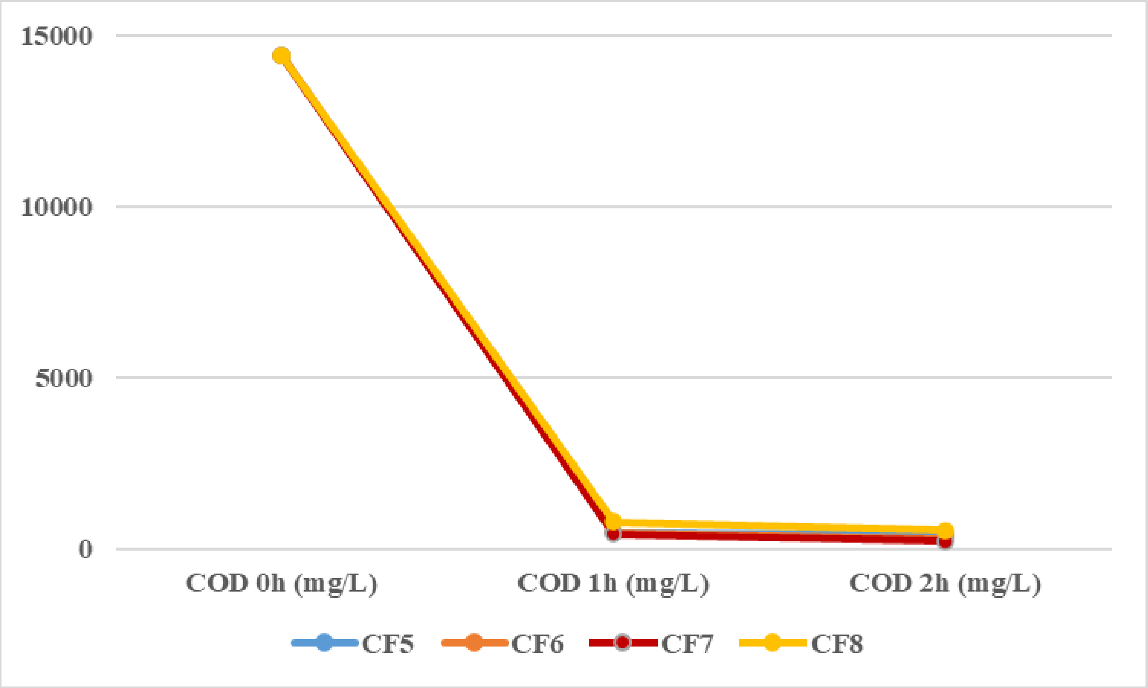


Figure 5. The COD removal capacity of four biochar materials after 1h and 2h (mg/L).

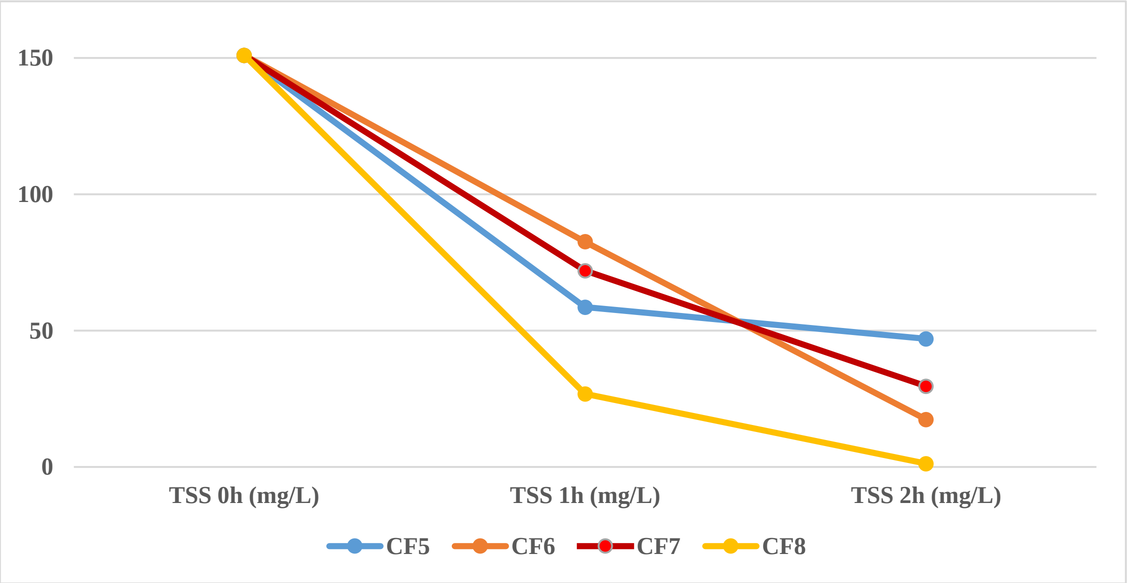


Figure 6. The TSS adsorption capacity of four biochar materials after 1h and 2h (mg/L).

sample while CF8 material achieved the highest adsorption capacity at the TSS value of 1.2 mg/L 2h after the reaction time in comparison with the initial wastewater sample of 151 mg/L.

The treatment efficiency (Table 4) recorded the highest value of 99.21% for CF8 sample after 2h and the lowest of 45.30% for CF6 sample after 1h. This result proved that the reaction time played an important role to the treatment efficiency of the biochar materials. From 1 to 2 hours after the reaction time, all four absorbents (CF5-CF8) reported the removal capacity of TSS output requirements according to the Vietnamese standard QCVN 11: 2015/MONRE.

3.2.3. NH_4^+ removal capacity

NH_4^+ content after the adsorption by the biochar materials changed according to the reaction time and is presented in Figure 7 and Table 5. 2h after the reaction time, NH_4^+ content in all four samples (CF5-CF8) decreased from 385.14 mg/L to 19.97; 15.55; 21.50 and 19.16 mg/L, respectively. Their NH_4^+ removal efficiency was quite similar with the value of above 94%. Our experimental results indicated that the NH_4^+ adsorption process not only depended on the pyrolyris time and temperature, but also depended on the reaction time of the biochar materials (1 and 2h). The highest treatment efficiency of NH_4^+ was observed after 2h with the adsorption efficiency of up to 95% after 1 and 2 hours recorded in CF6 sample, respectively.

Table 5. The NH_4^+-N treatment efficiency of four biochar materials after 1h and 2h.

Time (hours)	H% (amoni)			
	CF5	CF6	CF7	CF8
1h	94,17	95,45	92,7	94,51
2h	94,82	95,96	94,42	95,03

3.2.4. The microorganism removal capacity

The analyzed result of microbiological parameter is illustreated in Figure 8 and Table 6. It is shown that the coliform density significantly decreased compared to the untreated wastewater sample and met the output requirements according to the Vietnamese standard QCVN 11:2015/MONRE. Among the four materials, CF7 had obtained the highest coliform removal capacity of 98.61% with only 640 MPN/100 mL 2h after the reaction time and the lowest value for CF5 sample after 1h was of 4.300 MPN/100 mL with the treatment efficiency of 90.65% compared to the initial wastewater sample.

Table 6. The microorganism treatment efficiency of four biochar materials after 1h and 2h.

Time (hours)	H% (Coliform)			
	CF5	CF6	CF7	CF8
1h	90.65	96.74	97.78	92.17
2h	96.74	97.61	98.61	98.37

Our results are similar to the previous research published by Vu Thi Mai et al., (2016) when using synthezed biochar material obtained from coffee grounds to treat dyes and organic pollutant in textile wastewater; the FTIR measurement results were found similar wavelengths and peaks. The removal capacity with dyes and organic matter (COD parameter) had reached the treatment efficiency of up to 96.5%; or the study reported by Trinh Thi Thu Huong et al., (2015) who used biochar material modified corn cob H_3PO_4 and NaOH to treat organic pollution in wasterwater. The study also showed that the adsorption process occurred well at pH ≥7 and 60 minutes after the reaction time, the adsorption equilibrium had reached the highest value. In fact, the previous studies mainly

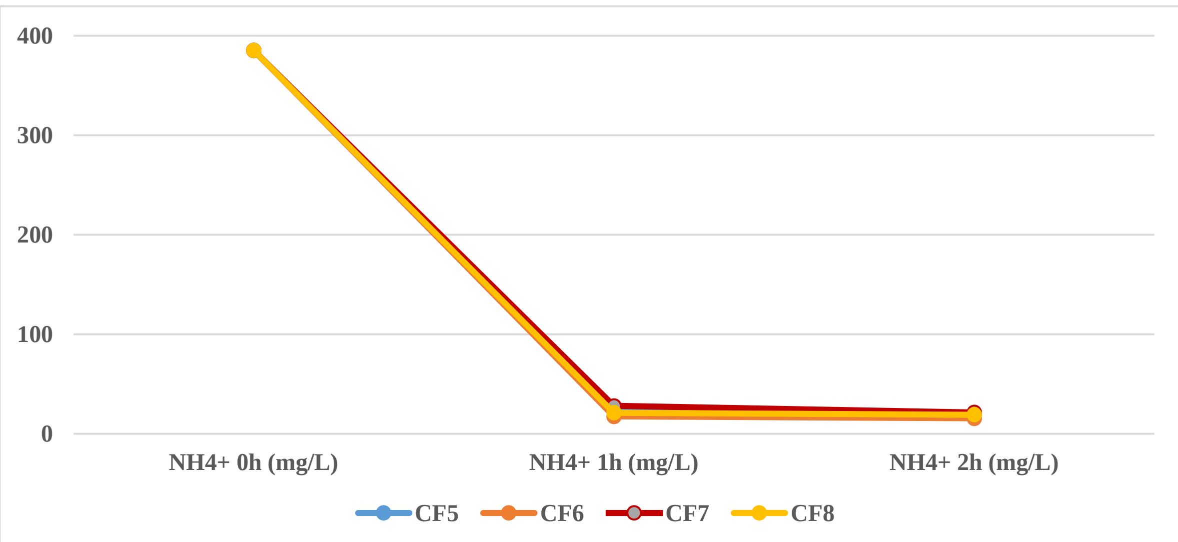


Figure 7. The NH_4^+-N adsorption capacity of four biochar materials after 1h and 2h (mg/L).

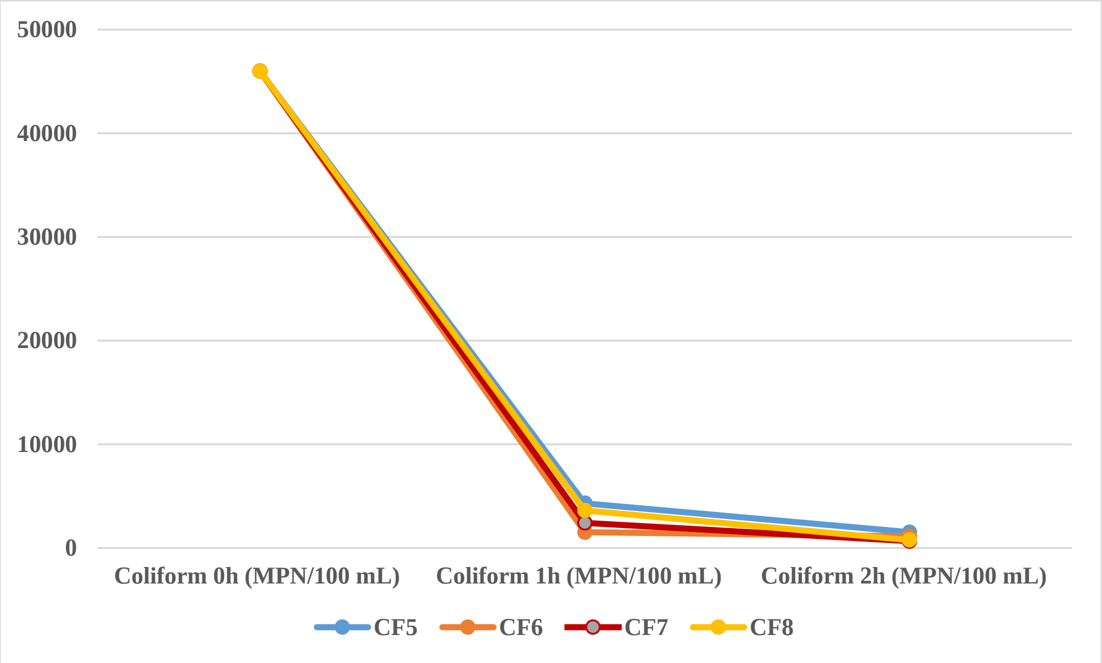


Figure 8. The microorganism adsorption capacity of four biochar materials after 1h and 2h (MPN/100 mL).

used biochar to absorb dye pollution (Hirata et al., 2002; Gehan et al., 2015), pesticides (Liu et al., 2015) or remove pathogenic bacteria (Cui et al., 2015). The seafood processing wastewater with a high content of organic pollutant leads to the difficulty of treatment and not many studies investigated this issue have been reported. Biochar is a material characterized by a high specific surface area and the highly microporous density due to having layers of graphene stacked, and aromatic structure alternating with graphene layer. This special structure of the material enhances physical adsorption capacity leading to increase treatment efficiency. For these characteristics, the biochar material has been proposed as the potential material source for treating organic pollution (Rebeca et al., 2012; Deng at el., 2017).

The removal capacity of NH_4^+ by the biochar material has been reported in many previous studies (Trinh Thi Thu Huong et al., 2015; Haider et al., 2014). The NH_4^+ treatment efficiency was quickly achieved with the results recorded of 77% by biochar material from the cob 1h after the reaction time (Trinh Thi Thu Huong et al., 2015), or ranged from 20.04÷94.3 % when using biochar modified organic acid (Haider et al., 2014). It can be seen that with the same modified biochar for ammonium removal, the different studies have noted different treatment results. According to Zhu et al. (2012), the ammonium content was adsorbed with certain efficiency basing on the chemical ion exchange mechanism on the biochar material surface from rice husk. Although the ammonium content had not fully completely removed, NH_4^+-N adsorption biochar can be used as a preservative fertilizer due to the concentration of NH_4^+-N removed from the wastewater solution which may increase in the samples. Similarly, the removal capacity of microbiological parameter showed that the number of coliform bacteria significantly decreased compared to the initial wastewater sample.

According to Cui et al. (2015), the biochar from rice husk modified with nano silver showed that inhibiting effect for pathogenic bacteria was much higher than the biochar without nanosilver. In the biochar samples without nanosilver, this inhibition was not as high as that of materials containing silver nanoparticle, but the effect on bacterial growth was recorded. It was indicated that Ag^+ ions play a major role in inhibiting bacterial growth, damaging the cell membrane and killing bacterial cells (Cui et al., 2015). Afrooz et al. (2016) reported that the microorganism removal process by biochar absorption depends on several factors such as surface area, hydrophilic and hydrophobic properties of the absorbent since these properties play the important role for attaching microorganism on the material surface. The porous structure can be a good habitat for bacteria (Afrooz et al., 2016; Gorovtsov et al., 2020). It is also noted that their physical and chemical properties (like surface area, particle size, hydrophobicity, polarity, etc.) are very different depending on the type of raw material used and method of biochar synthesis.

4. Conclusion

This study used four biochar samples modified $AgNO_3$ from coffee grounds including CF5 (600°C/1.5h), CF6 (600°C/3h), CF7 (700°C/1.5h), and CF8 (700°C/3h) for treatment of the organic contaminants and microorganism in the seafood-processing wastewater. The results found that the CF7 biochar material sample acheived the highest organic pollutant removal capacity (COD) with the adsorption efficiency of 98.30% 2h after the reaction time and the lowest value was the CF8 sample with 94.58% after 1h. However, the COD value after the treatment of all four material had not yet met the output requirements according to the Vietnamese standard QCVN 11: 2015/MONRE and the COD values were from 1.6 to 5.2 times higher than the standard limit values.

The three parameters (TSS, ammonium and microorganism) met the standard limit value requirement. 2h after the reaction time, the TSS content decreased to the lowest in CF5 sample (68.87%) and attained the highest in CF8 sample (99.21%); NH_4^+ content of the four absorbents (CF5-CF8) decreased from 385.14 mg/L to 19.97; 15.55; 21.50 and 19.16 mg/L, respectively, with the adsorption efficiency over of 94%. The coliform removal capacity of CF7 sample had the highest efficiency of 98.61% with the value of 640 MPN/100 mL and the lowest efficiency (90.65%) was observed in the sample CF5 with the decrease value of 4.300 MPN/100 mL. These results showed that the biochar modified by $AgNO_3$ is the potential material for removing pollution contamination in wastewater.

Author contributions

Huong Thu Thi Tran contributes to the idea, data acquisition, analysis, and writes the manuscript. Tong Xuan Nguyen, Toan Ngoc Vu, Nga Thanh Pham, Cuong Manh Nguyen, Van Thanh Doan, Cuong Manh Tran, Hieu Duc Tran, Huan Quoc Nguyen contribute to collecting the data.

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