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Dieu Tien Bui
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Proceedings of the International Conference on Innovations for Sustainable and Responsible Mining

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Preface

We would like to welcome you to the International Conference on Innovations for Sustainable and Responsible Mining - ISRM 2020, which will be held during October 15–17, 2020, at Hanoi University of Mining and Geology (HUMG), Hanoi, Vietnam. ISRM 2020 is organized by HUMG to celebrate the 55th anniversary of the Department of Surface Mining, Faculty of Mining, HUMG. The conference is the effective cooperation of HUMG and both TU Bergakademie Freiberg (Germany), and Hanoi University of Public Health (Vietnam). Especially, the event is financially supported by Vietnam National Coal-Mineral Industries Holding Corporation Limited (VINACOMIN), Dong Bac Corporation (NECO), and other organizations.

The main aim of the ISRM 2020 is to provide a platform for researchers, academicians, and engineers in the field of mining, earth resources, civil engineering, and geospatial technologies to present their recent research results. Besides, the conference provides a setting for them to exchange new ideas, innovative thinking, and application experiences face-to-face, to establish research or business relations, and to find partners for future collaboration.

The conference program was organized into four sessions covering topics of sustainable development and responsible mining, earth sciences and geospatial technologies, occupational safety and health, and Industry 4.0 in mining. The ISRM 2020 has received 344 submissions, and among them, 68 high-quality manuscripts were recommended to submit for the section earth sciences and geospatial technologies of this Springer proceedings book for a double-blind peer-reviewing. Herein, each manuscript has been reviewed for its merit and novelty by at least two reviewers by matching the content areas. As a result, a total of 21 papers have been finally selected for this book. We believe that this proceedings book provides a broad overview of recent advances in earth sciences and geospatial technologies for readers.

Finally, we would like to express our sincere thanks to the university council of HUMG, the rector and vice-rectors of HUMG, and the International Office of HUMG for their help in administrative works and other supports. Special thanks to Dr. Nguyen Quoc Long, the secretary of the ISRM2020, and Pierpaolo Riva at

Springer for help and always responding promptly. We would like to thank all the reviewers for their timely and rigorous reviews of the papers and to thank all the authors for their submissions.

October 2020

Dieu Tien Bui
Hai Thanh Tran
Xuan-Nam Bui

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Prof. Dieu Tien Bui is currently a full professor at the GIS group, Department of Business and IT, University of South-Eastern Norway (USN), Bø i Telemark, Norway. He is a global highly cited researcher 2019, ranking among the top 1% by citations for Cross-Field in Web of Science. Besides, he is an editorial board member of scientific journals, Geomorphology (Elsevier), Remote Sensing (MDPI), Journal of Mountain Science (Springer), and Vietnam Journal of Earth Sciences (VAST). Also, he is a representative of the National Norwegian IAG Geomorphology Group (GeoNor) members at Norwegian universities. He received the M.Sc. degree in Cartographic Engineering from Hanoi University of Mining and Geology, Hanoi, Vietnam, in 2004, and the Ph.D. degree in Geomatics at Norwegian University of Life Sciences (NMBU), Ås, Norway, in January 2013. He was a postdoctoral researcher at NMBU between 2013 and 2014. From 2004 to 2007, he was a university lecturer at Faculty of Surveying and Mapping, Hanoi University of Mining and Geology. In 2008, he was a geospatial analyst at Ugland IT Group, a geographic information services company in Lysaker, Oslo, Norway. He has more than 200 publications, and out of them, >180 articles were published in Science Citation Index (SCI/SCIE) indexed journals, >10 book chapters published by Springer and Elsevier, and one edited book published by Elsevier. He is a reviewer for more than 30 SCI/SCIE journals. His research interests are GIS and geospatial information science, remote sensing, and applied artificial intelligence and machine learning for natural hazards and environmental problems, such as landslide, flood, forest fire, ground biomass, and structural displacement.

Prof. Hai Thanh Tran is currently a professor of Geology at the Department of Geology, Hanoi University of Mining and Geology (HUMG), Vietnam. He received the MSc in Geoscience in 1997 and the Ph.D. degree in 2001, both from the University of Regina, Canada. He has been working for more than 30 years on regional geology, structural interpretation, tectonic evolution, and their relationship to natural resources and geohazards in Vietnam and other countries. He is the

author and co-author of nearly 40 international scientific papers, six books, and the presenter of more than 40 international presentations relating to his scientific interests. He is a member of the editorial board for several domestic and international scientific journals. Besides his official duty of the rector at the HUMG, he has also been a member of several professional organizations both in Vietnam and internationally, including vice president and president of the Earth Science Council of the National Foundation for Science and Technology Development of Vietnam (NAFOSTED), vice president (2014, 2017), and president (2015–2016) of the Solid Earth Section (SE) Session of the Asia-Oceana Geological Society.

Prof. Xuan-Nam Bui is currently a full professor at the Surface Mining Department, Faculty of Mining, Hanoi University of Mining and Geology (HUMG), Vietnam. He received the M.Eng. degree in Mining Engineering from HUMG in 2001 and the Dr. Eng. Degree in Mining Engineering from TU Bergakademie Freiberg (Germany) in 2005. He has been working at HUMG since 1996. His research interests are environment-friendly mining technology and engineering, occupational safety and health in the mining industry, and applications of artificial intelligence and machine learning in geoengineering, mining, and environmental issues, such as ground vibration, overpressure, fly rock, air pollution, and slope stability. He is the editor-in-chief of the Journal of Mining and Earth Sciences, HUMG, and an editorial board member of several international scientific journals. He has more than 60 articles published in ISI and Scopus Indexed Journals and Springer chapters. Currently, he is a member of the Society of Mining Professors and vice president of the Vietnam Association of Mining Science and Technology.

Initial Results of Using Biochar Derived from Spent Coffee Grounds to Remove Pollutants from Livestock Wastewater in Vietnam

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Abstract. Biochars derived from spent coffee grounds were pyrolysed at different temperatures and retention times, including CF1-CF4 samples (500 °C for 0.5, 1.5, 3, 6 h); CF5-CF8 samples (600 °C for 0.5, 1.5, 3, 6 h) and CF9-CF12 samples (700 °C for 0.5, 1.5, 3, 6 h). These biochars were examined to determine their ability to remove pollutants (COD, TSS, total N and total P) from livestock wastewater. The initial livestock wastewater was treated with 12 types of biochar with masses of 2, 4 and 6 g at reaction times from 1, 2, 4 to 8 h to assess the adsorption efficiency of the biochar. Adsorption efficiency for these pollutants increased with increasing reaction time and biochar mass. The combination of 8 h reaction time and 6 g biochar weight showed the highest adsorption efficiency. At an 8 h reaction time with 4 g biochar, only COD content was adsorbed by the CF4 biochar sample at a level meeting the output requirements according to the Vietnam standard QCVN 40:2011/MONRE national regulation for industrial wastewater; the remaining 11 treated wastewater samples retained pollutant concentrations that were 1.6 to 3.6 times higher than the acceptable values. The TSS content in all 12 samples met the standard limit value requirement. The total N content was 3.3 to 4.2 times higher (excepting the CF4 sample) and the total P content was 1.07 to 1.15 times higher (excepting the CF4, CF8, CF9 and CF11 samples) than the standard limit values. With 6 g biochar and 8 h reaction time, all four parameters adsorbed with 12 biochar samples were significantly reduced, producing water with concentrations lower than the required limit according to the QCVN 40:2011/MONRE regulation. The results showed that the biochar made from spent coffee grounds is a potential sorbent to remove pollutants from livestock waste water.

Keywords: Adsorption capacity · Biochar · Coffee ground · Livestock wastewater · Pollutants · Vietnam

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1 Introduction

Organic pollution from livestock wastewater is a major problem in many countries because of its danger to the environment and human health [1]. Many technological solutions to neutralize pollutants have been developed to protect the environment. Currently, wastewater treatment technologies are available to remove pollutants at various scales and levels. However, none of them is optimal for all goals and economic conditions. Many treatment methods have been successfully applied to many types of wastewater, but they usually have high operating and maintenance costs, generate secondary toxins, or involve complicated operations [2]. One of the most concerning issues is the need to find alternative materials to support wastewater treatment technology. Biochar modified from agricultural wastes or other carbon-rich materials is potentially a low-cost environmental treatment material, particularly for organic pollution remediation [2]. Recently, biochar has received a lot of attention worldwide when applied in wastewater treatment thanks to its high adsorption efficiency and reusability. In addition, its production cost is low and it is safe for the environment [1]. Many researchers have used biochar adsorption for organic pollutants [3], such as sulfamethoxazole [4], phenanthrene and triazine pesticides [5], and heavy metals including mercury, lead and cadmium [6] from waste water. Popular processes often used to synthesize biochar include pyrolysis, gasification, and hydrothermal carbonization [1].

Coffee is a widely consumed drink with beans grown in more than 80 countries worldwide, and is considered one of the most important goods in these countries [7]. The total amount of coffee consumed worldwide exceeds 11 billion tons/year [7]. According to the International Coffee Organization (ICO), the global coffee yield has steadily increased during the last 150 years [8]. Coffee brew production is estimated at 151.6 million bags (60 kg/bag) for 2015–2016, and coffee grounds have filled up large landfills [7]. It is estimated that more than 4.4 million tons of brewed coffee waste are disposed of by this industry every year [9]. Coffee grounds contain high levels of substances known to be toxic to many life processes, such as acids, free phenols, caffeine, and tannins (polyphenols) [10]. Coffee waste thus constitutes a source of serious environmental problems in coffee-using countries [11]. Many authors have already reported that coffee sub-products and their wastes could be used in many ways [12]. Biochar has been used as a useful and economical material in many fields such as food additives, biogas, caffeine, pectin, feeds, protein, antioxidants, beverages, enzymes, compost, and the production of bioactive compounds [12]. According to some previous studies, protein content in coffee grounds accounts for about 10%, pectin content for 52.62–55.14%, and cellulose for 15.29–17.04% of the mass of the grounds, which also have high carbon content (above 50%) [13]. Coffee grounds are a lignocellulosic material that can separate heavy metals and dye in water based on their porous structure and cellulose composition. The surface of carbonaceous materials contains many phenolic hydroxyl and carboxyl groups. In cellulose materials, these groups play an important role in ion exchange, and studies of treatment effectiveness indicated that the adsorption of pollutants depends upon the surface polar groups on the carbonaceous materials [13]. Therefore, spent coffee grounds are an effective material in the synthesis of biochar in order to remove pollution from livestock wastewater.

The livestock industry accounts for about 40% of global agricultural output, creating jobs for more than 1.3 billion workers and supporting the livelihoods of more than 1 billion people in poor countries [1, 14]. Agricultural economic development associated with large-scale livestock production is an important means to help farmers increase their incomes [15]. Farming with large numbers of livestock will release large masses of waste; the aquatic environment is seriously threatened by pollutants such as phosphorus, ammonium and heavy metals. In Vietnam, the total amount of livestock waste produced is about 73 million tons/year, of which pig waste accounts for about 33.4% or 24.38 million tons/year, and 25–30 million cubic meters of liquid (liquid faeces, urine and rinse water). Of this, about 50% of solid waste (36.5 million tons) and 80% of liquid waste (20–24 million m^3) are released directly into the environment or without treatment, which causes serious environmental pollution [16]. Biochar's potential as an adsorption technology in wastewater treatment, especially for the low-risk handling of typical pollutants in livestock wastewater, is a current focus of efforts to make agricultural activity more environmentally sound. Many authors previously reported the result that the typical pollutants in livestock wastewater, such as organic pollutants [17], heavy metals [18], nitrogen, and phosphorus [19] could be adsorbed well by biochar. Therefore, biochar can be used as a slow-release fertilizer and is a material with agricultural environment-friendly characteristics [20].

According to the Ministry of Agriculture and Rural Development, the coffee-growing area in Vietnam in 2014 was estimated at 653,000 hectares, and the output reached 1.49 million tons. Vietnam is an agricultural country with the second-largest coffee export mass in the world (after Brazil). The total domestic consumption of coffee is 60,000 tons/year, of which instant coffee accounts for about 19,000 tons, roast/grind coffee accounts for 35,000 tons, and the remainder is unbranded roast/grind coffee [21]. Spent coffee grounds are a carbon-rich material with many advantageous characteristics [8, 9, 12]. Vietnamese scientists have carried out experiments on biochar mainly to improve soil characteristics for agriculture [22–24]. Studies of biochar for pollutant adsorption in Vietnam have included investigating the characteristics of biochar from rice husk [25], biochar modified H_3PO_4 and NaOH for ammonium removal [26], the fixation of pesticides such as propoxur by biochar [27], using biochar modified from corn cobs to remove ammonium in mixture wastewater [28], etc. However, studies of biochar's utility for removing pollutants/organic pollution or pathogenic microorganisms from livestock wastewater are very limited. From the above data, it can be seen that Vietnam's annual yield of coffee grounds is huge; they are usually discarded, wasting a potential source of raw materials. To best utilize this strength of a third world coffee-exporting nation, we propose the synthesis of biochar materials by the slow pyrolysis method from spent coffee grounds to remove pollutants from livestock wastewater in an eco-friendly manner. In this study, laboratory experiments were performed to survey the removal potential of organic pollution from livestock wastewater by biochar pyrolysed from spent coffee grounds.

2 Materials and Methods

2.1 Preparation of Carbon Material from Spent Coffee Grounds by Pyrolysis

The coffee grounds used as raw materials were collected from a local company in Dak Lak province (An Thai Development and Investment Joint Stock Company, Lot B03–04 – Hoa Phu Industrial Pack – Buon Ma Thuot City – Dak Lak Province – Viet Nam) and a coffee shop in Ha Noi city (Titi Coffee Shop, No. 20 Vien Street, Duc Thang Ward, Bac Tu Liem District, Ha Noi city, Viet Nam). The steps to collect raw material are described in Fig. 1 and Fig. 2 as follows:

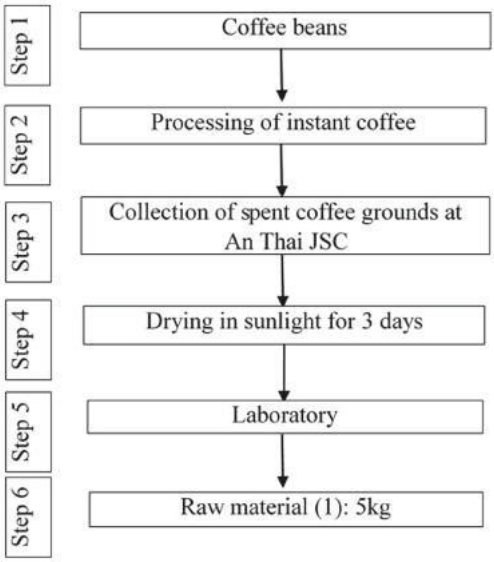

Fig. 1. Process of collecting raw material from the An Thai JSC

To remove the chemicals left in coffee grounds after brewing, the spent coffee grounds were washed multiple times with water by centrifugation methods and then filtered [39]. Next, the raw material was mixed together thoroughly and then dried at 105 °C in an oven for 10 h to evaporate the solvent completely. The pyrolysis of the material was then conducted at 500 °C, 600 °C and 700 °C in the presence of nitrogen. The heating rate of the furnace was set at 5 °C/min and temperature was maintained for 0.5 h, 1.5 h, 3 h and 6 h [30]. After the end of the pyrolysis phase, the material was manually ground to enhance the homogeneity in the whole material. 12 types of biochar material including CF1 (500 °C/0.5 h), CF2 (500 °C/1.5 h), CF3 (500 °C/3 h), CF4 (500 °C/6 h), CF5 (600 °C/0.5 h), CF6 (600 °C/1.5 h), CF7 (600 °C/3 h), CF8 (600 °C/6 h), CF9 (700 °C/0.5 h), CF10 (700 °C/1.5 h), CF11 (700 °C/3 h), CF12 (700 °C/6 h) and a control sample (raw spent coffee grounds) were prepared for further experiments. All chemicals and reagents used in the current research were ordered from Merck with A.C.S. certification.

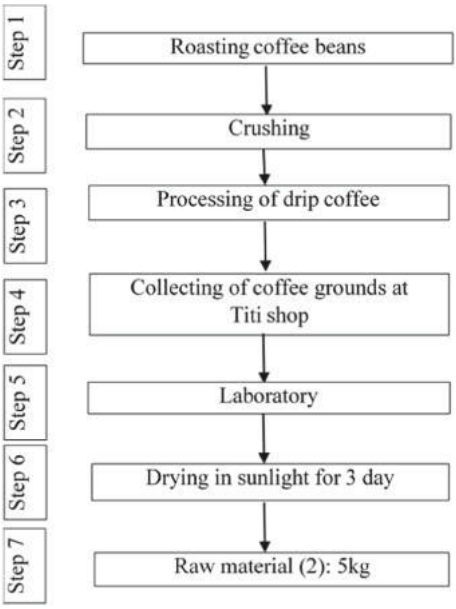

Fig. 2. Process of collecting raw material from Titi Coffee Shop

The collected materials were evaluated for their ability to remove pollutants (COD, TSS, Total N and total P) based on three characteristics, including: (1) reaction time, (2) adsorbed biochar weight and (3) effective adsorption.

2.2 Identification of Material Characteristics

The biochar material samples were characterized in the following manner: energy-dispersive X-ray spectroscopy (EDX) was used to determine the elemental composition of the material, and a scanning electron microscope (SEM) system was used to analyse the surface size and morphology. The Brunauer Emmett Teller (BET) technique was used to analyse the surface area and properties; the N2-BET surface area and properties of the biochar samples were measured by Tristar 3000. A Fourier Transform Infrared Spectrometer (FTIR, TESOR II- BRUCKER, USA) was also used to record and determine the surface functional groups on the sample.

2.3 Determination of the Adsorption Capacity of the Materials

Sampling and Chemical Analysis

The initial livestock wastewater was collected from a farm in Vinh Phu province and preserved before experiments in the laboratory. The wastewater samples were immediately analyzed for contamination factors to determine the initial pollution concentrations for COD, TSS, total N and total P. The analysis results in Table 1 showed that all four parameters exceed the permitted values according to the Viet Nam national regulation QCVN 40:2011/MONRE for industrial wastewater: COD concentrations exceeded their

limit by a factor of 52, TSS by a factor of 2.7, Total N by a factor of 13.66 and total P by a factor of 3.66.

Table 1. Analysis results of pollutants in the initial livestock wastewater sample

Parameter	Unit	Concentration	Vietnam national standard 40:2011/MONRE	Exceedance factor
COD	mg/L	7800	150	52
TSS	mg/L	270	100	2.7
Total N	mg/L	546.32	40	13.66
Total P	mg/L	21.95	6	3.66

Experimental Setup

To determine the adsorption capacity and efficiency of the material, the raw livestock wastewater was treated with 12 types of biochar samples at different reaction times of 1, 2, 4 and 8 h. After the livestock wastewater was filtered through filtering columns made from twelve types of biochar with various combinations of reaction time and biochar mass, the treated wastewater sample was collected and analyzed for indicators of COD, TSS, total N and total P.

The adsorption capacity q_e(mg/g) at equilibrium time is determined by the formula [31]:

$$q_e = (C_o - C_e.V/m) \quad (1)$$

The adsorption efficiency H (%) at equilibrium time is determined by the formula [32]:

$$H = (C_o - C_e)/C_o \cdot 100(\%) \quad (2)$$

where q_e is the adsorption capacity at equilibrium (mg/g), C_o is the initial concentration (mg/L), C_e is the concentration at equilibrium (mg/L), V is the volume of solution (L), and m is the mass of adsorbent material (g).

To study the effect of the reaction time, the concentration of the initial adsorbent solution C_o (mg/L), the adsorption volume (V = 100 mL), and the amount of the adsorbent (m = 4 g) were fixed, and the reaction time was varied with t = 1, 2, 4 and 8 h. For asssesing the effect of the adsorbent mass, the concentration of the initial adsorbent solution C_o (mg/L), the adsorption volume (V = 100 mL), and the reaction time (t = 8 h) were fixed, and the amount of the adsorbent was changed with m = 2, 4 and 6 g.

The pollution parameters were analyzed following the standard methods listed in Table 2.

Table 2. Standard methods of water analysis used in this study

Parameter	Method
COD	ISO 6060:1989
TSS	ISO 11923:1997
Total nitrogen	ISO10048: 1991
Total Phosphorus	ISO 6878:2004

The optimization experimental conditions are described in the chart as follows (Fig. 3):

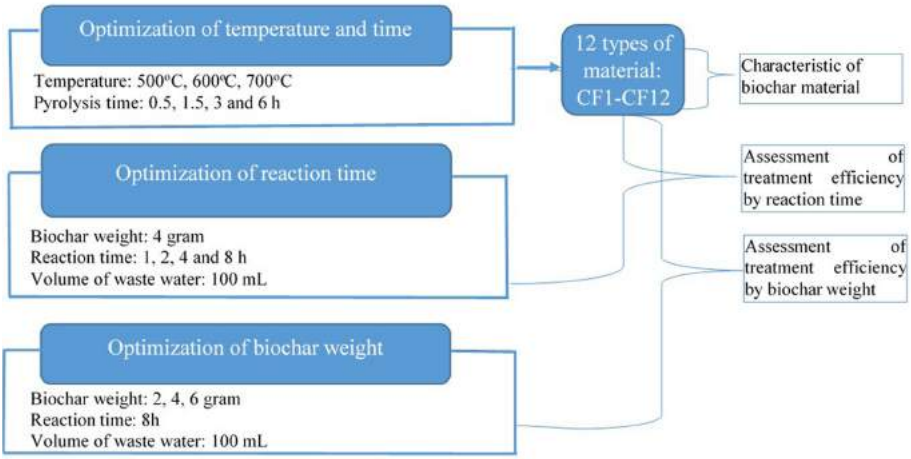

Fig. 3. Summary of optimization experimental conditions

3 Results and Discussion

3.1 Characteristics of the Biochar Samples

The selected properties of 12 biochar samples are listed in Table 3. The ash content of all these sorbents was relatively high (>25%), especially CF4 (31.25%). The CF4 sample also had the largest BET surface area of the 12 tested biochar samples. The BET surface area of the other samples was quite low, varying from 0.7917 to 1.2466 m^2/g. The results of elemental analysis showed high C content in all 12 biochars (over 80%). The surface element contents in Table 3 showed that the CF4 biochar sample had a C content of 90.61% on its surface, while the CF9 sample had the smallest surface C content (80.58%). However, the CF9 sample had the highest O content (13.46%), indicating that the CF9 sample may have the most functional oxygen groups.

Table 3. Biochar physical and chemical properties

Biochars	Pyrolysis temperature and time (°C)	Ash content (%)	BET surface area (m^2/g)	Surface elements		
				C (%)	O (%)	N (%)
CF1	500 °C/0.5 h	28.6	0.7917	84.55	9.98	3.91
CF2	500 °C/1.5 h	29.6	0.8564	84.61	9.06	4.82
CF3	500 °C/3 h	30.94	1.2466	84.65	9.53	4.06
CF4	500 °C/6 h	31.25	1.5016	90.61	6.86	1.41
CF5	600 °C/0.5 h	27.42	1.085	83.29	10.10	3.92
CF6	600 °C/1.5 h	27.57	0.9766	84.15	10.38	2.24
CF7	600 °C/3 h	27	0.7233	84.13	10.43	2.55
CF8	600 °C/6 h	26.55	0.8425	81.09	12.25	2.95
CF9	700 °C/0.5 h	25.11	0.956	80.58	13.46	3.29
CF10	700 °C/1.5 h	25.51	1.1071	81.76	12.73	2.62
CF11	700 °C/3 h	26,41	1.1602	81.63	12.79	2.94
CF12	700 °C/6 h	25.30	1.0569	80.97	11.26	3.76

SEM images of the material structures were consistent with some previously published studies [17, 33–36]. The SEM micrographs (Fig. 5, 6 and 7) of the 12 biochar samples showed that their structures were homogeneous and differed little from each other. It was also observed that the biochar samples had pores in them, appearing in microporous form, which did not exist in the raw material (Fig. 4). The raw material has a dense material surface without porous structure and lacking in cellulose fibres. Most biochar samples produced from carbonized coffee grounds have many large-diameter pores on their rough surfaces. However, the CF4 biochar sample showed a significant difference in the surface structure compared to the remaining biochar samples: its porous surface structure was observed to be the most homogeneous among the 12 samples. CF4 is the sample with the highest C surface content (90.61%). The rough surface and porous structure with small channels of these biochars can be clearly observed and may have been either produced during the pyrolysis process or a characteristic of the raw material [34]. These characteristics will strongly affect their adsorption properties [33, 34]. It is also shown that biochar materials have better adsorption capacity than raw materials.

The FTIR results of the 12 biochar samples are shown in Fig. 5, 6 and 7. The spectra of sample groups pyrolysed at different temperatures clearly differ from each other. In this study, the adsorption spectra of 12 biochar samples varied from 400 to 4000 cm^{-1}. All samples show spectral features associated with bonds such as C=O, O-H, C-C, C=C, etc. Due to high C contents of over 80%, the FTIR spectra of all 12 samples show peaks in the range from 3423 to 3434 cm^{-1} for adsorption groups of O=H. The C=C groups also appeared in most samples, with a peak at 1623 to 1628 cm^{-1} (CF2, CF4 and CF5-CF8); the CF1 and CF3 samples appeared to show C=C adsorption groups as a peak at 1574–1583 cm^{-1}. However, the biochar samples pyrolysed from coffee

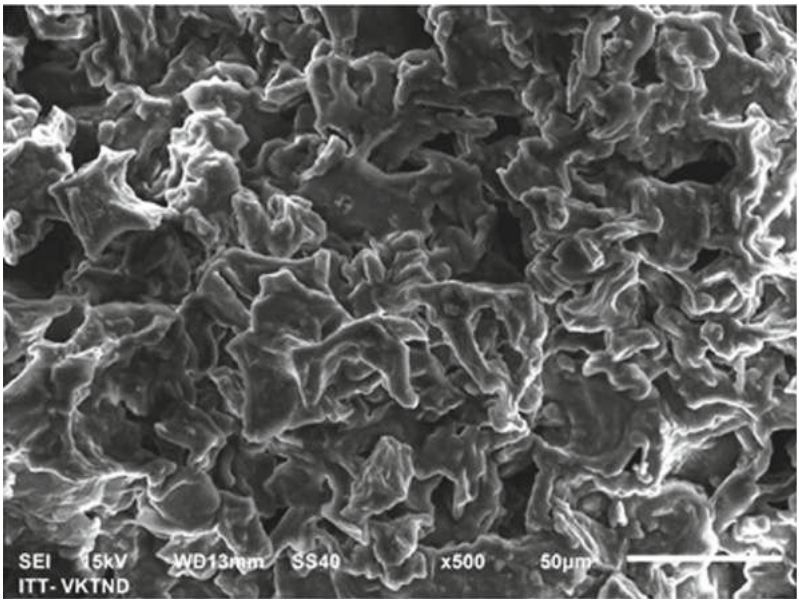

Fig. 4. SEM images of raw material samples (spent coffee grounds)

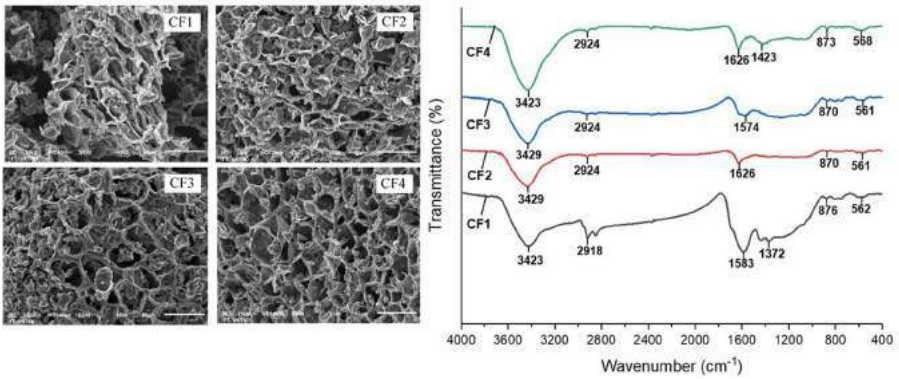

Fig. 5. SEM images and FTIR spectra of biochar samples pyrolysed from spent coffee grounds at 500 °C

grounds at 500 °C also show the adsorption groups of a C-H stretch (peak at 2918–2924 cm^{-1}), C-H aromatics (out-of-plane bend, peak at 873–876 cm^{-1}), C-Cl groups (peak at 561–568 cm^{-1}) and N=O (peak at 1372–1423 cm^{-1}). Moreover, adsorption groups in biochar samples pyrolysed from coffee grounds at 700 °C are observed that appear in the 1000–1400 cm^{-1} band for C-F bonds. Finally, of the biochar samples pyrolysed from coffee grounds at 600 °C, only the CF8 sample contains adsorption groups of C-F (1089 cm^{-1}) and C-Cl (596 cm^{-1}). These results indicated that pyrolysis time and temperature significantly affected the material characteristics. Some authors

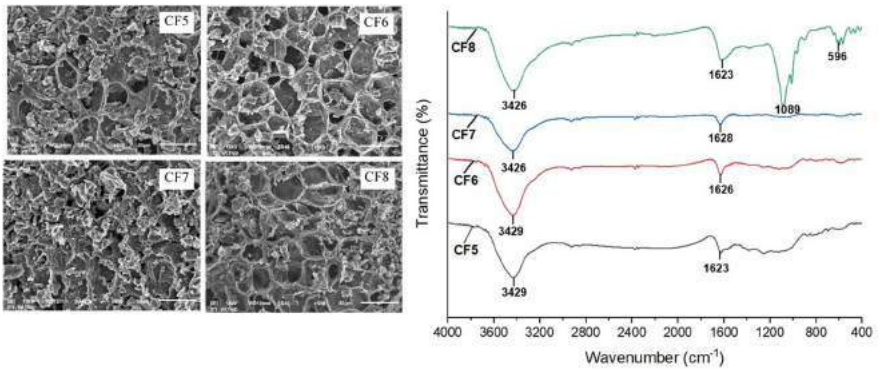

Fig. 6. SEM images and FTIR spectra of biochar samples pyrolysed from spent coffee grounds at 600 °C

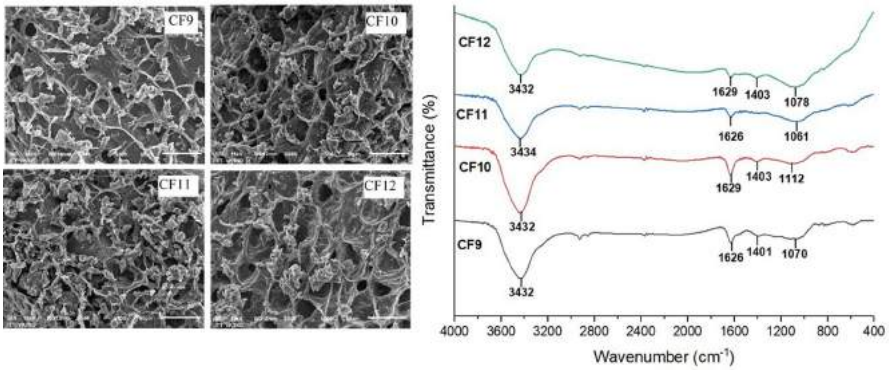

Fig. 7. SEM images and FTIR spectra of biochar samples pyrolysed from spent coffee grounds at 700 °C

have reported that the combination of micropores with larger pores [35] will lead to an increase in porous structure and decrease in surface area; therefore adsorption capacity for pollutants increased accordingly [35].

3.2 Effect of the Reaction Time

The concentrations of COD, TSS, total N and total P before and after adsorption by the 12 biochar samples are shown in Fig. 8 and Table 4. The differing results of COD, TSS, total N and total P were analysed in 12 different biochar samples, showing that the biochar had complex compositions. These results are consistent with the FTIR results obtained for the material characteristics. After different reaction times (from 1 to 8 h), the COD contents of almost all samples were significantly reduced (Fig. 7). The fastest reduction efficiency was recorded after 1 h of treatment; the reduction speed tended to slow down after 2 to 4 h and reached the highest efficiency after 8 h. The amount of COD adsorbed by the CF4 biochar sample was highest (98.08%) at the reaction time of

8 h and lowest (47.18%) after 1 h adsorbed with CF12 biochar. However, only the COD parameter in the sample treated with the CF4 biochar sample for 8 h met the output requirements according to the Viet Nam standard QCVN 40:2011/MONRE - national regulation on industrial wastewater. The other samples remained 1.6 to 3.6 times higher than the standard limit values.

Figure 8 and Table 4 shows the TSS analysis results after adsorption onto the biochar samples. The results showed that when the reaction time was altered from 1, 2, or 4 h to 8 h, the capacity of the 12 biochar samples to adsorb the TSS increased, removing colour and clear contaminants in wastewater. As seen for the COD parameter, the adsorption efficiency of TSS reached its highest value after 8 h, the fastest adsorption efficiency was obtained at the reaction time of 1 h. Between 2 h and 4 h, the adsorption speed decreased. The TSS content of wastewater treated with all 12 biochar samples after adsorption met the output requirements according to the QCVN 40:2011/MONRE standard. Of these, the CF4 biochar sample was observed to have the highest adsorption efficiency (95.56%) at 8 h while the lowest value was observed for the CF12 sample (63.7%) at 1 h. The remaining samples showed reductions in TSS content varying from 67.78% (CF5) to 93.7% (CF6) after 1 and 8 h adsorption, respectively.

Compared to pollutant parameters in the initial wastewater, the total N concentrations of all water samples after adsorption onto biochar were reduced significantly at a reaction time of 1 h. Between 2 h and 4 h, the reaction speed slowed. The total N value of the CF4 biochar water sample was reduced with the highest adsorption efficiency (93.16%) at a reaction time 8 h, while the lowest adsorption efficiency (25.56%) was observed in the CF3 sample at a reaction time of 1 h. Only the total N value obtained from the CF4 biochar sample after 8 h met the output requirements according to the Viet Nam standard QCVN 40:2011/MONRE, similar to the COD parameter results. The other 11 water samples had total N values 3.3 to 4.2 times higher than the standard limit values.

Results of the experiments to assess the effect of reaction time on total P are shown in Fig. 7. As with COD, TSS and total N, total P decreases significantly after a reaction time of 1 h. At 2 h, a further decrease is only observed in some samples (CF6, 7, 8 and CF10). At 4 h reaction time, the adsorption speed also decreased. Adsorption efficiency of total P increased significantly and reached its maximum (77.04%) after 8 h in the CF4 sample. The lowest result (15.44%) was obtained from the CF12 sample at 1 h reaction time. However, the total P parameter in biochar samples differed significantly from other parameters such as COD, TSS and total N. The total P parameter concentrations in the CF4, CF8, CF9 and CF11 samples was lower than the QCVN 40:2011/MONRE limit values, and the other samples (CF1 to CF3, CF5 to CF7, CF10, CF12) were 1.07 to 1.15 times higher than the standard limit values.

The reaction time in a batch sorption process is an important parameter in determining the capacity of a sorbent. The longer the adsorption time, the higher the processing efficiency [33]. The adsorption capacity (q_e) of the pollutants changed with the reaction time as shown in Table 5. It can be clearly seen that when increasing the reaction time, the adsorption capacity for these pollutants increased as well. A similar result was found for adsorption efficiency. The result in Table 5 shows that the adsorption capacity of the COD parameter reached a maximum of 191.2 mg/g for the CF4 sample at 8 h, and a minimum of 92 mg/g for the CF12 sample at 1 h. Similarly, the adsorption capacity of

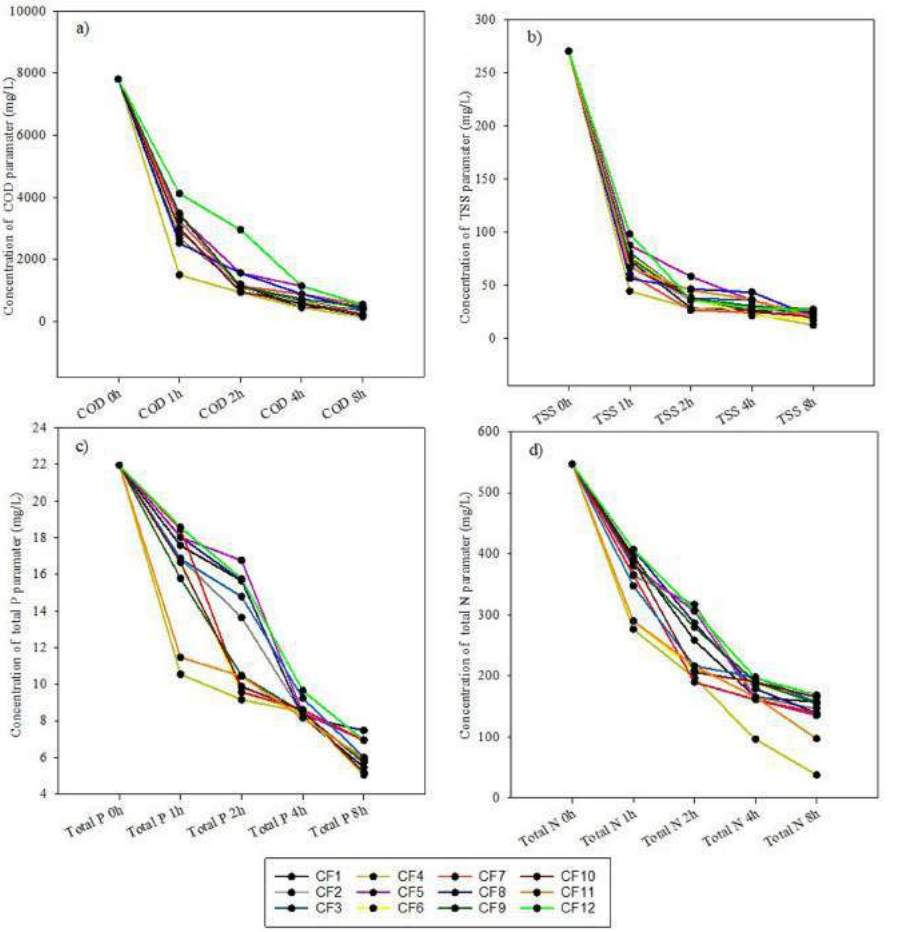

Fig. 8. Changes in concentration in wastewater of (a) COD, (b) TSS, (c) total P and (d) total N parameters adsorbed with biochar (mg/L) at the reaction times of 1, 2, 4 and 8 h

TSS also reached 6.45 mg/g (CF4 sample) at 8 h and 4.3 mg/g (CF12 sample) at 1 h. Total N adsorption capacity reached as high as 12.725 mg/g when adsorbed with the CF4 biochar sample for 8 h, and as low as 3.492 mg/g with the CF3 biochar sample at 1 h. The adsorption capacity of total P is also similar to the above three parameters: the highest adsorption capacity value is 0.423 mg/g for the CF4 sample at 8 h and the lowest value observed is 0.085 mg/g for the CF12 sample at 1 h.

Different biochar types exhibit different adsorption capacities for pollutants with different reaction times. However, all biochar samples in this study had an adsorption capacity which allowed removal of pollutants in the livestock wastewater sample. In the three groups of biochar materials, the group of materials pyrolysed from spent coffee grounds at 500 °C has the highest adsorption efficiency, followed by the 600 °C group and finally the 700 °C group. Moreover, this result is consistent with results obtained

Table 4. The adsorption efficiency (H%) of COD, TSS, total N (ΣN) and total P (ΣP) by 12 biochar samples

	H (%)											
	CF1	CF2	CF3	CF4	CF5	CF6	CF7	CF8	CF9	CF10	CF11	CF12
COD 1 h	55.90	62.56	55.38	80.76	56.41	64.62	58.46	67.69	55.38	61.54	65.64	47.18
COD 2 h	85.38	84.62	86.15	88.01	80.00	86.15	85.38	80.00	85.64	87.69	85.38	62.18
COD 4 h	94.36	93.08	91.79	94.36	85.38	92.05	88.72	88.72	90.77	92.56	93.08	85.38
COD 8 h	94.36	95.38	95.38	98.08	93.08	94.87	93.08	94.87	93.85	96.92	97.69	93.08
TSS 1 h	72.96	74.81	70.37	83.70	67.78	71.11	77.78	79.26	72.22	75.19	75.56	63.70
TSS 2 h	89.63	87.04	86.30	89.63	78.52	86.30	90.37	82.96	86.30	85.93	83.33	86.67
TSS 4 h	90.37	88.89	86.67	91.48	87.04	92.22	91.11	84.07	88.89	90.74	86.67	89.63
TSS 8 h	90.74	93.33	93.33	95.56	91.85	93.70	91.85	92.59	90.00	92.59	92.59	90.37
ΣN 1 h	27.56	33.42	25.57	49.50	30.64	47.01	33.21	36.54	29.65	28.70	47.00	25.60
ΣN 2 h	52.82	42.06	47.70	64.04	44.01	61.86	65.32	60.50	48.83	62.35	60.50	43.88
ΣN 4 h	69.85	69.83	67.33	82.48	70.57	65.87	70.57	63.84	65.32	65.29	69.84	64.16
ΣN 8 h	71.32	73.30	74.63	93.17	75.30	71.10	74.63	71.36	71.69	69.82	82.29	69.18
ΣP 1 h	19.95	23.14	17.81	51.98	17.90	23.74	15.90	23.28	28.11	24.19	47.79	15.44
ΣP 2 h	28.75	37.86	28.75	58.31	23.69	56.54	56.40	32.67	52.35	55.08	52.48	28.29
ΣP 4 h	62.60	62.00	62.51	61.37	61.69	61.14	60.82	57.95	61.50	60.96	62.87	56.08
ΣP 8 h	75.13	73.35	65.97	77.04	68.34	76.49	68.43	72.67	73.99	76.54	72.85	68.43

Table 5. The adsorption capacity (q_e) of COD, TSS, total N (ΣN) and total P (ΣP) by 12 biochar samples

	q_e (mg/g)											
	CF1	CF2	CF3	CF4	CF5	CF6	CF7	CF8	CF9	CF10	CF11	CF12
COD 1 h	109	122	108	157.5	110	126	114	132	108	120	128	92
COD 2 h	166.5	165	168	171.62	156	168	166.5	156	167	171	166.5	121.25
COD 4 h	184	181.5	179	184	166.5	179.5	173	173	177	180.5	181.5	166.5
COD 8 h	184	186	186	191.2	181.5	185	181.5	185	183	189	190.5	181.5
TSS 1 h	6.05	5.875	5.85	5.6	4.575	5.825	5.25	5.95	4.875	5.075	6.1	4.3
TSS 2 h	4.925	5.05	4.75	5.35	5.3	4.8	6.1	6.05	5.825	6.325	6.25	5.85
TSS 4 h	6.1	5.875	5.825	5.675	6.15	6.225	6.15	6	6.075	6.05	5.975	6.05
TSS 8 h	6.125	6.3	6.4	6.25	6.4	6.325	6.2	6.175	6	6.375	6.45	6.1
ΣN 1 h	3.764	4.564	3.492	6.761	4.185	6.421	4.537	4.990	4.049	3.920	6.419	3.497
ΣN 2 h	7.214	5.745	6.515	8.747	6.011	8.449	8.922	8.264	6.669	8.516	8.264	5.994
ΣN 4 h	9.540	9.537	9.196	11.266	9.638	8.996	9.638	8.720	8.922	8.917	9.539	8.764
ΣN 8 h	9.741	10.012	10.193	12.725	10.284	9.712	10.193	9.747	9.791	9.537	11.239	9.449
ΣP 1 h	0.110	0.127	0.098	0.285	0.098	0.130	0.087	0.128	0.154	0.133	0.262	0.085
ΣP 2 h	0.158	0.208	0.158	0.320	0.130	0.310	0.310	0.179	0.287	0.302	0.288	0.155
ΣP 4 h	0.344	0.340	0.343	0.337	0.339	0.336	0.334	0.318	0.338	0.335	0.345	0.308
ΣP 8 h	0.412	0.403	0.362	0.423	0.375	0.420	0.376	0.399	0.406	0.420	0.400	0.376

for the adsorption capacity of biochar by Cui et al. [33] and Hirata et al. (2002) [13, 33]. According to Cui et al. [33], the surface functional groups such as O-H, C-H, C=O, C-C, etc. on the biochar material have a strong influence on ion adsorption capacity and therefore enhance their adsorption efficiency [33]. Hirata et al. (2002) indicated that the surface area contains many functional groups such as O-H and C-O, and a specific surface area of less than 1 m^2/g will easily remove pollutants [13] due to the polarized surface and the large base consumption level of these materials. The fact that the C content is over 80% in all biochar samples shows that the adsorption mechanisms are based on the amounts of organic pollutants adsorbed and the properties of the carbonaceous material.

3.3 Effect of Adsorbent Material Mass

The effect of reaction time on pollutant removal was investigated for four time values from 1 h to 8 h. The results showed that the adsorption capacity of material increased with longer reaction times from 1 h to 8 h. At a reaction time of 8 h, the highest efficiency for pollutant removal was achieved. Thus, 8 h was chosen as the optimum reaction time and used in the subsequent experiments. The initial livestock wastewater was adsorbed

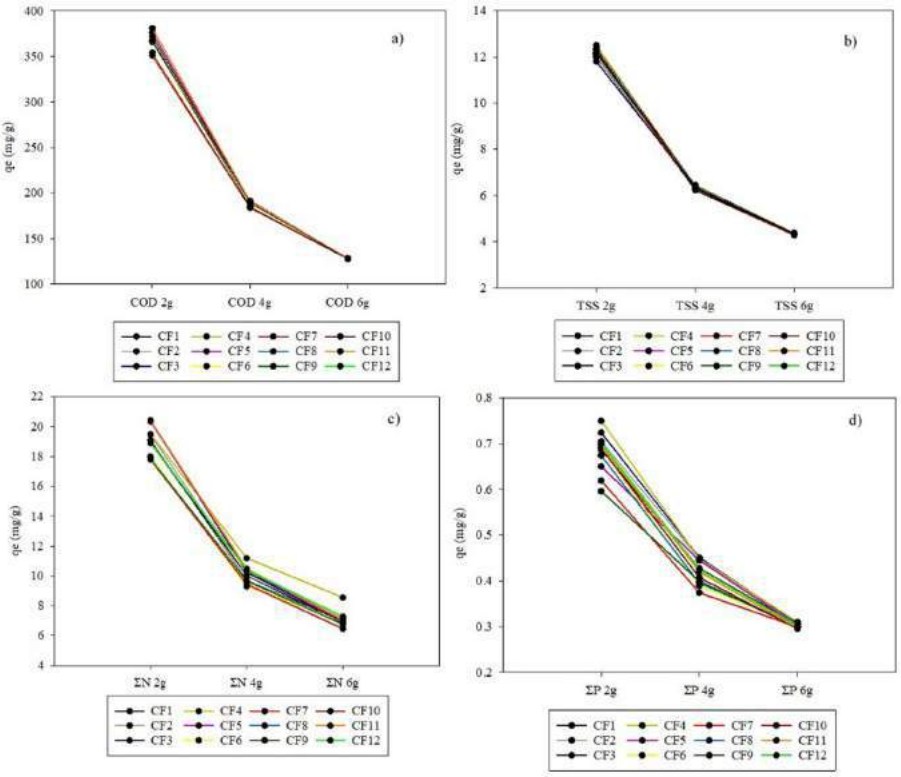

Fig. 9. Changes in adsorption capacity of (a) COD, (b) TSS, (c) total N and (d) total P parameters adsorbed with biochar at the adsorbent material masses of 2, 4 and 6 g.

Table 6. The adsorption efficiency (H%) of COD, TSS, total N (ΣN) and total P (ΣP) by 12 biochar samples

	H (%)											
	CF1	CF2	CF3	CF4	CF5	CF6	CF7	CF8	CF9	CF10	CF11	CF12
COD 2 g	94.36	95.38	95.38	94.36	96.41	93.85	90.00	94.36	90.77	95.38	97.69	93.85
COD 4 g	98.21	97.95	97.69	97.13	97.95	94.87	94.36	94.87	94.62	96.92	97.95	97.18
COD 6 g	98.36	98.36	98.36	98.77	98.72	98.36	98.46	98.18	98.59	98.67	98.56	98.36
TSS 2 g	90.74	89.63	87.41	92.59	90.74	90.00	91.11	91.48	88.89	90.74	91.85	90.37
TSS 4 g	96.67	95.19	96.67	97.04	95.93	96.30	95.19	96.30	95.56	96.30	96.67	96.30
TSS 6 g	96.67	95.19	96.67	97.04	95.93	96.30	95.19	96.30	95.56	96.30	96.67	96.30
ΣN 2 g	69.35	71.17	74.63	71.36	74.48	65.87	65.14	69.84	65.32	69.82	74.79	69.18
ΣN 4 g	73.30	73.30	74.97	81.92	76.03	68.06	69.13	73.30	70.71	71.11	76.66	76.05
ΣN 6 g	74.95	76.40	76.19	93.84	76.26	76.43	70.91	76.19	74.26	76.28	77.54	79.78
ΣP 2 g	62.60	64.24	65.97	68.34	59.23	62.82	56.40	61.37	54.26	62.87	63.37	63.87
ΣP 4 g	77.72	81.69	82.19	81.59	81.09	70.80	68.11	71.98	72.76	74.17	76.49	77.36
ΣP 6 g	83.19	82.82	83.92	84.65	82.28	82.92	81.82	82.78	81.50	80.59	81.05	82.23

onto biochar masses of 2, 4 or 6 g over 8 h. The experimental results are shown in Fig. 9 and in Tables 6 and 7. As with reaction time, the solid/solution ratio in a batch sorption process is an important parameter determining the capacity of adsorption of pollutants. The results of this study showed that when the adsorbent material weight was increased from 2 g to 6 g (weight/volume) with a reaction time of 8 h, the adsorption capacity/efficiency for pollutants (COD, TSS, total N and total P) increased accordingly.

With different adsorbent masses (from 2 g to 6 g) and after a reaction time of 8 h, the adsorption capacity with all four parameters of the 12 biochar samples were significantly reduced (Fig. 9). The maximum reduction efficiency was found for the adsorbent mass of 6 g, with lower reduction efficiency for 4 g and lowest for 2 g. Unlike the results for different reaction times, adsorption with 6 g of material for all 12 biochar samples produced water with concentrations lower than the wastewater limit values for all four

parameters. The highest COD adsorption capacity was found for 2 g of the CF11 biochar sample (381 mg/g), and the lowest for 6 g of the CF8 sample (127.63 mg/g). As seen for the COD parameter, the adsorption capacity of TSS content was highest for 2 g of CF4 material (12.5 mg/g) and lowest for 6 g of adsorbent material (4.333 mg/g). The total N and P parameters showed maximum adsorption capacities of 20.429 mg/g (CF11) and 0.75 mg/g (CF4) on 2 g of biochar, and minimum adsorption capacities of 6.456 mg/g (CF7) and 0.295 mg/g (CF10) on 6 g of biochar, respectively.

The analysis results showed that the removal of these pollutants increased as the weight of the adsorbent increased. This result is consistent with some previous results obtained for the adsorption capacity of biochar with different substrates, such as pesticides, heavy metal, textile dye, ions (NH_4^+, PO_4^{3-}), etc. [4, 18, 20, 35]. Zheng et al. (2010) assessed the absorption ability for two pesticides (triazine–atrazine and simazine) by unmodified biochar. The analysis parameters included contact time, solution pH, particle size and solid/solution ratio [4]. The authors showed that adsorption affinity for the two pesticides increased from 451 to 1158 mg/kg and from 243 to 1066 mg/kg, respectively, when the solid/solution ratio decreased from 1:50 to 1:1000 (g/mL) [4]. The results obtained by Yao et al. [20] showed that nanoscale MgO (periclase) particles provided the main adsorption sites on biochar surfaces for phosphate in solution [20]. Vu et al. (2017) used a modified biochar material synthesized from corn cobs to remove ammonium from synthetic water in which the ammonium concentration varied from 10 to 100 mg/L. The research results showed that when the weight/volume ratio decreased from 1:1 to 1:5, the adsorption capacity for ammonium increased accordingly [35]. The sorption capacity of Hg(II) in water by two biochar samples synthesized from bagasse and hickory chips was assessed by Xu et al. [18]. The results showed that the sorption of Hg(II) metal can be conducted by functional group complexation on the biochar surface, ion exchange adsorption or co-precipitation with mineral [18].

Furthermore, the results of SEM images and FTIR spectra in this study also indicated that carbonaceous materials have many carboxyl and phenolic hydroxyl groups on the surface area combined with a porous structure. This property leads to a high organic pollutant adsorption capacity, influenced by the interaction between pollutants and the surface or pores of carbon materials or by interactions with functional groups [4]. Therefore, the different sorption mechanisms occurred on carbonized and non-carbonized phases of biochar [37, 38]. These results in currently study showed that the CF4 biochar sample is the highest adsorption capacity with all 4 pollutant parameters. These results of the current study showed that the CF4 biochar sample has the highest adsorption capacity for all four pollutant parameters. There is a high potential that biochar material pyrolysed from coffee grounds could become a suitable activated carbon for removal of organic pollution from livestock wastewater. However, it is necessary to fully analyze other characteristics of this material such as TEM (Transmission Electron Microscopy), sorption isotherms, the effect of particle size on sorption kinetics, etc. to find out the optimal pyrolysis conditions to produce biochar material. In addition, reaction time and the biochar mass also need to be fully studied to find out the optimal experimental conditions in which the adsorption efficiency is maximized.

Table 7. The adsorption capacity (q_e) of COD, TSS, total N (ΣN) and total P (ΣP) by 12 biochar samples

	q_e (mg/g)											
	CF1	CF2	CF3	CF4	CF5	CF6	CF7	CF8	CF9	CF10	CF11	CF12
COD 2 g	368	372	372	368	376	366	351	368	354	372	381	366
COD 4 g	191.5	191	190.5	189.4	191	185	184	185	184.5	189	191	189.5
COD 6 g	127.87	127.87	127.87	128.4	128.33	127.87	128	127.63	128.2	128.27	128.13	127.87
TSS 2 g	12.25	12.1	11.8	12.5	12.25	12.15	12.3	12.35	12	12.25	12.4	12.2
TSS 4 g	6.425	6.325	6.375	6.4	6.3	6.325	6.225	6.25	6.225	6.375	6.45	6.375
TSS 6 g	4.35	4.283	4.35	4.367	4.317	4.333	4.283	4.333	4.3	4.333	4.35	4.333
ΣN 2 g	18.945	19.442	20.386	19.493	20.345	17.993	17.793	19.079	17.843	19.073	20.429	18.898
ΣN 4 g	10.012	10.012	10.239	11.188	10.384	9.295	9.442	10.011	9.657	9.712	10.470	10.387
ΣN 6 g	6.824	6.956	6.937	8.544	6.944	6.959	6.456	6.938	6.761	6.945	7.061	7.264
ΣP 2 g	0.687	0.705	0.724	0.750	0.650	0.690	0.619	0.674	0.596	0.690	0.696	0.701
ΣP 4 g	0.427	0.448	0.451	0.448	0.445	0.389	0.374	0.395	0.399	0.407	0.420	0.425
ΣP 6 g	0.304	0.303	0.307	0.310	0.301	0.303	0.299	0.303	0.298	0.295	0.297	0.301

4 Conclusion

The results of this study showed that when increasing the reaction time and adsorbent mass, the adsorption efficiency for COD, TSS, total N and total P increased accordingly. The greatest removal of pollutants was achieved at a reaction time 8 h with adsorbed biochar weight of 6 g. It can be clearly seen that the CF4 sample performed the best in removing contaminated substances from wastewater. At a reaction time of 8 h with a biochar mass of 4 g, only the COD parameter as adsorbed by the CF4 biochar sample met the output requirements following the Vietnam national regulation QCVN 40:2011/MONRE on industrial wastewater; the other 11 wastewater samples had concentrations 1.6 to 3.6 times higher than the standard limit values. Three remaining parameters including TSS, total N and total P exhibited different biochar adsorption capacities at different reaction times: the TSS parameter met the standard limit requirements in all samples, total N was 3.3 to 4.2 times higher than its limit (except for the CF4-treated sample) and total P was 1.07 to 1.15 times higher than the standard limit values (except for samples treated with the CF4, CF8, CF9 and CF11 biochars). When an adsorbent mass of 6 g and a reaction time of 8 h were used, all four parameters adsorbed with 12 biochar samples were significantly reduced in concentration, and the output values were all lower than the standard limits. Therefore, this application can be extended to treating other types of wastewater. The results showed that the biochar from spent coffee grounds is a potential sorbent to remove pollutants from livestock wastewater. However, further studies on the optimal temperature conditions to produce biochar material, desorption/adsorption, etc., need to be fully conducted before application.

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