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Coffee Ground-Based Modified Biochar for Effective Treatment of Nutrient-Rich Swine Wastewater

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Abstract: Swine wastewater contains nutrient materials such as Ammonium (NH_4^+), Phosphate (PO_4^{3-}), Total Kjeldahl Nitrogen (TKN), and Total Phosphorus (TP) which can result in eutrophication and disrupt the surrounding environment. So, this study aims to develop a nutrient-adsorbing material from spent coffee grounds for the treatment of swine wastewater. The adsorbent was produced through anaerobic pyrolysis of coffee grounds impregnated with various concentrations of $MgCl_2$ under different pyrolysis durations (30, 60, 90, 120, 150, and 180 minutes) and temperatures (500°C, 550°C, 600°C, 650°C, and 700°C). Subsequent experiments were carried out to determine the optimal conditions for the adsorbent in treating swine wastewater. The preliminary results were then used to test real-world scenarios using coffee-ground biochar in wastewater treatment. The results indicated that the M 2/650/30 sample—prepared with 2% $MgCl_2$, pyrolyzed at 650°C for 30 minutes—was the most effective adsorbent. In real-scenario testing, this sample achieved an ammonium removal efficiency of 66.53% and a phosphate removal efficiency of 88.82%. Moreover, the adsorbent was also capable of adsorbing other nitrogen and phosphorus forms, as evidenced by its higher adsorption capacities for TKN and TP compared to NH_4^+ and PO_4^{3-}. The material modifications significantly enhanced nutrient recovery from wastewater, such as ammonium and phosphate, while simultaneously reducing the environmental emissions associated with coffee waste. This study demonstrates the potential of spent coffee grounds as an effective adsorbent for treating swine wastewater. The findings may have important implications for the management of livestock agricultural waste, which remains an urgent environmental issue.

Keyword: ammonium ion; phosphate ion; adsorption; livestock wastewater; biochar; coffee ground.

1. Introduction

Pig farming in Vietnam is the most prevalent form of livestock farming, with a whopping 7475 pig farms (making up 42.2% of all livestock farms). The North has 3069 farms, which is 41.1% of the total, while the South has 4,406 farms, which is 58.9%. Over the past three years, pig farming has been rapidly expanding due to the positive

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correlation between profit and the number of pigs raised. Most sow farms (71.3%) have a scale of 20-50 pigs, while the majority of pig farms for meat (75.5%) have a scale of 100-200 pigs[1)]. Livestock wastewater treatment has long been a pressing environmental issue. The wastewater generated from livestock farming is typically nutrient-rich, with high concentrations of suspended solids, nitrogenous organic compounds, and harmful coliforms and bacteria[2,3)]. Swine wastewater poses a significant environmental pollution risk as it contains high levels of organic substances, suspended solids, nitrogen (N), phosphorus (P), and pathogenic microorganisms. About 70-80% of the solids separated from wastewater are organic components, including hydrocarbon compounds, proxies, amino acids, fats, and their derivatives from feces and leftovers. The wastewater also has relatively high levels of nitrogen and phosphorus due to their presence in food and excretion in feces and urine[4)]. So, when nutrients and organic matter from swine wastewater are untreated and accumulate, these nutrients can cause eutrophication of surface water bodies[5)]. Despite the widespread adoption of small-scale household breeding models, many owners of these operations only provide preliminary treatment to their livestock wastewater, using biogas or directly discharging it into the environment. This practice contributes to the worsening of the environmental problem caused by the livestock industry. To address this issue, it is essential to implement effective wastewater treatment methods that can remove contaminants and reduce the environmental impact of livestock farming. However, there is still a need to improve these methods and develop innovative approaches that can reduce the cost and energy consumption associated with treatment processes while maintaining high treatment efficiency[6)]. Wastewater treatment with medium adsorption technology from natural materials is a solution that still exists due to being considered cheaper and environmentally friendly.

Not only wastewater from pig farms, another waste problem that is urgent is sludge waste in Vietnam. Vietnam's rapid industrialization and modernization have led to the development of a modern urban system, including the construction of industrial parks and economic zones. However, this growth has also resulted in increased production and consumer demand for goods, raw materials, and energy, which in turn has led to environmental concerns, particularly with regards to waste management and water scarcity[7)]. One of the biggest challenges facing many developing countries, including Vietnam, is the collection, transportation, treatment, recycling, and reuse of waste, especially sludge from urban drainage systems and sanitation facilities. Despite this pressing issue, there is currently a lack of synthesis, analysis, and assessment of the volume, collection, and transportation of sludge, as well as a lack of forecasting for the volume of sludge generated from drainage systems and inadequate sanitation works[8)]. Furthermore, there is a significant lack of planning when it comes to the location, scale, and placement of sewage sludge treatment facilities, and when they are included in plans, they are often not combined with solid waste treatment. The lack of planning for dumping, discharging, and treatment areas creates further challenges in the implementation of effective waste management practices. To address these issues, there is a need for comprehensive planning, utilizing appropriate treatment technologies, and proper management of sludge and other waste to ensure the sustainable development of Vietnam's economy and urban systems[9)].

Adsorbents used are generally diverse, ranging from carbon materials and metal-non-metal oxidizers[5)]. Biochar has a lot of carbon content in it which is created from the thermal degradation of biomass[10)]. Biochar has unique psycho-chemical characteristics, namely a porous and three-dimensional networked structure that has high carbon storage potential for maximum pollutant absorption efforts[11)]. Biochar is produced through a heating process without the presence of oxygen or less oxygen or known as pyrolysis process[12)]. Pyrolysis is a process of thermal decomposition of organic material, which can be categorized into fast, medium, and slow pyrolysis based on the temperature and time taken. The slow and medium pyrolysis processes typically take from a few minutes to a few hours, or even a few days, with the highest solids recovery, producing about 12-30% of biochar[13)]. On the other hand, fast pyrolysis primarily produces fuel oil (75%). Therefore, slow, and medium pyrolysis are typically used for biochar production. The main products of medium and slow pyrolysis are solids, mainly carbon, which have a higher calorific value than the initial state[14)]. Additionally, syngas is produced as a mixture of gases including carbon monoxide, hydrogen, methane, and various other volatile organic compounds. In recent years, there has been a surge in research on using various chemical agents to modify biochar, fueled by advancements in science and technology[15)]. Some of the most promising approaches include using organic solvents, surfactants, and coating the surface of biochar with metal oxides, carbon nanotubes, or graphene. These techniques have the potential to enhance biochar's properties, such as its porosity, surface area, and adsorption capacity, making it an even more effective tool for a wide range of applications, such as soil remediation, water treatment, and energy storage. By leveraging the unique properties of these chemical agents, researchers are working to unlock the full potential of biochar and help create a more sustainable future[15)]. Some research about using swine wastewater treatment such as Le Quoc Vi et al., about biogas for household pig farm wastewater[16)], combination of biological lake (*Eichhornia crassipes*) and biochar of Nguyen Lich et al.[17)] biochar from bamboo for ammonium removal[18)], coffee ground for heavy metal[19)], $HCl/FeCl_3$/

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wheat straw for nitrate and phosphate removal[20], etc.

Vietnam was an agricultural country so there are a lot of crop residues such as straw, bagasse, rice husk, corn stalk, cob, etc.[21]. For a long time, these waste products have been considered by farmers in the Mekong Delta as waste products, often burned or thrown indiscriminately into rivers, canals, or residential areas. This method is both wasteful and pollutes the environment. Coffee is a beloved beverage in many countries, with its stimulating effect on the nervous system, stress-reducing properties, and ability to combat aging. It's estimated that in 2017-2018, the world produced approximately 9.5 billion tons of coffee[22]. However, with such high levels of coffee consumption comes an equally high amount of waste. Every day, around the world, approximately 6.6 million tons of coffee are consumed and subsequently discarded as waste[22]. Vietnam boasts 730,000 hectares of land devoted to coffee cultivation, making it the world's second-largest coffee exporter[23]. However, a large amount of coffee grounds is generated as waste every day, potentially posing environmental and economic challenges, particularly in terms of coffee process waste (CPW) management. Approximately 90% of the coffee fruit biomass is converted into waste during the processing stage, such as pulp, mucilage, husk, or chaff, depending on the processing method used[24,25].

Coffee grounds waste has been widely sought to be reused because of its massive quantity and still has a relatively high content of sugar, antioxidants, oils, and other contents[26]. The use of coffee grounds is very diverse, ranging from making organic fertilizers, biofuels/biodiesel, supplements, briquettes, to pollutant degradation materials[27]. Research related to the use of spent coffee ground (SCG) as biodiesel provides high efficiency results. Judging from Wang's (2024) research, the highest biodiesel production efficiency from coffee grounds reaches 95.24% using pomegranate/orange peel-derived activated carbon (POPAC) green nanocatalysts, this result also allows for short retention time[28]. Another study related to the role of SCG in its use is its use as a compost and fertilizer medium. SCG added directly to broccoli, radish, leek, and sunflower plants experienced poor growth due to its high carbon to nitrogen ratio (C/N) content, acidity level, and phenolic levels. However, in some cases, the addition of SCG and its combination with several other materials can improve soil quality and crop yields. Relatively low levels of SCG in soil can increase levels of antioxidants, mineral content, and bioactive compounds [27,29]. Meanwhile, in its use as a pollutant degradation material, SCG is widely developed as biochar by adsorption method. Pollutants that can be adsorbed by SCG are metal content in water, such as chromium, lead, lithium, etc. [30–32]. Lead (Pb^{2+}) removal in an aqueous solution with coffee grounds biochar made by hydrothermal synthesis reached an adsorption capacity of 321.90 mg/g[32], while another study on Lithium (Li^+) metal removal in an aqueous solution with H_3PO_4 activated coffee grounds biochar had the highest adsorption capacity of 38.10 mg/g after 6 hours of contact time, and can remove up to 95% of Lithium[30].

Based on several problems that are of interest in this study, this research aim is to solve three waste problems in Vietnam: (i) swine wastewater treatment (ii) sludge management issue (iii) and coffee ground issue. The obvious gap of the problem is the lack of waste treatment capabilities in Vietnam with such a large quantity of waste. By creating materials capable of adsorbing applications in wastewater treatment, this research is an effective solution in the waste utilization cycle for waste treatment with biochar from combination of sludge from water treatment plant and coffee ground for ammonium and phosphate removal.

2. Materials and Methods

2.1. Collection of coffee ground waste and biochar production

The coffee ground was collected about 2 kg wet ones (each times) from Thu Duc district, Viet Nam. Coffee ground was fine, humidity and impurity so it had to be sieved (≤ 2 mm) and dry at 80°C for 24h before modification. The sludge from Tan Phu ground water treatment plant Ho Chi Minh city, Viet Nam was crushed, sieved ≤ 2 mm and dried at 80°C till it evaporated.

Table 1 presents the combination of coffee ground. The combination of coffee ground and sludge was prepared as following steps[33]. 80 mL $MgCl_2$ X % (0%, 0.5%, 1%, 2%, 3%, 4%, 5%, 10%, 15%, 20%, 25%, 30%) with 2 g sludge and 2 g coffee ground (or 4 g coffee ground alone) was shaken at 120 rpm for 24 hours, and then dried it at 80°C till evaporated. The mixture was dried again at 105°C for 6 hours and heated at Y°C (500°C, 550°C, 600°C, 650°C, 700°C) in Z minutes (30, 60, 90, 120, 150, and 180 minutes) and conserved in a zipper. The samples were sign as follow M X%/Y °C/Z minutes (M 0.5/500/30 with 0.5% $MgCl_2$ pyrolysis at 500°C in 30 minutes).

2.2. Characterization of coffee ground waste and biochar

The chemical included in this study was purity (< 99%) and dissolved with distilled water. The chemical for modification and synthesis wastewater consisted of $MgCl_2$. The urgency of using $MgCl_2$ (magnesium chloride) as a biochar modification material is its benefit to optimize its ability to absorb various pollutants, especially phosphates, by expanding the surface area, adding positive charges, and encouraging the formation of magnesium compounds[34,35]. The Scanning Electron Microscope (SEM) is an advanced type of electron microscope used to observe a sample's surface by scanning it with a highly

Table 1: Sample naming and variables

No. of Sample	Name of Sample	Variable		
		X (%)	Y (°C)	Z (minutes)
1	M 0,5/500/30	0.5	500	30
2	M 0,5/500/60	0.5	500	60

X: concentration of $MgCl_2$, (%), Y: temperature of pyrolysis, (°C), Z: time of pyrolysis, (minutes)

focused electron beam in a vacuum. In addition to observing the sample's structure, the SEM is also capable of identifying and quantifying elements. To analyze solid-body microstructures, Energy Dispersive X-ray Spectroscopy (EDX), also known as EDS, is frequently utilized in electron microscopy. This technique utilizes high-energy interactions with solid objects to obtain images. X-ray Diffraction Analysis (XRD) is another analytical method used in this study. XRD, performed using the D2 Phaser by Bruker in Germany, is a powerful technique used to determine the crystallinity and phase composition of solids and materials.

2.3. Experimental analyzation

The synthesis swine wastewater was same as the real swine wastewater which collected at household farm in District 12, Ho Chi Minh city. The concentration of synthesis wastewater was about 120 mg P – PO_4^{3-}/ L and 100 mg N-NH_4^+/L. Weight 0.1 g biochar M X%/Y °C/Z minutes into an 250 mL Erlenmeyer with 40 mL synthesis wastewater or real wastewater. The mixture was shaken for 14, 16, 18, 20, 22, 24 hours. Then the mixture was filter through the paper (Newstar θ 100 μm), and analyzed NH_4^+ (Buchi distillation unit K-355) and PO_4^{3-} with (DR 6000 EDU). Each experiment was replicated 5 times for ensuring the accuracy. The swine wastewater of this study had the detailed elements as shown in Table 2.

The removal efficiency of ammonium ion and phosphate ion was calculated as the equation:

$$H\ (\%) = ((C_o - C)/C_o) * 100 \qquad (1)$$

Where H (%): removal efficiency of adsorbate removal, C_o: inlet concentration of adsorbate removal (mg/L), C: concentration at equilibrium times of adsorbate (mg/L).

The adsorption capacity of ammonium ion and phosphate ion was calculated as the equation:

Table 2: The properties of swine wastewater (on December n = 5)

No	Name	Units	Value	Average
1	pH	-	7.00 – 8.00	7.50
2	COD	mg/L	261.00 – 264.00	262.50
3	SS	mg/L	151.00 – 162.00	157.00
4	TKN	mg/L	381.44 – 406.40	395.95
5	N-NH_4^+	mg/L	168.98 – 176.64	173.23
6	TP	mg/L	158.22 – 175.48	165.36
7	P-PO_4^{3-}	mg/L	140.97 – 149.00	144.69

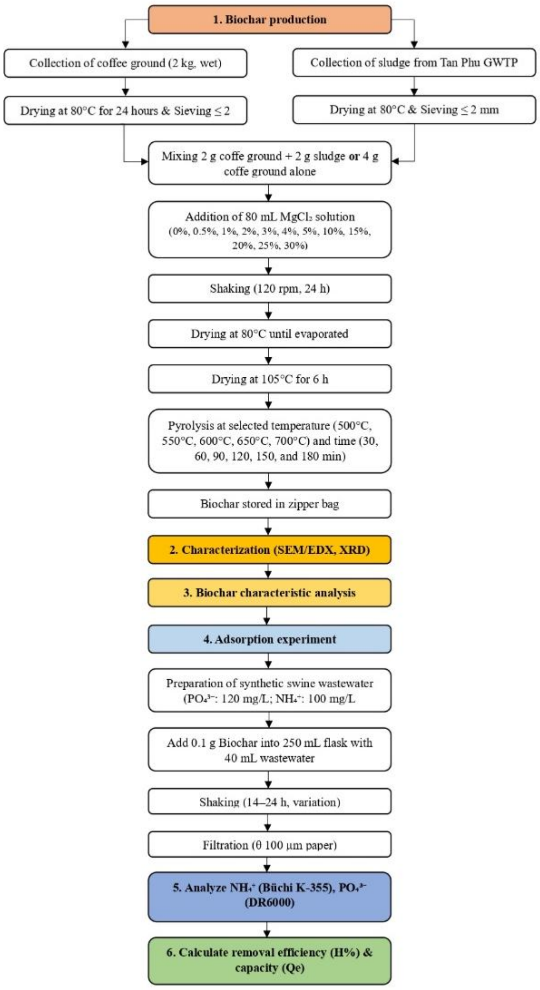


Fig. 1: Research method flowchart

$$Q_e\ (mg/g) = ((C_o - C)/m) *V \qquad (2)$$

Where Q_e (mg/g): adsorption capacity of ammonium and phosphate ion, C_o: inlet concentration of adsorbate (mg/L), C: concentration at equilibrium times of adsorbate (mg/L), m: mass of adsorbent (mg/g), V: volume of sample (L).

Overall, the method of this research is shown in the following Figure 1.

3. Results and Discussion

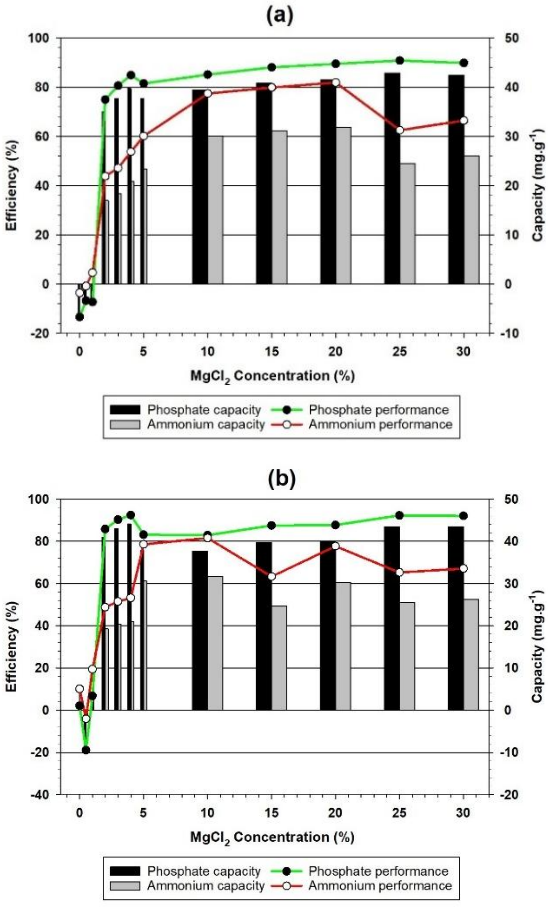
3.1. Influences of factors on coffee ground biochar (synthesis wastewater)

3.1.1. The effect of concentration of $MgCl_2$ (%) on the ammonium and phosphate ion adsorption

Figure 2 demonstrated the effect of % $MgCl_2$ to removal efficiency and adsorption capacity of ammonium ion and phosphate ion in various pyrolysis temperatures. As can be seen the results in Figure 2(a) at the point of 2% $MgCl_2$ had the break point in both adsorption capacity and removal efficiency of ammonium ion and phosphate ion. However, at temperature from 550°C to 700°C (Figure 2(b-e)) the break point was from 1% $MgCl_2$. For the ammonium ion, biochar M 20/500/30 had the highest removal efficiency 81.87% with adsorption capacity of 31.86 mg/g. Among the samples tested, only the $P\text{-}PO_4^{3-}$ sample impregnated with M 0.5/550/30 did not undergo the adsorption process. Samples that were not modified with $MgCl_2$ and those impregnated with 1% $MgCl_2$ showed a very low treatment efficiency. However, samples impregnated with 2% to 30% $MgCl_2$ exhibited a significant increase in efficiency, ranging from 85.81% to 92.40%. The corresponding adsorption capacity increased from 40.93 mg/g to 44.07 mg/g. Notably, the material sample M 4/550/30 had the highest adsorption efficiency of 92.40% and an adsorption capacity of 44.07 mg/g.

Out of all the samples tested for $N\text{-}NH_4^+$, only sample M 0.5/550/30 did not participate in the adsorption process. However, for the remaining samples (from M 1/550/30 to M 30/550/30), the removal efficiency of NH_4^+ ion increased from 19.26% to 81.58%. This raise in efficiency corresponded to an adsorption capacity ranging from 7.67 mg/g to 31.75 mg/g. The material sample M 10/550/30 exhibited the highest adsorption efficiency of 81.58%, corresponding to an adsorption capacity of 31.75 mg/g.

The research of Li et al., displayed that the enlarged of Mg% content in biochar from 0.44% to 23.07% maintained the adsorption capacity of ammonium at 20 mg NH_4^+/g [6]. Research by Otieno et al. (2021) revealed that when the ammonium concentration is low, the ion flux density is also low, so the adsorption capacity is not maximized. Conversely, when the ammonium concentration is high, the flux ion density will increase as well as the adsorption power on the biochar [36]. On the other hand, with the raise in MgO content in material, the phosphate efficiency increased significantly 660% [6].



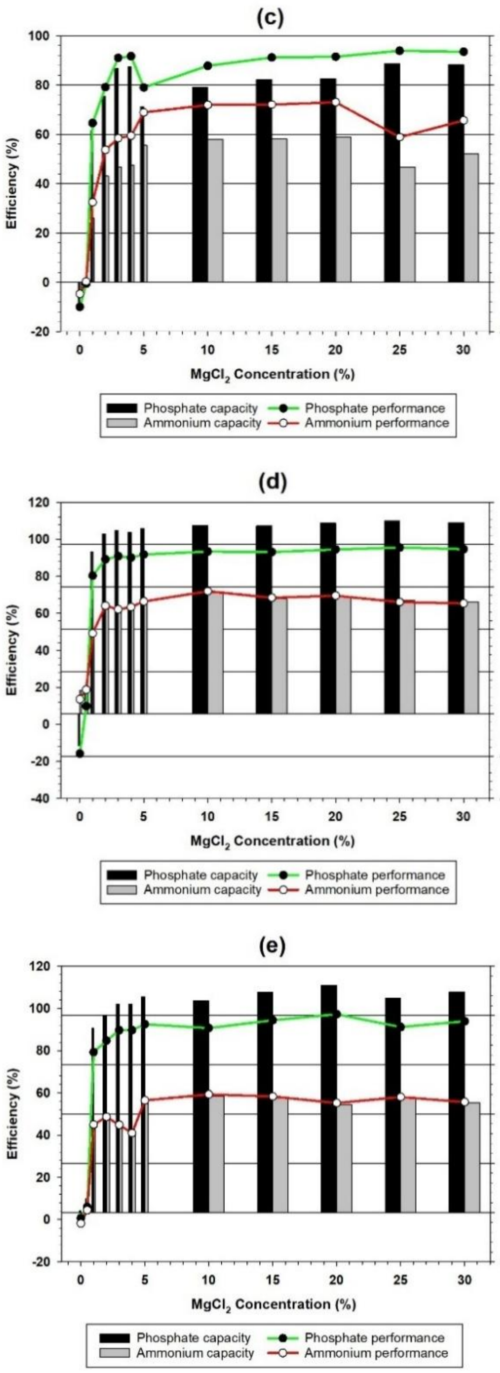


Fig. 2: The effect of concentration of $MgCl_2$ (%) on the ammonium and phosphate ion adsorption with different pyrolysis temperature (a) 500°C, (b) 550°C, (c) 600°C, (d) 650°C, (e) 700°C

The efficiency showed a significant improvement from 80.37% in impregnated sample 1/650/30 to 95.59% in M 30/650/30, with corresponding adsorption capacity rising from 38.29 mg/g to 45.60 mg/g. Among all the material samples, M 25/650/30 exhibited the highest adsorption performance of 95.59%, with an adsorption capacity of 45.60 mg/g. For N-NH_4^+ criteria, models M 0/650/30 and M 0.5/650/30 exhibited less than 20% efficiency. Model M 1/650/30 showed a significant improvement, achieving an efficiency of 49.22%. From models M 2/650/30 to M 30/650/30, the trend was generally towards an incremental improvement in performance, with a slight increase from 64.11% to 71.97%. Material sample M 10/650/30 exhibited the highest adsorption efficiency of 71.97%, corresponding to an adsorption capacity of 28.62 mg/g. Other research had the same trend as the improvement of Mg% content in material lead to the increase of phosphate adsorption capacity including $MgCl_2$ – brown marine macroalgae[37)], Mg – diatomite[38)].

The adsorption of phosphate was based on the surface area of biochar. As the research of Jung et al., the pyrolysis temperature from 200°C to 400°C, the growth in phosphate removal was significant[37)]. The optimal pyrolysis temperature led to the creation of a larger surface area in biochar as volatile substances were quickly released from the biomass structure, resulting in the formation of numerous pores. However, at extremely high temperatures, the porous structure collapsed, leading to the blockage of pores and the reduction of active sites. Additionally, different types of biomasses exhibited varying surface areas under the same pyrolysis conditions due to their intrinsic nature[12,37)]. Therefore, as can be seen in Table 3 after 650°C the adsorption capacity of both ammonium and phosphate had the same trend.

Based on the findings of adsorption experiments carried out on various material samples using wastewater, it was observed that samples impregnated with a 1% $MgCl_2$ solution often produced low efficiency and some of them even released PO_4^{3-} and NH_4^+. In contrast, solutions impregnated with $MgCl_2$ at a concentration of 2% resulted in a significant enhancement in efficiency. The efficiency of the material samples impregnated with $MgCl_2$ solutions at concentrations ranging from 2% to 30% displayed a general upward trend, but there were also points on the yield graph where the efficiency marginally declined. Furthermore, the material sample subjected to pyrolysis at a temperature of 650°C demonstrated the highest removal efficiency and adsorption capacity which was 42.50 mg P-PO_4^{3-}/g and 25.31 mg N-NH_4^{3-}/g (Table 3). Hence, to strike a balance between economic benefits and performance, the M 2/650/30 material was opted for further experimentation.

Table 3: The adsorption capacity of ammonium ion and phosphate ion (mg/g) with various temperature and 2% $MgCl_2$ coffee ground biochar

Pyrolysis temp.	Ammonium ion		Phosphate ion	
	Sample	Q_e (mg/g)	Sample	Q_e (mg/g)
500	M 2/500/30	16.97	M 2/500/30	35.08
550	M 2/550/30	19.26	M 2/550/30	40.93
600	M 2/600/30	21.50	M 2/600/30	37.67
650	M 2/650/30	25.31	M 2/650/30	42.50
700	M 2/700/30	19.54	M 2/700/30	39.96

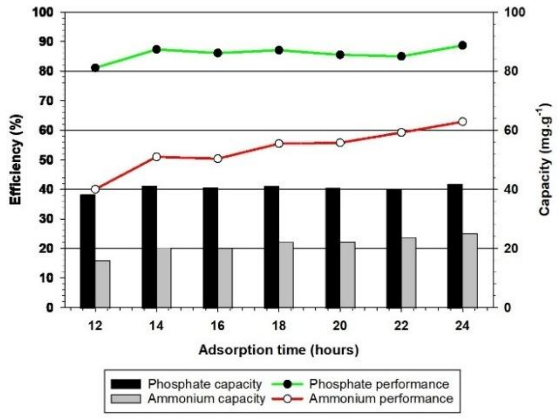


Fig. 3: The influence of contacting time on ammonium ion and phosphate ion adsorption capacity and removal efficiency of M 2/650/30 coffee ground

3.1.2. The influence of contacting time on the ammonium and phosphate ion adsorption

Figure 3 demonstrated the effect of contacting time of M 2/650/30 coffee ground biochar. An experiment was conducted on material sample M 2/650 to test its adsorption efficiency and capacity at different time intervals ranging from 12 to 24 hours. Samples were taken every 2 hours during the experiment. The results showed that the longer the adsorption time, the higher the efficiency and capacity. Specifically, the sample that was adsorbed for 14 hours was only slightly lower than the 24-hour sample for PO_4^{3-} and NH_4^+. However, it could save 10 hours of adsorption time. As a result, we selected 14 hours as the optimal adsorption time.

3.1.3. The influence of pyrolysis time on the ammonium and phosphate ion adsorption

This Figure 4 described an experiment conducted to find the optimal pyrolysis time for a material fabricated at 650°C. The material was produced with varying pyrolysis times ranging from 30 to 180 minutes, and then tested for its adsorption capacity in a shaking experiment with wastewater at an adsorption time of 14 hours. The results revealed that the pyrolysis time did not have a significant impact on the adsorption capacity of PO_4^{3-}. However, the

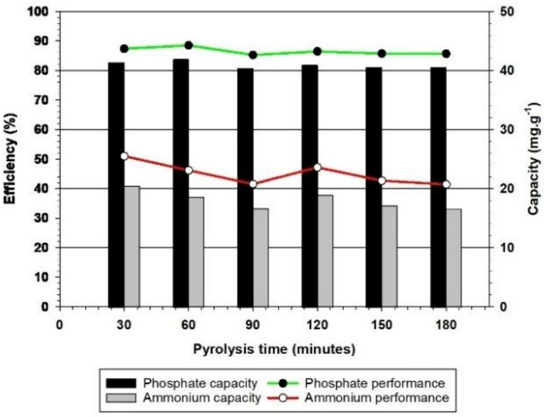


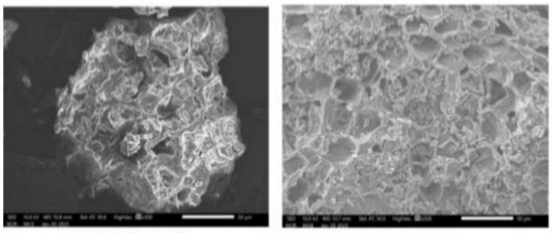
Fig 4: The influence of pyrolysis time on ammonium ion and phosphate ion adsorption capacity and removal efficiency of M 2/650 coffee ground

treatment efficiency for NH_4^+ was highest at 50.97%, with an adsorption capacity of 20.55 mg/g.

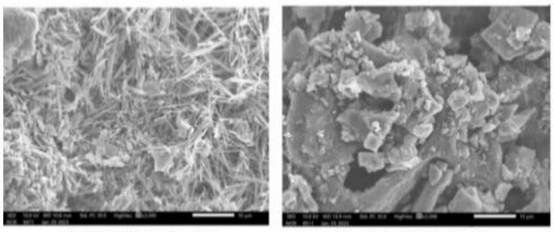
3.2. Biochar characterization

SEM Figure 5(a) revealed the presence of numerous pores on the surface of coffee grounds, ranging in size from 5-25 µm in diameter and exhibiting an unusually smooth yet uneven surface texture. After pyrolysis at 650°C for 30 minutes (without $MgCl_2$ impregnation), Figure 5(b) provided a clearer view of the material's surface, with more uniformly sized pores averaging approximately 25 µm. Remarkably, the resulting M 0/650/30 material exhibited an even more porous structure than the original coffee grounds.

In Figure 5(c), $MgCl_2$ flakes were observed to be evenly distributed on the material's surface or within the pore system following impregnation with a 2% $MgCl_2$ solution.



(a) Coffee grounds (b) M 0/650/30



(c) M 2/650/30 pre-adsorption (d) M 2/650/30 after adsorption

Fig 5: SEM image of (a) coffee ground scaled up to x500 times (b) M0/650/30 coffee ground biochar scaled up to x500 times (c) M 2/650/30 before adsorption scaled up to x2000 times (d) M 2/650/30 after adsorption scaled up to x2000 times

Figure 5(d) showed the capture result of M 2/650/30 material after adsorbing wastewater for 14 hours, revealing the disappearance of previously observed MgO flakes and the appearance of new crystals on the biochar surface, indicating that the biochar acted as a substrate for crystal adhesion. The porous structure detected in the modified biochar may have been caused by the dehydration and decomposition of $MgCl_2.6H_2O$ that occurred during biomass pyrolysis. This process released water and other chemical compounds, resulting in the formation of porous structures within the biochar matrix[39]. Other research related to biochar modification using $FeCl_3$ and $MgCl_2$ found the same fact that modified biochar has a more textured or rougher surface than the original biochar as well as surface expansion and increased pore volume[10].

From results of Table 4 and Figure 6, coffee grounds, biochar, and modified biochar were all carbon-rich materials, with a carbon content of over 50%. M 0/650/30 and M 2/650/30 had a carbon content of over 79% after adsorption. Other elements, including oxygen, magnesium, chlorine, and phosphorus, were also abundant in these materials. The results showed changes in the mass of elements within the material sample, which could be attributed to denaturation caused by chemical reactions and high-temperature pyrolysis. Notably, impregnating the material with the chemical agent $MgCl_2$ resulted in a significant increase in the magnesium and chlorine content. The O/C ratio of M 2/650/30 (0.39) was lower than coffee ground ones, indicating that biochar organic functional group was lost because of high temperature. Moreover, if the O/C and H/C ratio observed in the Mg-modified

Table 4: The percentage chemical element of coffee ground biochar (Weight %)

Eleme-nt	**Coffee ground**	**M 0/650/30**	**M 2/650/30**	
			Before adsorption	After adsorption
C	58.48 ± 0.11	79.43 ± 0.13	55.42 ± 0.13	79.97 ± 0.15
O	40.65 ± 0.2	12.51 ± 0.12	21.91 ± 0.12	12.39 ± 0.13
N	-	-	-	-
Mg	0.23 ± 0.03	0.73 ± 0.03	1.54 ± 0.04	3.16 ± 0.06
Si	-	2.50 ± 0.06	-	-
P	0.54 ± 0.05	0.99 ± 0.05	0.38 ± 0.04	1.17 ± 0.06
Cl	0.10 ± 0.03	1.60 ± 0.06	12.69 ± 0.11	3.79 ± 0.09
S	-	-	0.26 ± 0.02	0.53 ± 0.04
K	-	1.12 ± 0.06	0.22 ± 0.04	-
Ca	-	0.11 ± 0.07	-	1.99 ± 0.09

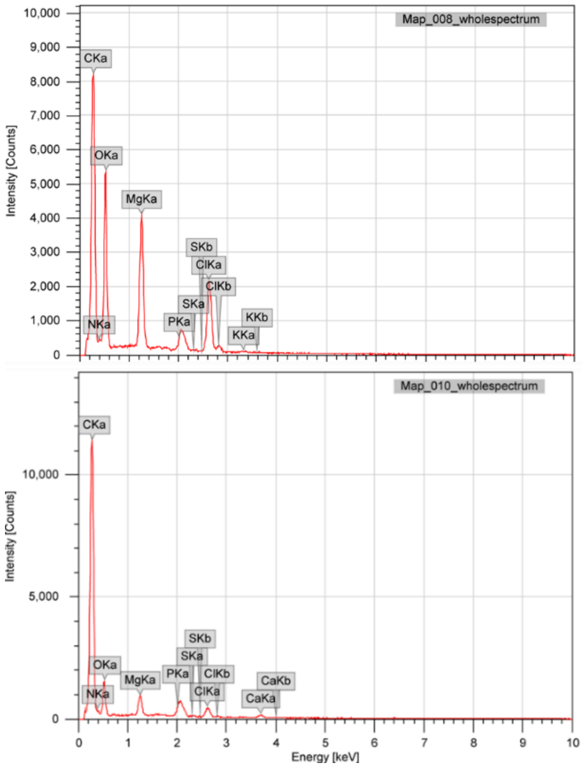


Fig. 6: EDS images before adsorption and after adsorption

biochar samples was greater compared to that in the raw biochar samples. This suggests that the modification of biochar was advantageous in preserving the functional groups present[39]. The removal efficiency of NH_4^+ was affected by the presence of acidic and basic functional group[40]. As the O:C ratio of M 2/650/30 before adsorption was about 0.39 was in the range of combustion residues or biochar ($0.2 < O:C < 0.6$) [41]. Char or charcoal, depending on the black carbon spectrum, is a term used for combustion residues that preserve the structure of the parent biomass and are composed of black carbon. Biochar was situated across the entire spectrum of black carbon due to the range of O:C molar ratios (0-0.6). It should be noted that the position on the black carbon continuum determined the thermal and chemical stability of the biochar, with lower O:C ratios leading to a more stable carbon product [41,42]. The oxygen to carbon of this study was about 0.39 which was revealed the residency time about 100 – 1000 years [43,44].

Figure 7 presented the XRD analysis results of the material samples before and after adsorption. The pre-adsorption sample (B3) displayed distinct MgO peaks at specific crystal positions with an angle of 2θ at 37.1°, 43.1°, and 62.5°, as well as MgCl2 at positions 22.5°, 23.7°, 29.7°, and 40°. After adsorption (B4), new crystals with a strong signal at 15.04°, 16.5°, 20.91°, 21.5°, and 33.29° (JCPDS 15e762) were identified, in addition to MgCl2. SEM, EDX and XRD analysis confirmed that these signals corresponded to the MgNH4PO4.6H2O crystal structure, which suggested the formation of struvite on the crystal surface. The biochar surface acted as a substrate for struvite to attach to, facilitating the adsorption process and allowing for chemical reactions to occur:

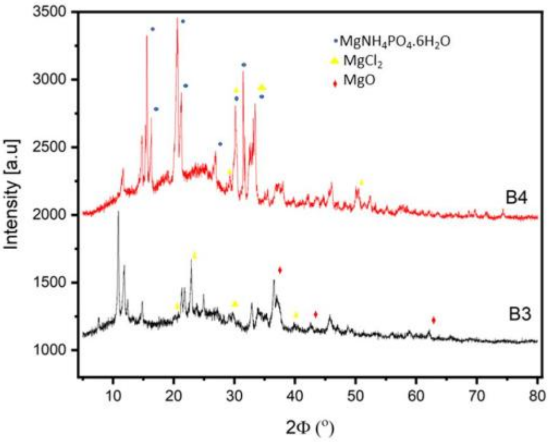


Fig. 7: XRD pattern of M 2/650/30 coffee ground biochar before (B3) and after adsorption (B4)

$$MgO + H_2O \rightarrow Mg(OH)_2 \rightarrow Mg^{2+} + OH^-$$

$$MgCl_2 \rightarrow Mg^{2+} + 2\ Cl^-$$

$$Mg^{2+} + NH_4^+ + PO_4^{3-} \rightarrow MgNH_4PO_4.6H_2O$$

3.3. Combination of coffee ground and sludge

The results of the experiment were presented in Figure 8. M 50%T/50%B (50% coffee ground and 50% sludge) demonstrated a PO_4^{3-} adsorption capacity of 43.03 mg/g, indicating a 1.61 mg/g increase compared to material sample M 2/650/30. Although this difference did not meet statistical significance, a marked disparity was observed in the ammonium index. M 50%T/50%B had an NH_4^+ adsorption capacity of 7.91 mg/g, significantly lower than the capacity of 20.55 mg/g recorded for Material sample M 2/650/30. While both materials had comparable PO_4^{3-} adsorption capacity, Material sample M 2/650/30 exhibited a notably higher NH_4^+ adsorption capacity. Hence, it was recommended to conduct further tests of sample M 2/650/30 adsorption capacity with real wastewater to ascertain its utility.

3.4. Actual test result

The actual test results with the original wastewater swine are shown in Figure 9. After conducting further testing with real wastewater, M2/650/30 was analyzed for PO_4^{3-} and NH_4^+ criteria. The concentration of PO_4^{3-} in the synthetic wastewater and real wastewater inputs were 118.51 mg P-PO_4^{3-} /L and 121.78 mg P-PO_4^{3-} /L, respectively. The adsorption capacity of M 2/650/30 for synthetic and real wastewater was found to be similar for the PO_4^{3-} indicator. However, for NH_4^+, there was a significant difference in adsorption efficiency and capacity due to the substantial variation in concentration between

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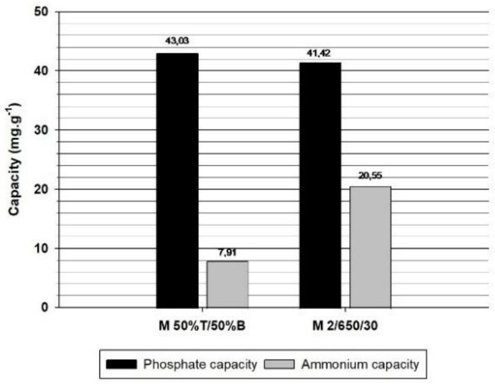

Fig. 8: Comparison between M 50% coffee ground/50% sludge and M 2/650/30 coffee ground biochar

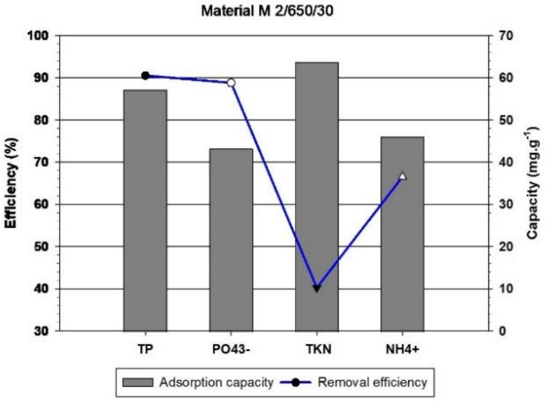

Fig. 9: The adsorption capacity (mg/g) and removal efficiency (%) of ammonium ion, phosphate ion with real swine wastewater

the actual and synthetic wastewater samples. While the synthetic wastewater had a concentration of N-NH_4^+ at around 100 mg/L, the actual wastewater had a concentration of 173.23 mg/L, which was 1.72 times higher. Despite the higher input, the adsorption efficiency of real wastewater was only 1.3 times higher than that of synthetic wastewater (66.53% and 50.97%). Nevertheless, Material M2/650/30 demonstrated an adsorption capacity of 46.10 mg/g for real wastewater, which was 2.24 times higher than that of synthetic wastewater (20.55 mg/g).

In general, natural wastewater such as swine wastewater contains more dissolved inorganic and organic ions than synthetic wastewater, which has the potential to cause ionic competition in the adsorption process, especially against ammonium (NH_4^+) and phosphate (PO_4^{3-}) ions. Under these conditions, ions such as Na^+, K^+, Ca^{2+}, and Cl^- can compete to fill the active sites on the surface of the biochar, which would theoretically decrease the efficiency of adsorption. However, the results of this study show that the efficiency of treating natural wastewater is actually higher than that of synthetic wastewater. This can be explained by the presence of additional components in the original wastewater (e.g. organic compounds, complex compounds Mg and Ca, and micronutrients) that can increase the formation of precipitate (e.g. struvite or magnesium phosphate) and aid in the activation of functional groups on the surface of the biochar [45,46]. In addition, the synergistic interaction between phosphate ions and ammonium in the real environment can encourage joint precipitation, especially when biochar has been modified with magnesium, resulting in higher removal efficiency than more controlled but less complex synthetic systems [47].

3.5. Potential ammonium and phosphate removal mechanism

The mechanism of removal of ammonium and phosphate compounds involves adsorbents and adsorbates for ion exchange, physical adsorption, surface complexity, and electrostatic attraction. In some related studies, biochar has a large surface area but the effectiveness of NH_4^+ adsorption capacity is lower [17]. Thus, in this study, the biochar from coffee grounds was modified with $MgCl_2$ in an effort to increase surface capacity and enlarge porosity. Biochar coffee grounds modified with $MgCl_2$ and produced through pyrolysis at 650°C are able to remove ammonium (NH_4^+) and phosphate (PO_4^{3-}) through different complementary mechanisms. Biochar modified with magnesium ions (Mg^{2+}) exhibits superior performance for the removal of phosphate (PO_4^{3-}) from water through two main mechanisms. First, precipitation of Mg-phosphate (such as $Mg_3(PO_4)_2$) occurs when phosphate reacts with Mg^{2+} on the surface of the biochar, resulting in deposits that are retained in the pore structure or biochar surface [48]. Second, there is a ligand exchange between the surface –OH group of the biochar and phosphate ions, allowing for stable chemical adsorption of phosphorus oxide [46,49,50]. Meanwhile, for ammonium (NH_4^+), although biochar from coffee grounds shows good NH_4^+ adsorption capacity (Q_{max} up to ~50 mg/g based on moderate-temperature biochar without Mg) through electrostatic adsorption and ion exchange, this mechanism tends to be limited to biochar that has been highly pyrolysis due to reduced cation exchange capacity (CEC) [17,51]. Studies on corn/magnesium biochar also show that NH_4^+ can be absorbed through a combination of ion exchange and precipitation/struvite-like precipitation with PO_4^{3-} on the surface of Mg-modified biochar [52].

3.6. Comparison with other biochar

Table 5 shows several types of biochar made from other raw materials other than coffee grounds that can reduce phosphate ions and ammonium ions in polluted wastewater. Some of them are combinations and modifications between biochar and other materials to improve the performance of biochar, such as the combination of biochar with chitosan, modification with magnesium, and so on.

Table 5: Comparison of phosphate and ammonium adsorption results with other biochar

Material	Biochar's Feedstock	Pyrolysis temp. (°C)	Highest adsorption capacity (mg/g)		Highest removal efficiency (%)		Ref.
			Phosphate	Ammonium	Phosphate	Ammonium	
Magnesium doped biochar (Mg/BC)	Pine bark	450	64.65	62.50	88.30	59.36	54)
Biochar	Oil palm shells	650	0.89	1.49	12	9	55)
Chitosan modified with magnesium and biochar (CS-MgCBC)	Corn straw	400	221.89	35.59	90.3	40	53)
Biochar	Pig manure	400 and 700 (mixed with ratio 2:3)	1.355	2.48	±14	±25	56)
Biochar	Sugar cane bagasse	500	2.76	4.46	-	-	57)
Modified biochar with oyster shell	Wheat straw	800	151.37	0.80	85.02	8.20	58)
Biochar	Pine wood	600 for phosphate; 400 for ammonium	42.26	5.30	28.70	- (not shown)	59)
Biochar	Conocarpus plant	600	4.80	3.50	- (not shown)	- (not shown)	60)
	Paulownia plant	300 for phosphate; 600 for ammonium	3.00	1.50	- (not shown)	- (not shown)	60)
Biochar modified with $MgCl_2$	Coffee ground (real wastewater test)	650	43.26	46.10	88.82	66.53	This study
	Coffee ground (synthetic wastewater test)	650	41.42	20.55	87.38	50.97	This study

From the results of the analysis of Table 5, it is known that the study by Li et al. (2022) combining biochar with chitosan has the largest phosphate adsorption capacity (221.89 mg/g) and ammonium adsorption capacity of 35.59 mg/g, with a pyrolysis temperature of only 400°C. This can happen because chitosan can increase specific surface area [53]. Each type of biochar has its own characteristics, so the adsorption ability is also different. When compared to other types of biochar, coffee grounds biochar can be said to be more effective in adsorbing phosphate and ammonium. Judging from the results of the balanced removal efficiency, although ammonium removal is lower than phosphate removal. The final results show that the value of adsorption capacity is substantially dependent on the type of biomass, the extent to which it is processed and activated, and the conditions of the biomass production itself [57].

4. Conclusion

The research project successfully developed adsorbents from coffee grounds that were modified with $MgCl_2$, and the resulting materials were analyzed using XRD, SEM, and EDS. The adsorption capacity of the material was evaluated for the $P\text{-}PO_4^{3-}$ and $N\text{-}NH_4^+$ criteria, and it was found that the optimal adsorption time was 14 hours, and the optimal pyrolysis time was 30 minutes. The best treatment efficiency was achieved by using Material M 2/650/30, which was modified with a 2% $MgCl_2$ solution and pyrolyzed at 650°C for 30 minutes. This material had an ammonium adsorption efficiency of 50.97% and a phosphate adsorption of 87.38% for synthetic wastewater. For the combination of biochar results from 50% coffee ground and 50% sludge (M T50%/B50%) have a phosphate absorption capacity of 43.03 mg/g, 1.61 mg/g higher than M 2/650/30. However, for ammonium absorption it is very far behind M 2/650/30. The material's effectiveness was further tested on real wastewater, and it was found that sample M 2/650/30 had an ammonium treatment efficiency of 66.53% and a phosphate treatment efficiency of 88.82%.

These results show that biochar coffee ground M 2/650/30 has the best effectiveness in treating wastewater from pig farms. Although the combination of biochar coffee ground with sludge showed successful adsorption of phosphate and ammonium, the adsorption power of ammonium was considered not optimal compared to M 2/650/30. This happens due to various factors, such as the type of sludge that affects the content in it. Therefore, further studies are needed to observe the problem.

Regardless of that, coffee ground biochar has very promising prospects in the future as a multifunctional solution in various fields, from climate change mitigation to sustainable agriculture and waste management. Its ability to store carbon in the long term makes it an important tool in greenhouse gas emission reduction strategies. In addition, the benefits of biochar in improving soil quality, absorbing pollutants, and supporting a circular economy show its broad potential both in terms of environment and economy.

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