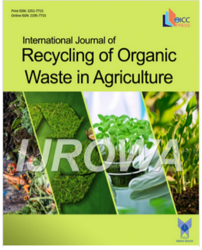


Volume 13, Issue 1, 132409 (1-9)

International Journal of Recycling Organic Waste
in Agriculture (IJROWA)

https://dx.doi.org/10.57647/j.ijrowa.2024.1301.09

Biochar production from agricultural waste (corncob) to remove ammonia from livestock wastewater

Hamza El Ouassif, Ulrike Gayh, Mohammad R. Ghomi*

Department of Water Technology, School of Engineering and Architecture, SRH University Heidelberg, Germany.

***Corresponding author**: mohamad.ghomi@srh.de

Received 26 July 2023 ; Accepted 05 Oct. 2023 ; Published Online 09 Oct. 2023 ORIGINAL RESEARCH

Abstract:
Purpose: With the constant increase in food demand, agriculture became the second-highest waste-generating industry. Crop residues and wastewater from livestock farming are the major contributors and irrational disposal of them has a serious impact on ecosystems as well as human health. In this study, corncob as agriculture waste was used as a treatment agent, after converting it to biochar that adsorbs ammonia nitrogen in swine wastewater.
Method: The biochar was prepared by pyrolysis at two different temperatures 350°C and 450°C. The physical properties of the biochar were investigated using scanning electron microscopy (SEM) analysis. The ammonia adsorption capacities and removal efficiencies of the two biochars were investigated using batch experiments by changing the pH of the wastewater solution as well as the contact time.
Results: The highest ammonia removal efficiency of 83.98% was recorded at 450°C after 90 min of contact time under alkaline pH (12) wastewater conditions whereas the lowest removal efficiency of 34.64% was obtained for 350°C after 30 min exposure to wastewater at normal pH (7.32) condition.
Conclusion: This study contributed to the ongoing research on the potential of feedstock-derived biochar to remove pollutants from wastewater.

Keywords: Biochar; Corncob; Ammonia nitrogen removal; Livestock wastewater treatment

1. Introduction

With the constant growth of the global population reaching 8 billion in mid-November 2022 and an expected increase up to 9.7 billion in 2050 (UN 2022), the food demand will also escalate leading by consequent to the expansion of the agriculture sector either crop or meat production. The growing amount of food produced is matched by the load of waste generated. In agriculture, two types of waste are generated: Crop residues which are the remained materials from cultivated crops (Sharma et al. 2018) and livestock wastewater which includes animal manure and urine (Parihar et al. 2019). Corn is one of the four major primary crops worldwide with a total production of 1,216.87 million tons in 2022 (USDA 2022), only the kernels or grains that were consumed. Consequently, a lot of corn waste is generated, mainly corn cob which is the core on which the grains are arranged and corn leaves. Open-air burning was, and still is, in many developing countries a way of disposing of crop residues. Thus, it releases a significant load of greenhouse gases such as methane (CH_4), carbon monoxide (CO), nitrous oxides (NOx), and many more that have a serious impact on human health if it is inhaled as well as the environment (Saxena et al. 2021).

Biochar, a considerable source of agricultural waste, can be defined as a carbon-rich solid material obtained from the thermochemical conversion of biomass in an oxygen-limited environment (IBI 2014). The global biochar market size was valued at $184.90 million in 2022 and is projected to grow to $450.58 million by 2030 (Insights 2023). Pyrolysis is considered the most frequently used technique for the production of biochar. It consists of decomposing biomass under elevated temperatures and low oxygen environment (Deng et al. 2017). The physiochemical properties and the effectiveness of the obtained biochar depend on the waste used and the preparation conditions. Thus, the selection of the feedstock and the preparation parameters are of high concern (Quach et al. 2022). Depending on the operating conditions, pyrolysis can be divided into slow pyrolysis, fast pyrolysis, and flash pyrolysis (Onay and Kockar 2003). Slow pyrolysis occurs at a temperature ranging from 300°C to 700°C and generally takes hours generating by consequent a higher biochar yield between 35 and 50 wt%. Fast pyrolysis is carried out at a temperature between 400°C and 600°C for a short period $<$ 10 seconds which means that the heating rate needs to be higher generating by consequent a yield of less than 30 wt% (Onay 2007). Flash pyrolysis generally takes less than 3 seconds at a temperature of $\sim$ 1000°C and generates a yield not exceeding 20 wt% which makes it the less adopted technique (Grima-Olmedo et al. 2016).

As the world's most precious resource and the basis of all forms of life, water is considered a unique and valuable asset and now is affected by global warming. Global warming has been one of the major environmental issues facing the world for decades and will remain one of the biggest threats to human existence for many years to come (Musa et al. 2021). Approximately 69% of global water use is dedicated to agricultural activities in all its forms, whereas the industrial sector is set to 19%, leaving 12% for municipal activities. This constant increase will affect societies and economies making it hard to maintain sustainability and leading to a water shortage that can reach up to 40% by 2030 (WWDR, n.d.). Even though since 2000, 2.1 billion people have gained access to basic sanitation services, still the waste generated is not properly managed according to regulations, leading to approximately 50% of the world's child deaths (WHO 2019). As the main concern is to afford global access to water and its services, the wastewater produced was left out of sight. Wastewater may contain huge amounts of nutrients, heavy metals, infectious agents, organic and inorganic chemicals as well as micropollutants. Neglecting wastewater can cause significant human health problems and environmental issues (WWAP, n.d.).

On the other hand, livestock wastewater contains high concentrations of nutrients (nitrogen and phosphorus) and organic matter presented as BOD (biological oxygen demand) and COD (chemical oxygen demand). The percentages of these pollutants vary depending on the species of elevated animals (pigs, chickens, cows, and sheep) and their feed constitution (Nagarajan et al. 2019). For instance, swine wastewater which is a mixture of pig excrement and the water used for cleaning pigpen. It was estimated that 4 to 8 L of effluent is generated daily per pig (Zhang et al. 2017) and with the tremendous increase in food demand, it was recorded as of April 2022 the housing of 784.2 million pig head worldwide (FAO 2022) leading by consequent to the release of huge loads of swine wastewater. Biodegradation of wastewater in a non-oxygen environment converts organic matter (BOD and COD) into methane that can be captured and used as biogas (Zhang et al. 2021). However, this technology is vulnerable to nitrogen compounds, especially ammonia (NH_3), formed during the decomposition of nitrogen substrates. It lowers the carbon-to-nitrogen ratio (C/N) which is crucial for methane production (Zhang et al. 2020). It is important to conserve the lands and water bodies that we have or might locate for the production of food and feed (Haoujar et al. 2022). If no advanced oxidation processes are used as the tertiary treatment stage, which most wastewater treatment plants do not dispose of due to its high maintenance costs, the pollutants will get discharged directly into water bodies or even reused for irri-

Figure 1. Preparation of corncob and pyrolysis process.

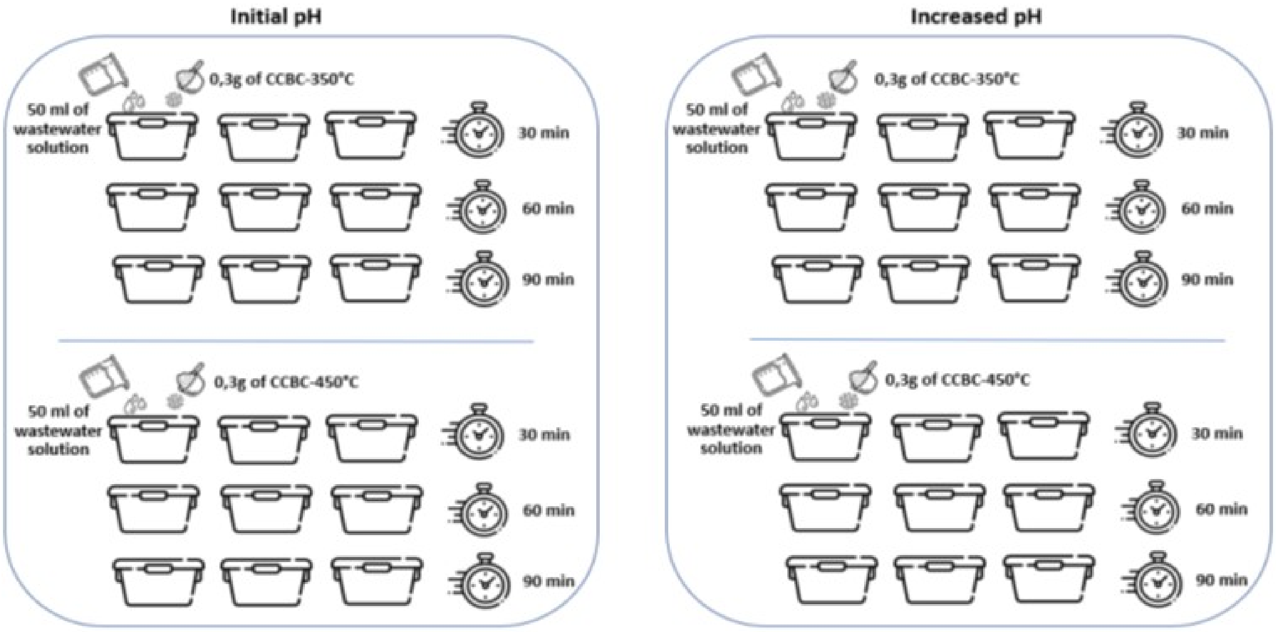


Figure 2. Layout of the experimental setup in a factorial design of 3 × 2 × 2 and 3 replicates for each. The initial pH was 7.32 and the increased pH was 12.

gation purposes threatening by consequent the aquatic life, ecosystems, ecosystem services and human health (Malik et al. 2020; Vries 2021).

Ammoniacal nitrogen (AN), an inorganic form of nitrogen, is formed by the breakdown of nitrogenous organic compounds (urea, proteins, uric acid, etc.) in its two forms (ammonia NH_3 and ammonium NH_4^+). NH_3 is considered the toxic form of AN mainly present in a relatively high pH and temperature solutions, has a strong smell and has a negative impact on aquatic organisms compared to NH_4^+. AN can be regularly found in numerous types of wastewater, notably municipal, industrial, and agricultural. Excessive AN concentration in water resources leads to one of the most serious environmental issues which is eutrophication (Vries 2021).

During the past decade, many technologies have been designed and developed for the removal of AN from wastewater. Those technologies can be divided into three main categories: physical (ammonia stripping, ion exchange and adsorption and membrane technology), chemical (struvite precipitation, electrochemical oxidation, and photocatalysis), and biological (nitrification–denitrification, microalgae treatment, biochar preparation methods, gasification, hydrothermal carbonization, and pyrolysis.

This study focused on corn waste, most precisely the cob part, suitability to be used as biochar and its ability to remove nutrients from swine wastewater with a special emphasis on ammonia nitrogen, to analyze the potential of the obtained biochar to remove ammonia from swine wastewater.

2. Materials and methods

2.1 Biochar preparation and analysis

Since the harvesting season for corn in Germany is early autumn (end of September – beginning of October), there wasn't a possibility to cultivate fresh corn out of a nearby field for the project. Therefore, the maize was obtained from a pet food store 'Futterplatz' in Deggendorf, Germany. The grains were peeled out of the corn, only the cob part was collected. The corn cobs were washed first with tape water to remove small particles stuck at the surfaces and then with distilled water to remove any remaining impurities. The washed cobs were then dried in a 'UN30 Memmert, Germany' lab oven at 100°C until constant weight in order to get rid of any mois-ture content (Fig. 1). The dried cobs (∼ 2 kg) were then sorted into relatively equivalent sizes and crushed using an 'MX1250 Rommelsbacher, Germany blender and then sieved into 0.5 mm - 2 mm fine particles. The obtained powder was then used as feedstock for pyrolysis.

2.2 Pyrolysis process

The powder corncob was divided into two equivalent batches. The first batch was placed in a 148 × 108 × 75 mm covered aluminum box (Fig. 1), then pyrolyzed for 3 hours in a muffle furnace (EFCO 180 KF kiln, Germany) at a heating rate of 3°C/min. After two hours, and when the temperature reached 350°C, it was saved for the remaining hour. The obtained biochar will be referred to as CCBC-350 (corncob biochar-350). Following the same procedure, the second batch was pyrolyzed for 3 hours at 4°C/min as heating rate, once the temperature reached 450°C, it was kept that way for another hour. The prepared biochar was labelled CCBC-450. The two obtained biochars were kept in airtight plastic containers.

2.3 Biochar characterization

To identify and visualize the morphology of the prepared biochar compared to its raw state which consists of the surface area and its porosity, three samples have been taken:

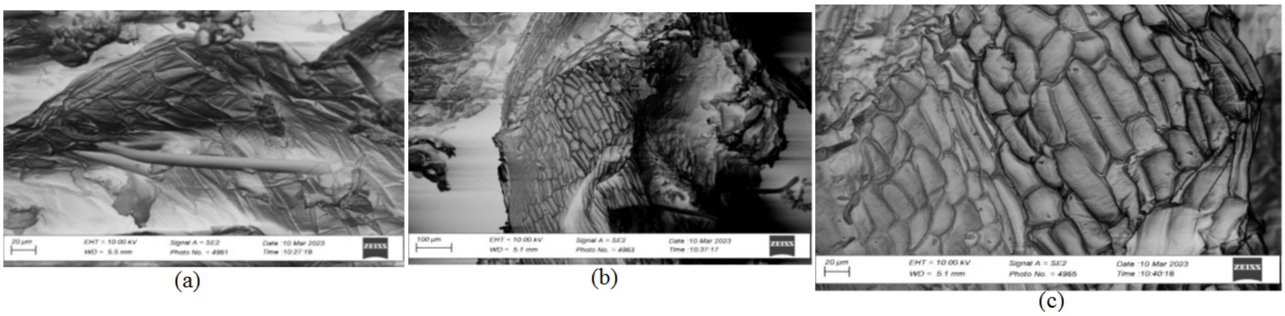

(a) (b) (c)

Figure 3. SEM images for the raw corncob sample: (a) 100x magnification; (b) 300x magnification; (c) 3000x magnification.

(i) powder corncob before pyrolysis CC, (ii) CCBC-350, and (iii) CCBC-450. The samples were analyzed using Zeiss Leo 1530 scanning electron microscope (SEM) by Electron Microscopy Core Facility (EMCF) in Ruprecht-Karls-Universität Heidelberg, Germany. The SEM produces an electron beam that irradiates bulk and thin film samples in a vacuum, with a primary beam of electrons ranging in energy from 0.2 to 30 kilovolts (kV). The beam is focused into a probe, $1-3$ nanometers in diameter, which is restored across the surface of the sample. Detectors mounted inside the chamber collect the secondary electrons emitted by the sample material, amplify the signal, and electronics reconstruct the information, point-by-point, to display the likeness of the sample surface. The images were taken with an energy of 10 kV, a working distance of 5 mm, a resolution of 2048 × 1536, a magnification of 100×, 300× and 3000×, and a secondary electrons detector (SE2) at 8× scan speed with no averaging.

2.4 Wastewater sampling and analysis

Wastewater samples were taken from a piggery farm at Edingen-Neckarhausen, Germany. The number of pigs found at the moment of sampling was 26 pigs. Wastewater samples were collected in 300 ml plastic sampling bottles washed previously with distilled water to remove any remaining impurities. The bottles were then stored in an ice pack and then transferred to SRH University's lab and kept at 4°C.

The pH and temperature of the wastewater were measured using a pocket pH meter at the moment of the sampling and using an HQ40D Multimeter for more accurate measurement at the laboratory.

The ammonia concentration in the wastewater was measured using ammonia nitrogen powder pillows from Hach (Germany) following the salicylate method for a concentration ranging from 0.01 to 0.5 mg/L and DR3900 VIS Spectrophotometer (Hach, Germany). The wastewater sample was diluted by a factor of 10, by taking 1 ml of the wastewater using a pipette, and adding it to 9 ml of distilled water. Then, the diluted sample was placed in the first sample cell, whereas the second sample cell was filled with 10 ml of distilled water to constitute the blank. The presence of ammonia in the diluted wastewater sample was proven by the change of the colour to green. The displayed value was then multiplied by 10 to get the real ammonia concentration of the wastewater as mg/L.

2.5 Set up of the experiments and data analysis

Batch experiments (Fig. 2) were carried out in a factorial design (3 × 2 × 2) to determine the ammonia adsorption capacity of the prepared biochar at different pH of the wastewater solution and different contact times. The plastic containers were filled each using a pipette with 50 ml of the wastewater solution, and 0.3 g of CCBC-350/450 was then added to each container (first factor). pH (second factor) was constituted in two values, firstly using the normal level of 7.32 that wastewater had, and 12 (NaOH was added with constant stirring until maintaining pH 12). Finally, for the third factor (contact time) the study was carried out in 30, 60, and 90 min. Constant stirring of the mixtures was maintained during the entire reaction time. Once the timer expired, ammonia concentrations were measured using the Hach powder pillow procedure mentioned in the previous section.

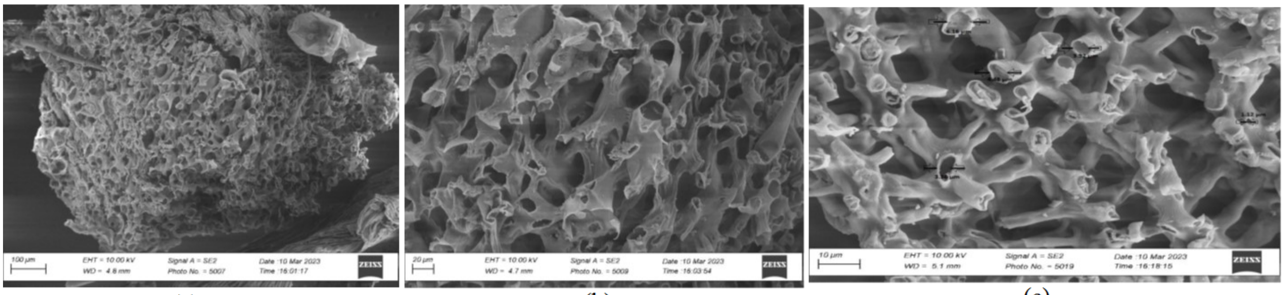
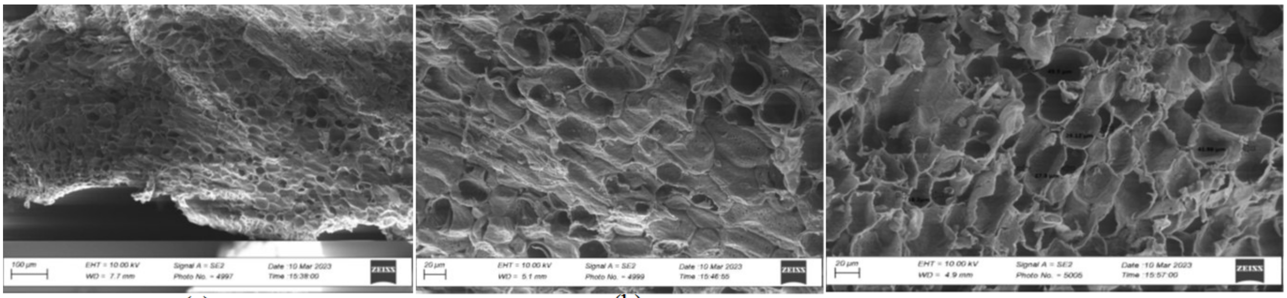

(a) (b) (c)

Figure 4. SEM images for the CCBC-350 corncob sample: (a) 100x magnification; (b) 300x magnification; (c) 3000x magnification.



(a) (b) (c)

Figure 5. SEM images for the CCBC-450 corncob sample: (a) 100x magnification; (b) 300x magnification; (c) 3000x magnification.

After getting ammonia concentrations from different samples, the adsorption capacity was calculated using the following formula (1) (Srivastava and Sharma 2013):

$$q_e = \frac{(C_i - C_e) * V}{M} \tag{1}$$

- C_i is the initial ammonia concentration of the solution (mg/l)
- C_e is the ammonia concentration of the solution after contact with biochar (mg/l)
- V is the volume of the solution (L)
- M is the dosage of biochar (g)
- q_e is the adsorption capacity (mg/g)

The removal efficiency (R_{eff}) (%) is calculated using the formula below (2) (Srivastava and Sharma 2013):

$$R_{eff} = \frac{(C_i - C_e) * 100}{C_i} \tag{2}$$

Data were analyzed with a factorial design (General Linear Model) of $3 \times 2 \times 2$ and 3 replicates for each using SPSS 26. In total, 36 experimental units were run for tests. Duncan's multiple range test was used to compare means at $P < 0.05$.

3. Results and discussion

3.1 Biochar characterization

The SEM images of the three samples (raw, 350°C, and 450°C) are displayed in Figs. 3-5. The difference in changing the nature of corncob's biochar rather than raw material (raw corncob) could be visible and differentiated particularly in 3000x magnifications.

The surface topography of raw corncob before inducing any change looks to be relatively compact as shown in Fig. 3. At higher magnification 3000x, the SEM image shows no sign of any kind of pores, the surface layer is continuous without any intercellular spaces in between. This means that the raw corncob has no potential to adsorb pollutants since the surface area does not provide any habitation zones for contaminants.

SEM images for the corncob-derived biochar prepared at 350°C in Fig. 4 show that the pyrolysis process produced a significant number of pores with different diameters on the surface area of the biochar varying between 1.12 μm and 4.48 μm. Some pores were half completed which looks like that the surface area is not fully firm and the pores are not uniformly distributed. The development of pores is due to thermal pressure applied inside the furnace which proves the potential of corncob as feedstock for pyrolysis.

Table 1. Interaction effect of all factors on efficiency (%) of corncob biochar in removal of ammonia.

Group	Contact time	Biochar temp.	pH	Mean ± SD
1	30	350	Normal (7.32)	34.64 ± 8.56 h*
2	30	350	12	43.01 ± 4.06 fg
3	30	450	Normal (7.32)	59.69 ± 4.99 cd
4	30	450	12	62.37 ± 6.52 bc
5	60	350	Normal (7.32)	42.27 ± 8.22 g
6	60	350	12	53.76 ± 2.46 e
7	60	450	Normal (7.32)	70.15 ± 1.32 b
8	60	450	12	79.57 ± 3.36 a
9	90	350	Normal (7.32)	53.16 ± 6.80 ef
10	90	350	12	57.53 ± 3.36 de
11	90	450	Normal (7.32)	71.24 ± 1.18 ab
12	90	450	12	83.98 ± 1.50 a

* Small superscript letters in the same column indicates no significant difference ($P > 0.05$).

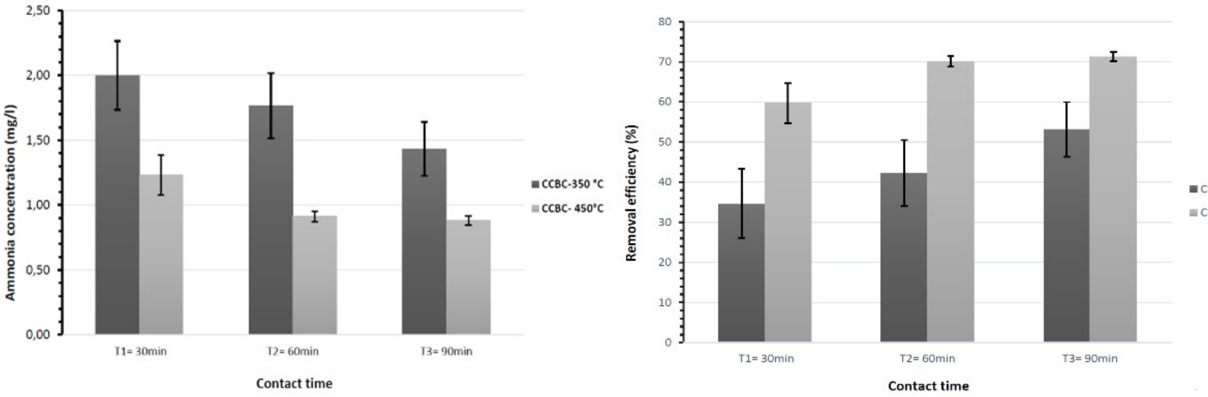


Figure 6. Adsorption capacity of CCBCs at normal pH (7.32) conditions in function of contact time.

The appearance of pores on the surface means that the produced biochar has the capability to adsorb pollutants and immobilize them inside the formed pores which confirms the results obtained in previous studies done on corncob (Zhang et al. 2014; Vu et al. 2016; Amen et al. 2020).

Increasing the pyrolysis temperature from 350 to 450°C has led to the formation of numerous and uniformly distributed pores on the surface area of the biochar with their walls open to each other as shown in Fig. 5, which collide with previous studies on the impact of increasing pyrolysis temperature on the surface morphology of the biochar (Quach et al. 2022; Sonu et al. 2020). The pores' radius also increased in the range of 18.2 μm and 45.5 μm. The structure of the surface became hard and homogenic. The increase in the number of pores is due to the further release of volatile matter from the corncob by the increase in temperature. The relatively big pore diameters will enhance the uptake of pollutants by providing more space for the contaminants to inhabit, therefore, increasing the adsorption capacity of the biochar similar to what the study by Giri et al. (2020) stated. Therefore, changing the preparation condition has a significant impact on the surface morphology of the obtained biochar. The higher the pyrolysis temperature increased, the higher the formation of pores.

3.2 Adsorption analysis

The ammonia concentration in the samples measured after adsorption by CCBC-350 and CCBC-450 at normal and changed conditions of pH incorporated by changing contact time are displayed in Figs. 6-7. The initial amount of ammonia concentration had been 3.06 mg/l for raw wastewater and when pH increased to 12, the amount of ammonia increased to 6.2 mg/l. The adsorption capacity of all groups has shown changes regarding the type of biochar (350 or 450°C), the contact time (30, 60, and 90 min), and normal pH and pH 12.

The interaction between all factors of the adsorption experiments (pH, contact time, and biochar preparation conditions) was analyzed and tabulated in Table 1. The results showed the best group to enhance the removal of ammonia occurred at 90 minutes of contact time, 450°C biochar, and pH 12 (group 12). Moreover, group 8 showed the highest efficiencies in adsorption of ammonia and its difference was not significant with group 12 ($P > 0.05$). Also, the least reduction efficacies happened for group 1, when biochar was used at 30 minutes contact time, 350°C biochar, and normal pH.

The residual ammonia concentrations in the wastewater samples after adsorption by CCBCs decreased with the increase in contact time. The average ammonia concentration decreased from 3.06 mg/l to 2 mg/l after 30 min of contact time then to 1.43 mg/l after 90 min for CCBC-350°C (Fig. 6), whereas for CCBC-450°C the average ammonia concentration left in the wastewater samples decreased from 1.23 mg/l after 30 min of contact time to 0.88 mg/l after 90 min. The values show that the biochar saturation is reached at 60 min since the concentrations only decreased slightly between 60 min and 90 min (by 0.03 mg/l for CCBC-450°C). It means that after 60 min of contact time, the pores on the surface of CCBCs are almost filled with ammonia substances. Similar results were reported by Assirey and Altamimi (2021) which confirms the values of the calculated adsorption capacities. The CCBCs adsorption capacity has increased as time passes but reached equilibrium after 60 min exposure time. CCBC-450°C was able to maintain the same adsorption rate of 0.36 mg/l between 60 min and 90 min which is also translated by the removal efficiency values. The removal efficiency increased from 36.64% after 30 min to 53.16% after 90 min for CCBC-350°C (Fig. 6), whereas 71.24% of ammonia was efficiently removed within 90 min contact time for CCBC-450°C.

Contact time has a crucial impact on the amount of ammonia adsorbed by CCBCs, finding the biochar adsorption equilibrium is the key to having the optimum removal efficiency. In this case, a contact time between 60 min and 90 min was enough to reach the maximum adsorption capacity of the produced CCBCs.

The significant difference in the performances of CCBCs (adsorption rate and removal efficiency) under initial pH conditions (pH 7.32) can be explained by the difference in the pyrolysis temperature from 350 to 450°C which changes the surface morphology of the biochar, leading to more pores on the surface area that can contain much ammonia substances.

After increasing the pH of the wastewater solution from 7.32 to 12, the ammonia concentration increased automatically

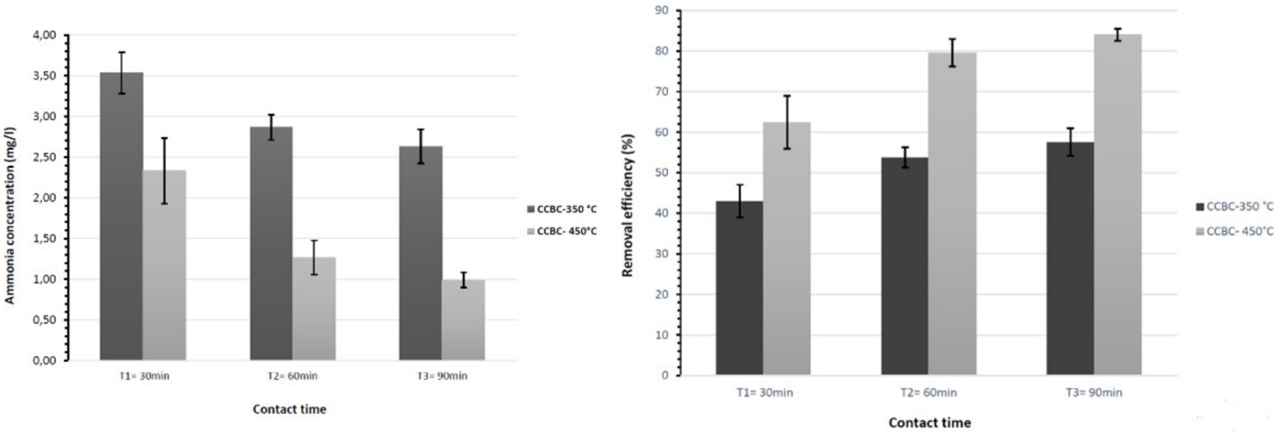


Figure 7. Adsorption capacity of CCBCs under pH 12 conditions in function of contact time.

from 3.06 mg/l to 6.2 mg/l. This significant increase can be explained by the fact that when the pH level reaches a value higher than 9 and the solution becomes alkaline, most of the ammonium ions present in the wastewater are converted into ammonia leading to higher ammonia concentration in the solution (Wurts 2003).

Under these new pH conditions, the ammonia concentration left in the wastewater samples after adsorption decreased with the increase in contact time the same as the initial conditions. For CCBC-350°C, the residual ammonia concentration decreased from 6.2 mg/l to 3.53 mg/l after 30 min, then to 2.63 mg/l after 90 min contact time (Fig. 7), whereas it decreases from 6.2 mg/l to 2.33, mg/l after 30 min, then to 0.99 mg/l after 90 min for CCBC-450°C. Similar to the initial condition batches, the CCBCs reached equilibrium after exposure time between 60 min and 90 min. The adsorption capacity of CCBC-350°C increased from 0.44 mg/g after 30 min to 0.56 mg/g after 60 min and then almost stabilized at the value of 0.59 mg/g until 90 min of contact time. The same goes for CCBC-450°C, the adsorption rate increased only by 0.05 mg/g in the period from 60 min to 90 min. Therefore, the pH increase didn't affect the CCBCs' adsorption equilibrium set at 60 min for the initial pH conditions. However, increasing the pH of the wastewater samples enhances the performances of the CCBCs. The removal efficiencies reached up to 57.53% and 83.98% after 90 min contact time for CCBC-350°C and CCBC-450°C (Fig. 7), respectively which are higher than the ones obtained at initial pH conditions.

The impact of pH increase on the performance of CCBCs can be explained by the fact that at higher pH levels, the surface of biochar is intensively charged which makes it adsorb more contaminants by consequently having a higher adsorption rate and removal efficiency (Zhang et al. 2014; Assirey and Altamimi 2021).

Wastewater solution at pH of 12 for 90 min and biochar prepared at 450°C gave the highest removal efficiency of 83.98%, whereas biochar prepared at 350°C exposed to wastewater solution at pH of 7.32 for 30 min gave the lowest removal efficiency of 34.64% (Table 1). This significant difference in removal efficiencies is related to the parameters set for each experiment which highlights the fact that the degree of adsorption depends essentially on the conditions where the biochar is used (pH, concentration, contact time, dosage, etc.).

4. Conclusion

Based on the surface morphology analysis results obtained using a scanning electron microscope, the change in pyrolysis temperature led to a change in the surface area of biochar between the two samples. The higher the temperature goes, the more pores with wide radiuses appear on the surface of the biochar. The highest removal efficiency of 83.98% which consists of preparing biochar at 450°C and exposing it to the wastewater solution at a pH of 12 for 90 min. Even though pH fluctuations cannot be controlled in livestock farms, based on this study using biochar prepared at 450°C for a 90-min contact time, reaching the highest ammonia removal efficiency can be expected. Thus, by getting a higher pH of 12, the amount of ammonia increased from 3.06 to 6.2 mg/l, the reduction rate of ammonia still showed the highest and enhanced to 83.98% more than other groups. This research has proven that corncob as an agricultural waste feedstock for pyrolysis to produce biochar is considered a promising stock for ammonia nitrogen adsorption from wastewater.

Acknowledgments:
The authors want to thank Mr. Klaus Friedrich (SRH University Heidelberg) for his kind contribution and help in the preparation of technical support for the study.

Author contributions:
The authors confirm the study conception and design: H. El Ouassif, U. Gayh, and M.R. Ghomi; data collection: H. El Ouassif; analysis and interpretation of results: H. El Ouassif, U. Gayh, and M.R. Ghomi; draft manuscript preparation: U. Gayh and M.R. Ghomi. The results were evaluated by all the authors, and the final version of the manuscript was approved.

Compliance with ethical standards
Conflict of interest: The authors declare that there are no conflicts of interest associated with this study.

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