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Chitosan-Activated Charcoal of Modified Corn Cobs as an Antibiotics Adsorbent

Gatut Ari Wardani [1*], Clarisa Kartika Sanusi [1], Firman Gustaman[2], Taufik Hidayat[1], Estin Nofiyanti[3]

[1] Program Study of Pharmacy, Faculty of Pharmacy, Universitas Bakti Tunas Husada, Cilolohan Street Number 36, Tasikmalaya City, West Java, Indonesia. 46115
[2]Program Study of Pharmacist Professional, Faculty of Pharmacy, Universitas Bakti Tunas Husada, Cilolohan Street Number 36, Tasikmalaya City, West Java, Indonesia. 46115
[3]Program Study of Environmental Engineering, Faculty of Engineering, Universitas Muhammadiyah Tasikmalaya, Tamansari Street KM 2,5, Mulyasari, Tamansari, Tasikmalaya City, West Java, Indonesia. 46196

*Corresponding author: gatutariwardani@universitas-bth.ac.id

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Abstract

Excessive use of tetracycline hydrochloride can cause water pollution. The concentration of tetracycline hydrochloride in an aqueous solution can be reduced by an adsorption method that utilizes activated charcoal as an adsorbent. This study aims to determine the effect of the addition of chitosan on the characteristics of activated charcoal, the effect of pH, adsorption kinetics, the adsorption isotherm, and the effect of sonication on adsorption ability. The method used in this research is the batch method combined with the sonication method. Based on this research, it is known that the use of ultrasonic waves (sonication) can streamline the adsorption process of tetracycline hydrochloride. The time required for the adsorption process is faster with greater adsorption capacity and efficiency. Tetracycline solution can be absorbed optimally at an acidic pH, which is pH 4. The adsorption process using the sonication method can increase the adsorption capacity from 2.092 mg.g^{-1} to 5.330 mg.g^{-1}. The adsorption mechanism in both methods (batch and sonication) that occurs follows the kinetic equation of the two Ho pessaries. In contrast to adsorption kinetics, the adsorption isotherm for the batch method corresponds to the Langmuir adsorption isotherm equation (R^2 = 0.950), while the use of the sonication method corresponds to the Freundlich isotherm equation (R^2 = 0.859). Thus, corn cob waste can be utilized by converting it into activated charcoal modified by chitosan as an adsorbent for antibiotic waste, especially tetracycline hydrochloride.

Keywords: *Pollution; waste; tetracycline; adsorption mechanism; adsorption capacity*

Introduction

The use of antibiotics or anesthetics in hospitals can potentially be a waste of the pharmaceutical industry. If the industrial waste is disposed of directly without any processing, it can be a source of environmental pollution, especially in the aquatic environment (Wildan & Mutiara, 2016). One type of antibiotic that is still used in various hospitals is tetracycline. Tetracycline is a type of broad-spectrum antibiotic for gram-positive and gram-negative bacteria, and works bacteriostatically (Fernanda & Chrisnandari, 2021).

Various methods have been applied to reduce the concentration of tetracycline in the water. One method that may be used is the adsorption method, whidc utilizes biochar or activated charcoal. Biochar or activated charcoal is a material rich in carbon content obtained from agricultural waste through an incomplete combustion process. The use of activated charcoal is considered effective because it has high porosity and a specific surface area (Yuxue Liu , 2018).

Activated charcoal has been widely used to degrade the concentration of various organic compounds. Sources of activated charcoal can be obtained from various types of agricultural waste, such as banana peels and coffee (Getachew , 2015), corn cobs (Saputro , 2018), sawdust (Wardani , 2021), rice husk (Firdus , 2021), and nimba leaves (*Azadirachta indica*) (Patel, 2020). The adsorbent material synthesized from various types of agricultural waste can be used to adsorb various types of pollutants, both organic and inorganic pollutants.

Corn cobs have been successfully developed into activated charcoal, including to adsorb lead (II) metal ions, (Saputro , 2018), mercury (II) metal ions (Z. Liu , 2020), methylene blue (Medhat , 2021), and dye (Sun , 2021). In addition, several antibiotic compounds have also been successfully adsorbed using activated charcoal from corn cobs using the batch method (Dang , 2021; dan Li , 2022).

Activated charcoal produced from the corncob pyrolysis process is hydrophobic (Enaime , 2020). The hydrophobic nature of activated charcoal means that tetracycline hydrochloride compounds are not optimally absorbed in activated charcoal because tetracycline hydrochloride is in an aqueous solution. The addition of chitosan to activated charcoal is known to increase the polarity and hydrophilicity of the activated charcoal (Shi , 2020).

The method used is not only the batch method but also the sonication method, which is known to potentially help the adsorption process become more effective (Alfanaar , 2017). The sonication method is a method that utilizes ultrasonic waves. The frequency generated by very high sound waves (ultrasonic) can cause molecular changes that accelerate reactions (Razavian , 2014). Thus, the use of ultrasonic waves in the adsorption process is predicted to increase the effectiveness of the adsorption process that occurs.

The modification of corncob-activated charcoal by using chitosan and the addition of the sonication method to the adsorption process of tetracycline hydrochloride have never been developed. This study aims to improve the performance of corncob-activated charcoal in absorbing tetracycline hydrochloride compounds, which can potentially be a source of pollution for the aquatic environment. Thus, corncob-activated charcoal modified with chitosan can be an alternative material that can be used for the adsorption process of antibiotic compounds in pharmaceutical or hospital industrial wastes so that the potential for contamination can be minimized.

Methodology

Material

The equipment that will be used in this research includes laboratory glassware, a UV-Vis spectrophotometer (Shimadzu), Scanning Electron Microscopy (SEM) (Phenom Desktop ProXL), and Fourier Transform Infra-Red (FTIR) (Diamond ATR, Agillent).

The materials used were corn cobs, tetracycline antibiotics, 4N hydrochloric acid (HCl) (Merck), 1% starch indicator, iodine, sodium thiosulfate ($Na_2S_2O_3$), 2% acetic acid, aqua DM, chitosan, glutaraldehyde, and 0.1 M NaOH.

Preparation of Tools and Materials

The equipment that will be used in this research includes an UV-Vis spectrophotometer, Scanning Electron Microscopy (SEM), Fourier Transform Infra-Red (FTIR), and X-ray Diffraction (XRD). The materials used were corn cobs, tetracycline antibiotics, 4N hydrochloric acid, 1% starch indicator, iodine, sodium thiosulfate, 2% acetic acid, chitosan, glutaraldehyde, and 0.1 M sodium hydroxide.

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Volume 5, Issue 2, 2022

Corn Cob Charcoal Making

Corn cobs that have been cleaned and then dried are used to make charcoal. After that, the corn cobs were heated in a kiln at 400°C for 1 hour to obtain charcoal. The charcoal obtained is then pulverized to form a powder. After it is in powder form, the charcoal is sieved using a 100 mesh sieve. The sifted charcoal samples were then characterized using FTIR and SEM (Wardani , 2021).

Corncob Charcoal Activation

Activation of Corncob Charcoal for the activation process, 60 grams of charcoal were soaked in 4N hydrochloric acid for 24 hours. Next, the mixture were filtered and washed with distilled water until the pH was neutral. The resulting charcoal is then dried in an oven at 110°C for 3 hours, then cooled in a desiccator. The samples were then characterized using FTIR, and SEM.

Activated Charcoal Characterization

Determination of Water Content (Badan Standarisasi Nasional, 1995)

A total of 1 gram of activated charcoal was placed in a porcelain dish whose mass was known, then dried in an oven at 105°C for 1 hour, and then cooled in a desiccator for 15 minutes. After that, the charcoal is weighed to determine the weight after drying.

Determination of Ash Content (Badan Standarisasi Nasional, 1995)

A total of 1 gram of activated charcoal is placed in a porcelain cup whose mass is known beforehand. The crucible containing the sample was put into a furnace at a temperature of 650°C until ash was formed. After that, the sample was cooled in a desiccator and weighed to determine its mass.

Determination of Iodine Absorption (Badan Standarisasi Nasional, 1995)

As much as 1 gram of activated charcoal was transferred to a dark, closed container. 50 mL of 0.1N iodine solution was put into the container and then shaken for 15 minutes and filtered. The filtrate is then pipetted 10 mL into an Erlenmeyer and then titrated with a 0.1N sodium thiosulfate solution. If the yellow color of the solution is almost gone, then a 1% starch indicator is added. The titration is continued until the endpoint is reached (the blue color just disappears).

Activated Charcoal Modification

Modification of activated charcoal is done by adding chitosan to the activated charcoal adsorbent. The comparison of chitosan and activated charcoal used in this study were: 1:3, 1:1, and 3:1. Chitosan and activated charcoal were dissolved in 60 mL of 2% acetic acid while shaking using a shaker and then added to 0.1 M NaOH solution and shaken for 4 hours. After that, the sample was filtered and immersed in 5.5% glutaraldehyde for 24 hours. Wash until the pH is neutral and then the sample is dried at 60°C until dry. The results were then characterized by FTIR and SEM.

Adsorption Test

Determination of Optimum Contact Time in the Adsorption Process

A total of 20 mg of adsorbent was put into 100 mL of a 20 ppm tetracycline hydrochloride solution. The mixture was then shaken using an orbital shaker at room temperature with various contact times of 15, 30, 45, 60, and 75 minutes. After that, the filtrate was taken from each contact time variation. The levels of tetracycline hydrochloride from each time variation were measured using a UV-Vis Spectrophotometer.

Determination of the Effect of Concentration on Adsorption Power

A total of 20 mg of adsorbent was put into 100 mL of tetracycline hydrochloride solution a several concentrations (10, 15, 20, 25, and 30 ppm). The mixture was then shaken using an orbital shaker for 60 minutes at room temperature. The filtrate was taken from the mixture and the concentration of tetracycline hydrochloride was measured using a UV-Vis spectrophotometer for each concentration variation.

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Determination of Optimum pH Value

The optimal pH value was determined by adding 20 mg of activated charcoal to 100 mL of tetracycline hydrochloride solution at four different pH levels (4, 7, and 10). The mixture was then shaken using an orbital shaker for 60 minutes at room temperature. The filtrate was taken from the mixture, and the concentration of tetracycline hydrochloride was measured using a UV-Vis spectrophotometer for each variation.

The Effect of Using the Sonication Method

The effect of sonication on adsorption power is known by performing the same procedure as the previous procedure. In the activated charcoal immersion step, ultrasonic waves are added.

Result and Discussion

Corncob Activated Charcoal Quality

Activated charcoal material is obtained from the activation process of corncob charcoal. The charcoal activation process is carried out by soaking in hydrochloric acid for 24 hours. Activation using hydrochloric acid is known to improve several properties of charcoal as an adsorbent, including cleaning the pores, expanding the surface, and providing better active groups (Rizkyi , 2016), (Nurbaeti , 2018). The quality of activated charcoal is determined by comparing several parameters such as water content, ash content, and iodine absorption, with the Indonesian National Standard No. 06-3730-1995 regarding technical activated charcoal (Table 1).

Table 1. Result of characterization of corncob activated charcoal

Parameter	SNI 06-3730-1995	Results
Water content	≤ 15 %	4.52 ± 0.28%
Ash content	≤ 10%	4.25 ± 0.17%
Iodine absorption	≥ 750 mg/g	988.35 ± 0.12 mg/g

The determination of water content aims to determine the water content contained in activated carbon after carbonization. The lower the water content, the less water is left and covers the pores of the adsorbent so that the pores can be more open, which results in the maximum absorption for activated charcoal (Rahman , 2020). The corncob activated charcoal produced has a moisture content of 4.52 ± 0.28%, which means it has met the quality standard of activated charcoal based on SNI 06-3730-1995. The low value of the water content of activated charcoal shows that the water content in the raw material has mostly evaporated at the time of activation.

The ash content of corn cob activated charcoal is used to calculate the metal oxide content of the adsorbent. The ash content produced in this study has met the quality standard for activated charcoal based on SNI 06-3730-1995 (Table 1). High ash content can reduce the ability of activated charcoal to absorb gas or solution because the mineral content (metal oxide) contained in the ash will spread in the activated charcoal lattice so that it covers the pores of the activated charcoal. The increase in ash content can occur due to the formation of mineral salts during the cooking process which, if continued, will form fine particles of the mineral salts. This is due to the mineral content contained in the starting material for carbon-producing biomass (Rahman , 2020).

The carbonation process of agricultural waste can affect the ash content of the activated charcoal obtained. The use of an oven in the carbonation process produces a lower ash content compared to using hot coals. The carbonation process of corn cobs using coals has been carried out with an ash content of 8.92% (Rizkyi , 2016). The use of an oven can produce cleaner charcoal so that there are no impurities in the form of fine particles that can potentially lead to further oxidation processes. This further oxidation process causes a higher ash content.

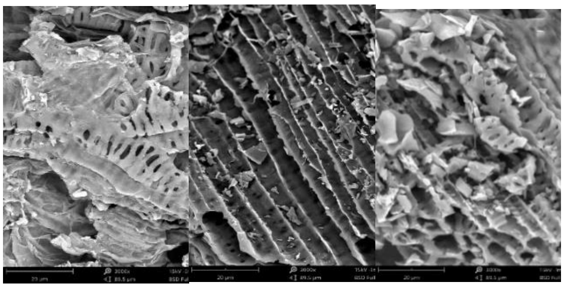
Testing the absorption of iodine on activated charcoal aims to determine the adsorption ability of colored solutions. The absorption of activated charcoal by iodine is the number of milligrams of iodine adsorbed by one gram of activated carbon (Erawati & Fernando, 2018). The activated charcoal obtained from this study has an iodine absorption capacity of 988.35 ± 0.12 mg/g. The magnitude of the iodine absorption value of activated charcoal is caused by the C and H bonds that are completely released so that there is a shift in the crystallite carbon plate to form new pores and expand the already formed pores. The adsorption power of activated carbon on iodine correlates with the surface area of the activated carbon. The greater the iodine number, the greater its ability to adsorb adsorbates or solutes (Rahman , 2020).

Chitosan Modified Activated Charcoal Characterization Results

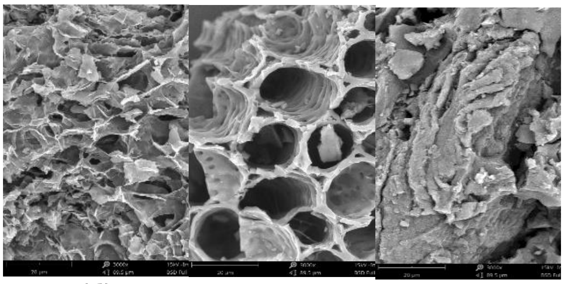
Activated Charcoal Surface Morphology

The surface morphology of the activated charcoal produced can be seen using SEM with an object magnification of 3000 times. The given treatment can provide a significant change in the surface morphology (Fig. 1).

The combustion process that converts corn cobs (Fig. 1a) into charcoal (Fig. 1b) can change the surface morphology. The resulting charcoal has more visible and regular pores. The activation process of charcoal into activated charcoal makes the pores that are formed more open because the water molecules that are trapped in the pores can be attracted by hydrochloric acid in the activation process (Verayana , 2018).



(a) (b) (c)



(d) (e) (f)

Figure 1. Surface morphology of (a) corn cobs, (b) charcoal, (c) activated charcoal, (d) chitosan-activated charcoal ratio 1:3, (e) chitosan-activated charcoal ratio 1:1, and (f) chitosan-activated charcoal ratio 3:1

A chitosan coating on activated charcoal material can affect the surface morphology of the activated charcoal. The ratio of chitosan-activated charcoal 1:1 (Fig. 1e) has the largest pores, and the regularity is very clear. The other comparisons do not show clear pores, even at the ratio of chitosan-activated charcoal 3:1 (Fig. 1f) a very closed surface is formed, and there are almost no visible pores in it. In general, chitosan added to activated charcoal must have the same ratio to get the expected pores. Chitosan coating can cause a rougher surface and the presence of crystalline substances that can enter and cover the pores. In addition, the addition of chitosan can also reduce surface area, pore distribution, and pore volume, but it has the potential to increase pore size (Shi , 2020).

Adsorbent Functional Group

The functional groups formed on the adsorbent can be characterized using FTIR. This characterization was to determine the effect of burning corn cobs and the effect of charcoal activation using hydrochloric acid (Fig. 2).

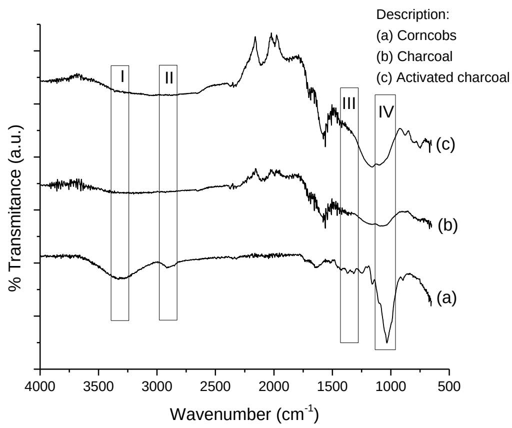


Figure 2. Effect of Corncob Burning Process and Corncob Charcoal Activation

The process of burning corn cobs can change the chemical structure of the corn cobs. Regions I and II (Fig. 2) indicate that the -OH or -NH group (3332 cm^{-1}) and the C-H group (2916 cm^{-1}) can be decomposed or separated from the surface of the material. The absorption at this wave number was not seen in the charcoal sample (Figure 3b). The absorption also remained invisible in the charcoal samples which had been activated using hydrochloric acid. Region III (Fig. 2) is the absorption of the alkene group (1653 cm^{-1}). The presence of C-O or C-N groups was seen in the spectra of corn cobs (Figure 3a), which was indicated by the absorption at a wave number of 1032 cm^{-1} (Region IV). The absorption is not visible in charcoal and activated charcoal, so the combustion process can break down these groups.

Activated charcoal that has been successfully made needs to be modified using chitosan to increase its ability to absorb adsorbed materials. The addition of chitosan to activated charcoal aims to increase the active groups on the surface of the activated charcoal adsorbent (Fig. 3). The process of coating chitosan onto activated charcoal can add active groups that have the potential to increase interactions with tetracycline hydrochloride compounds as adsorbates. Functional groups that appear include the -OH, -CH, and C-O or C-N groups. The more chitosan added, the clearer the absorption of the functional groups. The ratio of chitosan-activated charcoal 3:1 (Figure 4d) clearly shows the absorption in regions I, II, and III, namely at wave numbers 3339 cm^{-1}, 2927 cm^{-1}, and 1028 cm^{-1} (Sharififard , 2013). Thus, the activated charcoal coating process using chitosan can be said to be successful.

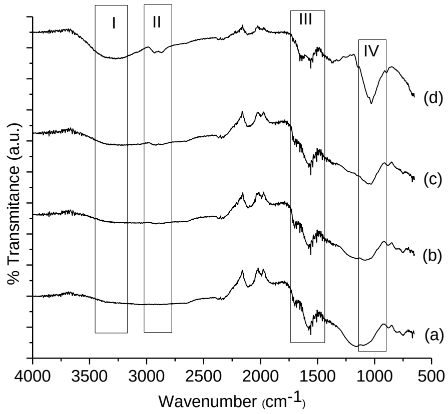


Figure 3. Effect of Addition of Chitosan into (a) Activated Charcoal with a ratio of Chitosan-Activated Charcoal (b) 1:3, (c) 1:1, and (d) 3:1

Adsorption Mechanism

The adsorption mechanism was studied using adsorption kinetics and adsorption isotherms. Kinetics and adsorption isotherms were used to observe the adsorption process following the Lagergren, Ho, or Santosa equations for adsorption kinetics and Freundlich or Langmuir equations for adsorption isotherms.

The adsorption kinetics are obtained by varying the contact time between the adsorbate and the adsorbent in the adsorption process. The effect of contact time is determined by the time required by chitosan-modified activated charcoal. The use of the sonication method can streamline the time required for the adsorption process. The sonication method is known to require a relatively shorter time to achieve optimal absorption compared to the batch method (Fig. 4).

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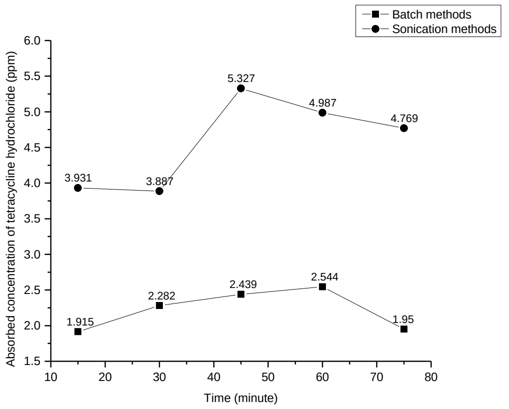

Figure 4. Concentration of tetracycline hydrochloride compounds adsorbed by the adsorbent with variations in contact time

Contact time is one of the factors that affects the amount of target compound that can be adsorbed by the adsorbent. Based on Figure 4, the two methods used, namely batch and sonication, had different results with an initial concentration of 20 ppm. The sonication method can absorb tetracycline compounds more optimally (5.327 ppm) in a shorter time (45 minutes). The decrease in the concentration of the adsorbed tetracycline after the optimum time is due to the desorption process because the surface of the adsorbent has been saturated so that it is no longer able to interact with tetracycline, which will affect the adsorption capacity (Jubilate , 2016).

The adsorption process of tetracycline hydrochloride compound by chitosan modified corn cob activated charcoal (Table 2) resulted in different adsorption kinetics between the batch method and the sonication method. The adsorption process in the batch method followed the equation Ho (R^2 = 0.934) with an adsorption capacity of 2.092 mg.g^{-1}. The adsorption kinetics in the sonication method also followed the equation Ho (R^2 = 0.968) with an adsorption capacity of 5.330 mg.g^{-1}. The kinetics of Ho are the mechanism for the pseudo two. Because Santosa's kinetics has a low R^2 value, the adsorption kinetics do not match. In the Lagergren kinetic equation, both methods produce a negative adsorption rate constant. Thus, the adsorption kinetics that occur only correspond to the kinetics of Ho. The sonication method can increase the adsorption capacity from 2.092 mg.g^{-1} to 5.330 mg.g^{-1}.

Table 2. Comparison of adsorption kinetics using batch and sonication methods

	Batch methods	**Sonication methods**
Ho equation	y = 0.478x + 1.212	y = 0.188x + 1.106
	R^2 = 0.934	R^2 = 0.968
	q_e = 2.092 mg.g^{-1}	q_e = 5.330 mg.g^{-1}
	k = 0.188 g.mg^{-1}.min^{-1}	k = 0.035 g.mg^{-1}.min^{-1}
Santosa equation	y = 0.0002x + 0.006	y = 0.0015x + 0,013
	R^2 = 0.049	R^2 = 0.482
	k = 0.006 min^{-1}	k = 0.013 min^{-1}
	K = 0.0002 L.ppm^{-1}	K = 0.0015 L.ppm^{-1}
Lagergren equation	y = -0.002x + 4.597	y = -0.0002x + 4.567
	R^2 = 0.974	R^2 = 0.462
	k = -0.002 g.mg^{-1}.min^{-1}	k = -0.0002 g.mg^{-1}.min^{-1}
	q_e = 99.147 mg.g^{-1}	q_e = 96.255 mg.g^{-1}

The adsorption isotherm was obtained by varying the initial concentration of the tetracycline hydrochloride compound that was absorbed by the adsorbent. The adsorption process that occurs in this study corresponds to the Langmuir isotherm when using the batch method (R^2 = 0.950), while the Freundlich isotherm occurs when using the sonication method (R^2 = 0.859).

Corn cobs have been widely used as research materials as adsorbents by modifying them into activated charcoal or activated carbon. The adsorption process by activated charcoal from corn cobs generally corresponds to the Ho or pseudo-second-order adsorption kinetics and the Langmuir adsorption isotherm (Table 3).

Table 3. Kinetics and Adsorption Isotems of several adsorption processes by activated charcoal from corn cobs

Adsorbate	Adsorption kinetics	Adsorption isotherm
Potassium permanganate (Getachew , 2015)	-	Langmuir (R^2 = 0.860)
Flouride (Gebrewold , 2019)	Pseudo-second order (R^2 = 0.9954)	Langmuir (R^2 = 0.9828)
Hg(II) (Z. Liu , 2020)	Pseudo-second order (R^2 = 0.99985)	Langmuir (R^2 = 0.9998)
Dye in sewage (Sun , 2021)	Pseudo-second order (R^2 = 0.9953)	Langmuir (R^2 = 0.9417)
Methylene blue (Medhat , 2021)	Pseudo-second order (R^2 = 1.00)	Langmuir (R^2 = 0.99)
Mercury Ions (Yuyingnan Liu , 2021)	Pseudo-second order (R^2 = 0.9917)	Langmuir (R^2 = 0.9939)

Adsorption Ability in Various Acidic Conditions

One of the important factors affecting adsorption is the acidity of the solution. Acidity influences the adsorption ability of a compound. The acidity will affect the charge at the active site and the charge of the ions in the solution. Under acidic conditions, the functional groups on the adsorbent will be easily protonated (Jubilate , 2016). The optimum absorption of the tetracycline hydrochloride compound occurred at pH 4 (Fig. 5). At pH 10 adsorption caused a very sharp decrease because the increase in the concentration of the –OH group as the pH of the solution increased caused the tetracycline hydrochloride compound to be in the solid phase (precipitate) (Ariyani , 2018), so that it could not be maximally adsorbed by the adsorbent.

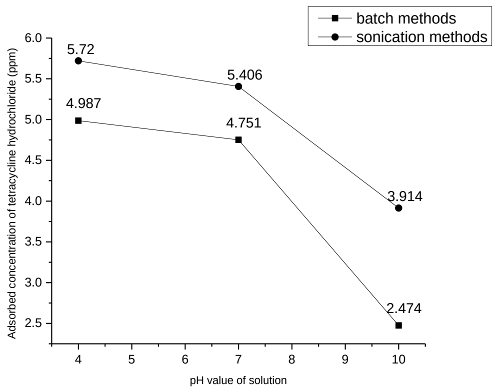

Figure 5. Adsorption of tetracycline hydrochloride compounds at various pH

Conclusion

Corn cobs waste can be used as an adsorbent for antibiotic compounds. Corn cobs can be converted into activated charcoal, which is then combined with chitosan to increase the adsorbent material's absorption capacity. The adsorption process was carried out by two methods, namely the batch method and sonication. The addition of the sonication method can increase the adsorption capacity by 323.80%. Activated charcoal from corn cobs has the potential to be used as an alternative to reduce antibiotic waste in the aquatic environment. Thus, pollution of the aquatic environment can be controlled to the greatest extent possible.

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