Adsorption Of Ciprofloxacin Antibiotics By Azadirachta Indica (Neem Leaf) And Their Characterisation

Payal Maharathi

***Abstract:** Adsoprtion of ciprofloxacin antibiotics using neem leaves biochar was studied. The neem leaves biochar had relatively BET specific surface area that was 8.6 m^2/g. In this research, the removal of ciprofloxacin was effective using the biochar.The adsoprtion occurred at the homogeneous sites of the biochar and obeyed langmuir model.The maximum removal efficiency was 70% of ciprofloxacin against 0.2g of biochar .Biochar is used for water remediation and for treating of water and wastewater.The biochar was prepared from pyrolysis that may fast or slow.The different removal efficiency was detected with various experiment. The characterisation of the biochar using SEM analysis showed the adsoprtion of ciprofloxacin in the non unifrom pores of the biochar..In this study biochar was produced at 250°C from neem leaves is seen as effective temperature for adsoprtion of the ciprofloxacin antibiotics. The samples were examined using UV-VIS spectrophotometer and it shows a good removal efficiency.*

Index Terms: *Adsorption, Biochar, Ciprofloxacin*

I. INTRODUCTION

Antibiotics are broadly used in medicinal treatment to humans for infections or bacterial treatment .The involvenment of ciprofloxacin antibiotic in surface waters have poorly interpreted.The presence of pharmaceuticals products have poorly understood .Water and wastewater treatement plants elliminate some few percentage of pharmaceuticals. The antiobiotics donot breakdown naturally in the environment.. Ciprofloxacin is an antibiotic compound that is used for several health problems like headaches, nervousness, nausea; vomiting etc…Ciprofloxacin is the second creation of quinolones. Ciprofloxacin present in drinking water if it is at more concentrations then it may cause major effects on the health. Ciprofloxacin fall under the fluroquinolone group. The presence of ciprofloxacin is not only adding poisonous and polluting substance to the water sources but also increased the risk to life. Ciprofloxacin plays an important responsibility in treatment guidelines given as per the major medical societies for treatment of infections, viruses and those infections which are caused due to gram- negative bacteria including Pseudomonas aeruginosa. But it gives attention to both gram positive and gram -negative bacteria. The amount of ciprofloxacin present in various sources of water is increasing as the need of the antibiotic is increasing by the population which is produced by the pharmaceutical industry. There are different methods available to treat wastewater containing antibiotics like chemical oxidation, biodegradation, adsorption, liquid extraction and membrane techniques. The adsorption process is more promising because it is presenting no difficulty in design and is simple to perform.Also, it is cost effective, highly effective, eco-friendly and produces less toxic. Emerging contaminants (ECs) are unstructured or not completely structured compounds even in the most developed countries and that can be dangerous to the environment and human health .Emerging contaminants are such as, pharmaceuticals and personal care products etc... have been discharged by household's activities and industries. Biochar is charcoal highly rich in carbon content and biochar is made from the biomass. Temperatures between 200 to 700°C produce more biochar through pyrolysis method. The physical and chemical properties of the biochar are decided according to the feedstock material and the technology for the production of the production of the biochar. The various impacts of the biochar is dependent on the properties of the biochar and the amount of the biochar that will be used. Activated carbon is good adsorbent for the removal of contaminants from water but it is quite very expensive. Sustainable environment requires less investment for treating of the pollutants that are having effect on the environment. Biochar absorb heavy metal ions and many other organic pollutants which show a good potential for purification of the water. There are several adsorbents among those biochar has shown good application for the adsorption removal of the organic pollutants and has become popular for water treatment. The contamination of the groundwater by the antibiotics compound is a great problem. TORCs are directed to the environment through human excretion and animal feeding operations.The presence of antibiotics in drinking water sources need to be investigated properly and the presence of various drugs that cause death to the microorganisms which are very much essential for the treatment plant and they decompose the organic material which are present in the effluent.

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Biochar are prepared from the material those are biodegradable products and the adsorption capacity of the biochar considered with other types of the adsorbent are more effective and removes a higher percentage of compound for treating of water. The biodegradable waste from household is increasing those waste can be used for the production of the biochar which will have no negative impact towards the environment.This study investigated the adsorption of ciprofloxacin antibiotics using biochar derived from neem leaves. The objective of this study was to find out the maximum removal efficiency, the optimum the temperature of the biochar and the optimum time. The adsorption isotherms were done to understand the adsorption.

II. EXPERIMENTAL

Materials

The ciprofloxacin was purchased from the Sigma Aldrich.100mg/l of standard solution was prepared using a buffer solution. The buffer solution was prepared using the methanol and sodium hydroxide diluted in water. Neem leaves were collected.

Biochar Preparation and Adsorption Procedure

The neem leaves were collected and washed with acetone then dried.10g of neem leaves was measured using weighing machine. The pieces of the neem leaves were taken in the crucible having a lid which tightly fitted and carried out for pyrolysis process. The biochar was produced at two different temperatures that are 300°C and 250°C. The biochar produced at 300°C turned into fly ash .The biochar after pyrolysing for a certain time period it was kept inside the muffle furnace to cool. Then crucible was removed and stored inside the desiccator.The biochar produced at 250°C was used for experiments.

The stock solution was prepared and with the help of stock solution the standard solution was prepared that is 100mg/l. Three different experiments were done.

- 0.2g of biochar which was added with 100ml of ciprofloxacin standard solution in four different flasks. Four sets of the conical flask were placed in the shaker at 80rpm speed for different shaking time that is 20minute, 30minute, 40minute and 50minute.
- Different concentration of biochar was added with 100ppm of standard solution in four different flasks were placed in the shaker at 80rpm .
- 100ppm of standard solution was spiked over 0.2g of biochar produced at 250°C at different pyrolytic time was added in four different flasks were placed in the shaker at 80rpm for constant shaking time .

The solution was containing some few biochar after filtration in the solution for complete removal of the biochar; the solution was placed in the micro-centrifuge tubes. All the tubes were centrifuged at 15000rpm for 5minutes which allowed the biochar to settle. At 296nm wavelength the samples were analysed using UV-VIS spectrophotometer.

Percentage of removal :-$(C0 - Ce) \times 100/ C0$

where Ce and C0 are final and initial concentration of ciprofloxacin respectively. Amount of adsorption was calculated by using the equation $(C0 - Ce) \times v/w$, where w is the weight of the adsorbent used v is the sample volume.

III. RESULT AND DICUSSION

Biochar preparation was carried out and the adsorption process was conducted. Organic material that was collected and was prepared at different pyrolytic temperature i.e. at 250°C and 300°C. The organic material was kept inside the muffle furnace for a certain period of time which is an important factor for the preparation of biochar. Biochar cannot be produced at a higher pyrolytic temperature from the organic material but it should be below 700°C.The pyrolytic temperature varies for different feedstock for the conversion of the biomass into biochar. The pyrolytic temperature is a neccessary factor.

Characterisation of biochar

Moisture content

8g of neem leaves were taken in a crucible and kept in the oven at 140ºC for one hour then the moisture content of the biochar was found to be 93.2%.

Ash content

6g of neem leaves were taken and kept inside the muffle furnace at 400ºC then the ash content of the biochar was found to be 11.72%.

Volatile matter content

6g of neem leaves were taken and placed inside in the muffle furnace at 400ºC for one hour then the volatile matter content was found to be 47.56%.

Biochar yield

The biochar yield of the neem leaves biochar was found to be 0.896.

Determination of removal efficiency different shaking time

The adsorption efficiency of the neem leaves was determined using a UV-VIS Spectrophotometer at 296nm.

Ciprofloxacin adsorption experiment was carried out using the neem leaves biochar that was produced at different temperatures i.e. 250°C was shaken for different time i.e. 20minutes, 30minutes, 40minutes and 50minutes in shaker gave different adsorption results.

The fig-1 show that the biochar that was produced at 250°C showed good removal efficiency of ciprofloxacin which is around 70% at 20 minutes. Hence the optimum time of adsoprtion is 20 minutes.

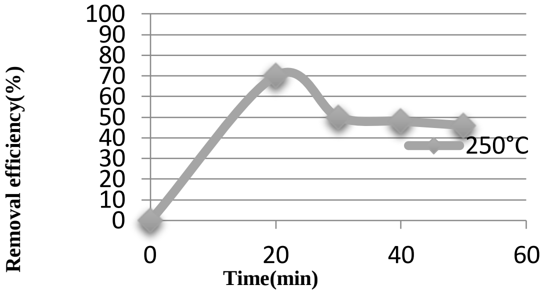

Fig1:- Removal efficiency v/s shaking time

Effect of different concentration of biochar

As shown in fig-2, ciprofloxacin adsorption on neem leaf biochar was minimum at 0.2g. All the conical flasks were shaken for a constant shaking time that is 20minutes.

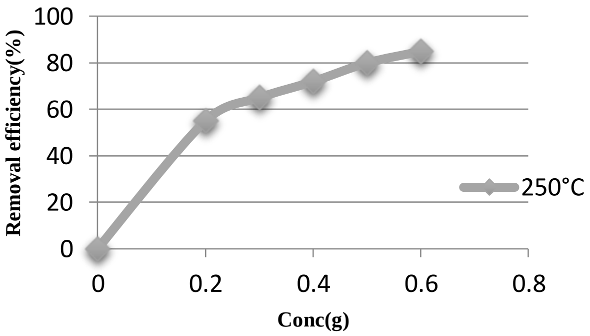

Fig 2 :- Removal efficiency v/s concentration of biochar

Effect of different pyrolytic temperature with different time kept inside the muffle furnace

As shown in fig- 3, the ciprofloxacin adsorption was maximum at 50minutes. The biochar with the standard solution was shaken for 20minutes.

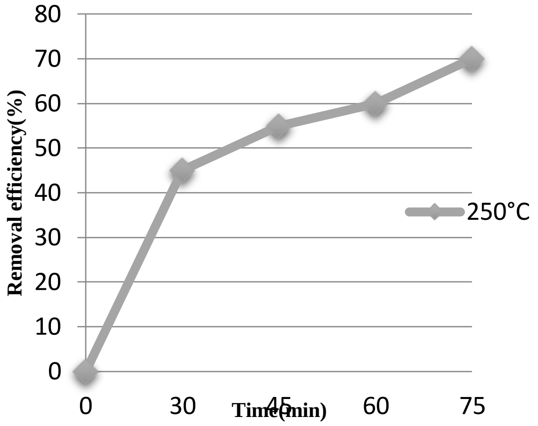

Fig 3:- Removal efficiency v/s heating time

Characterisation Results

The maximum removal efficiency of the ciprofloxacin is at 250°C for 20minutes of shaking so further analysis was carried out .The FT-IR spectra were analyzed to find the characteristic functional group of neem leaf biochar. The FT-IR spectra were analyzed from 500 to 4000cm^{-1} infrared spectral region of the neem leaf biochar before and after adsorption are shown in fig- 5. There are some peaks that are shown in the infrared spectra of the neem leaf biochar. According to the graph, the functional groups including the stretching vibrations of –OH at 3300 cm^{-1}, at 1550 cm^{-1} the C=C bond in the aromatic ring, at 1350 cm^{-1} the C-H alkanes and at 1200 cm^{-1} C-O in phenol are clearly identified. There are many peaks that are 2950,2900 shows symmetric and asymmetric stretching vibrations of Methylene. Compared to the infrared spectra after adsorption result, some of the peaks that are shown are 2970,1700,1200 cm^{-1} are assigned to N-H stretching vibration, secondary amine group, bending of methyl, aromatic nitro compounds-OH stretch, C-H stretching vibrations of phenolic, C=O stretching. The band frequencies of biochar after adsorption show a different change in the sorption band at 3300, 1550, 1350 and 1200 $cm^{-1}$are N-H stretching vibration , the C=C bond of the aromatic ring , a secondary amine group, symmetric bending of CH_3, bending vibration of methyl-H stretching vibration, C-OH stretch, C-O in phenol, C=O stretching play a vital role in ciprofloxacin adsorption.

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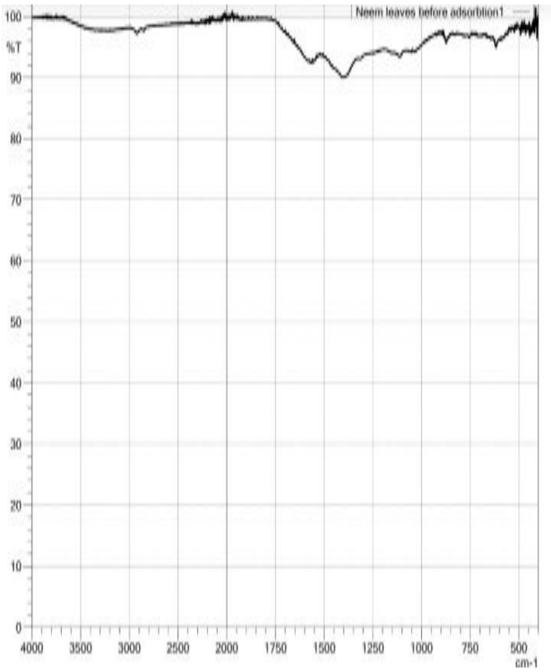
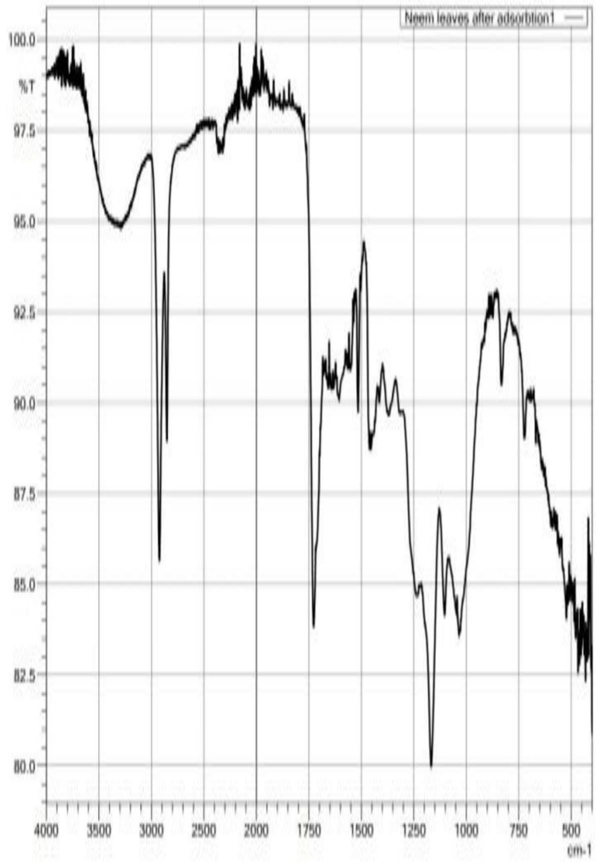
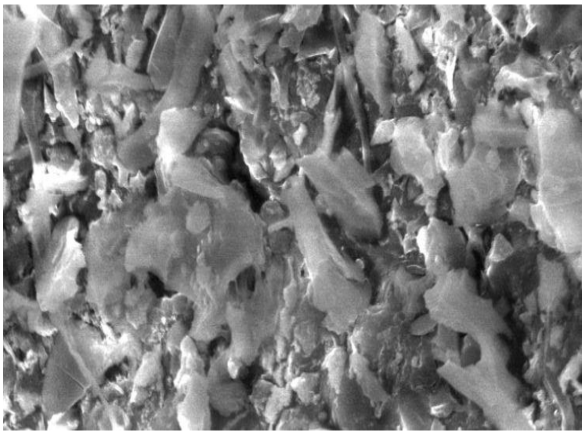


(A)



(B)

Fig 5:- FT-IR Result before and after adsorption



(A)

Fig 6:- SEM images of the biochar before and after absorption of the compound

From BET analysis,the average pore diameter, the pore capacity and the surface area of the biochar after adsoprtion were 4.44nm, 0.012cm^3/g and 8.6m^2/g. The morphology of the biochar before and after adsorption was different**.**

Adsorption isotherm

For these isotherm experiments, the biochar varying concentration experiment was considered. The experiment was conducted with the biochar that was produced at 250°C. The Langmuir model and the Freundlich model are the isotherm models that are studied for the experiment.

The Langmuir and freundlich equations are shown below;

$$\frac{1}{Q_e} = \frac{1}{Q^O} + \frac{1}{KQ^0C^e} \quad \ldots \quad (1)$$

Qe = KF * Ce^(1/n) … (2)

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Where Q_e is the adsorption capacity per unit mass of adsorbent,
K_f is the freundlich coefficient,
n is the freundlich constant
C_e is adsorbate concentration in the aqueous solution,
Q_0 is the adsorption capacity
K is the surface adsorption affinity constant

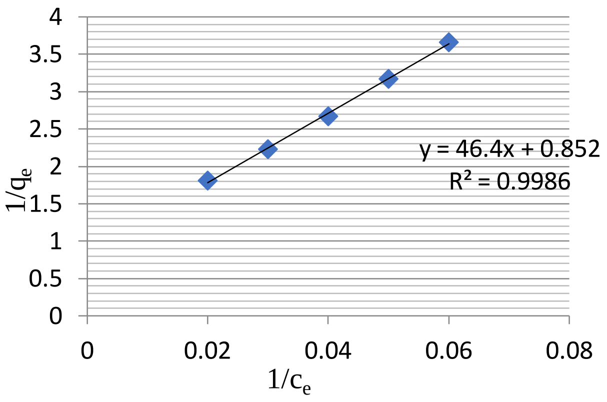

Fig 7 :-Langmuir model for 250 ^{0}C biochar

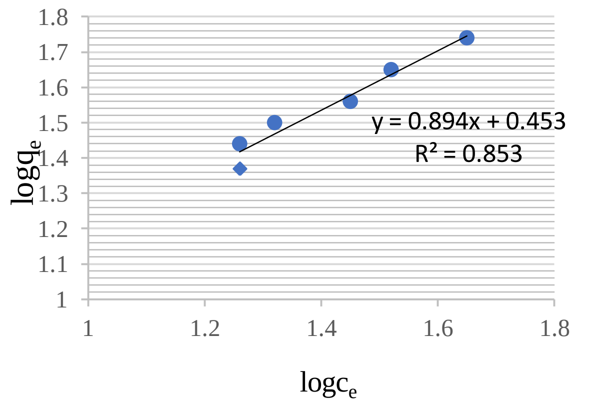

Fig 8:-Freundlich model for 250 ^{0}C biochar

The Freundlich isotherm R^2 value is 0.853 .The Langmuir model R^2value is 0.998 gives the best fit . The value of n should be greater than 1 ,according to this experiment ,the freundlich constant is greater than 1 suggesting ciprofloxacin was easily absorbed on the surface of the biochar.

IV. CONCLUSION

This experiment was conducted with an aim to find a economical way to removal of ciprofloxacin antibiotic that is destroying and polluting the various water sources. The neem leaf material was used for the preparation of biochar and further experiment was done.. The removal efficiency was found to be 70% at 20minutes using the biochar produced at 250°C.

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