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# Adsorptive Removal of Metformin from Aqueous Solutions Using Agro-Waste Materials

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***Abstract*: Water contamination is a critical global issue, contributing to thousands of deaths and widespread diseases. Aquatic pollution, or environmental degradation caused by the massive influx of pollutants into water systems, has significantly diminished water quality. The introduction of harmful chemicals from various sources has compromised the integrity of these water resources. As a result, the entire ecosystem is at risk, with detrimental effects on both plant and animal life. Polluted water not only disrupts aquatic environments but also threatens the broader biosphere. So to resolve this problem agro waste material has been used in this study. Activated carbon was prepared from seeds of *Azadirachta indica.* Physical activation was done for preparation of activated carbon. Characterisation of activated carbon carried out by FTIR and FESEM. Highly porous structure of activated carbon responsible for the adsorption of pollutant from aqueous solution. Batch experiment was conducted for studying the effect of various parameters on the adsorption of metformine.**

***Key Words:* Activated Carbon, Bionanocomosite, Pollutant, Nanoparticle**

## I. INTRODUCTION

Our environment composed of living and non living components. Life on earth cannot be imagined without water. Survival of human beings depends upon three main resources – water, air and soil. These three resources are the valuable gifts from nature to human beings. Among these water is the main resource, responsible for origin of life [1]. The contamination of water bodies such as lakes, oceans, and groundwater is known as water pollution. The entire biosphere which includes plants and living organisms affected by polluted water. Most precious natural resource which is also responsible for the existence of life on earth is water. Only 3% of the water on Earth is fresh water, with nearly two-thirds of it frozen in glaciers and polar ice caps. Ground water is found as un frozen freshwater on this earth. Due to increase in population and industrialization in India the demand for water increased. We can't imagine life without water on earth. The quality of water resources had been declined due to addition of toxic chemical compound directly or indirectly [2].

Water contamination is a foremost global concern and is responsible for thousands of death and diseases. The aquatic pollution generally referred as environmental degradation due to massive amount of pollutants released in water resources. The quality of water system has been declined due to the addition of noxious chemicals from various sources [3]. For water, air and soil contamination urbanization, progress in industries and excess use of various resources that may be natural or man- made are mostly responsible [4,5,6,7].

Various techniques has been developed for removing pollutants from waste water. It includes physical, chemical and biological methods. Nanocomposite is a solid phase material having size less than 100nm.The metal oxide nanoparticles already had been explored in different technological sectors. Recently production of nanomaterial with enhanced affinity, adsorption efficiency and selectivity for pollutants have gained much attention [8,9]. Carbon based composites has been explored for removing pollutants from waste water due to their chemical strength, structural range, low thickness and easy availability. *Azadirachta indica*, commonly known as neem, tree. *Azadirachta indica* plant belongs to family Meliaceae and is native to subcontinent India and most of countries of Africa. The products produced from neem tree have been used in traditional medicines of India for century. The activated carbon 'A' was prepared from seeds of *Azadirachta indica* (Neem). Activated carbon 'A' was haracterized by FTIR and FESEM. Then metal ferrites were haracteriz by co-precipitation method.

The metal ferrite was haracterized by FESEM, FTIR techniques. Bionanocomposite was prepared by mixing together activated carbon 'A' and silver nanoparticles and metal ferrites by refluxing method. Bionanocomposite was haracterized by FTIR, FESEM techniques. In this research paper we describe the preparation of activated carbon from agro waste material and their role in the removal of antidiabetic drug metformine from aqueous solution through adsorption process.

## 2. MATERIAL AND METHOD

### 2.1 Materials

All of the substances used in this experiment were of analytical grade. All chemicals such as Metformine (>95% pure, IUPAC: N, NDimethylimido-dicarbonimidic-diamide, Molecular weight-129.16364 g mol$^{-1}$, Molecular formula-$C_4H_{11}N_5$, $pK_1$-2.8, $pK_2$-11.5), hydrochloric acid (HCl), sodium hydroxide (NaOH), potassium chloride (KCl), were of analytical grade and purchased from CDH Pvt. Ltd., India. The stock solution (1000 mg/L) of Metformine was prepared by dissolving the accurate amount of Metformine in distilled water. By successive dilution of stock solution, working solution of desired concentrations was obtained. Using double distilled water all dilution and washing carried out. A digital pH meter (Elico LI- 10, India), UV-visible spectrophotometer (*Shimadzu Spectrophotometer*), Fourier transform infrared spectrophotometer (Perkin Elmer Spectrum-BX USA), scanning electron microscope (Nova-Nano SEM-450), and rotary flask shaker were used. The laboratory work performed in the chemistry lab. The waste agro materials required for the activated carbon collected from Joghon village at Solan district of Himachal Pradesh. These agrowaste material composed of hemicelluloses, lignin, lipids, proteins, extractives and starch containing functional groups. Hence these are selected as bio sorbent for environmental detoxification.

### 2.2 Preparation of Activated carbon 'A'

*2.2.1Preparation of Biosorbent 'A' from seeds of Azadirachta indica*

The seeds of *Azadirachta indica* were collected and washed repeatedly with double distilled water. Washing of seeds of *Azadirachta indica* continued until colourless water was obtained. After washing biomaterial was collected and desiccated in oven at $60^0$C for 48 hours. Dried material then crushed into small pieces. After crushing, the biomaterial was broken in harmonized sizes ranging from 0.3 mm to 0.6 mm. Shattered biomaterial was kept in desiccators for future study.

*2.2.2Preparation of Activated carbon 'A' from seeds of Azadirachta indica*

The dried, cleaned and smashed biomaterial of *Azadirachta indica* was transformed into activated carbon. The shattered biomaterial kept in muffle furnace for one hour at $600^0$C. After one hour activated carbon of seeds of *Azadirachta indica* was obtained. The activated carbon of seeds of *Azadirachta indica* stored in a neat and cleaned polybags.

*2.2.3Preparation of Starch stabilised Silver nanoparticles*

Silver nanoparticles using starch were prepared through reduction of silver ($AgNO_3$, 0.001 M) by utilizing D+ glucose (0.1 M) as reaction catalyst and stabilizing agent soluble starch. 40 ml of starch solution added into $AgNO_3$ solution (20 ml). After mixing both solution, kept the resulting solution in ultrasonicator apparatus for 15 minutes. It was followed by the addition of (0.5 ml) of D+ glucose solution and 3 ml of NaOH solution.

Reaction mixture was kept in sonicator at 30 degree centigrade for 60 minutes. Colour of solution changed immediately from colourless to pale brown in sonicator and after sometime to light yellow. The light yellow colour indicated the formation of nanoparticles.

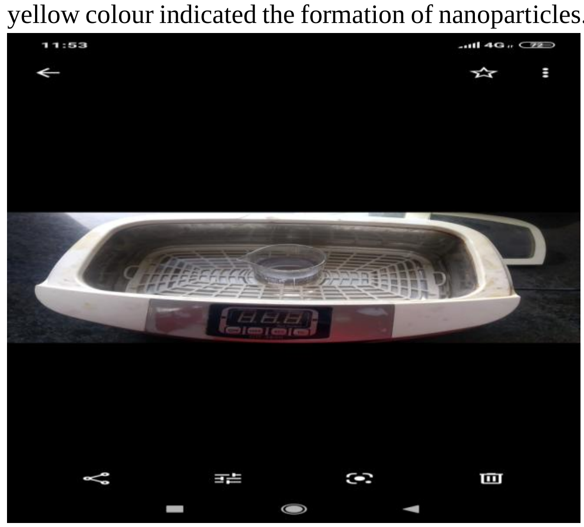


*Figure 3.6 Ultra sonicator*

*Figure 3.7 Silver nanoparticle*

*2.2.4Preparation of Metal Ferrite*

Synthesis of metal ferrite i.e.Cobalt ferrite ($CoFe_2O_4$) was carried out by co-precipitation method. Solutions of 0.1M Cobalt nitrate[$Co(NO_3)_2$] and 0.2M Ferric nitrate [$Fe(NO_3)_3$] prepared in 50 ml. water and then mixed . Add 6-8 drops of lemon juice and NaOH (20% solution) to maintain pH(7) with constant stirring at $50^0$C for half hour. Reflux the obtained solution for 6 hours at 80-$90^0$C at magnetic stirrer. Filter the solution and place the obtained precipitate in oven. The product obtained was cobalt ferrite.

*2.2.5Synthesis of C/Ag/$CoFe_2O_4$*

Bionanocomposite-1 composed of silver nanoparticles, cobalt ferrite and activated carbon of *Azadirachta indica*. Ag/C/$CoFe_2O_4$ has been prepared through single step simplistic refluxing technique. 1g of porous activated carbon from *Azadirachta indica* (Neem) seeds was added to 50 ml of double distilled water and physically agitated for 40 minutes to generate a homogenous mixture in this technique. In this procedure 1g of porous activated carbon of *Azadirachta indica* (Neem) seeds was added into 50 ml of double distilled water and mechanically stirred for 40 min. to form homogeneous mixture.

0.5 gm Silver nanoparticle and 1 M Cobalt ferrite were added into the above homogeneous mixture in a round bottom flask. The resulting mixture vigorously stirred on magnetic stirrer with magnetic bead along with refluxing for 6 hrs by maintaining temperature $70^0$C.The obtained sol was then filtered, washed with distilled water for removing impurities. After removal of impurities, resulting sol was dehydrated in vacuum oven $50^0$c. Consequently after drying the resulting product heated into muffle furnace at $450^0$c for 20 minute in order to assist incorporation of $CoFe_2O_4$ and Ag particles into carbon matrix.

2.3 Characterisation

Chemistry relies heavily on the identification of chemical substances. Characterisation strategies aid in the identification of substances with unknown structures. Characterisation of activated carbon 'A' was carried out by FTIR and FESEM.

*2.3.1 Fourier Transform Infrared Spectroscopy*

This technique identified chemical bonds present in a molecule. FTIR technique considered to be first step in the material analysis process. This technique used for analysing chemical composition of smaller particles, typically 10 – 50 microns, as well as larger area on the surface. FTIR of Activated carbon 'A' was measured using the KBr disc method on a Fourier transform infrared absorption spectrophotometer. Activated carbon'A' was mixed well with KBr pellets, powdered, and pressed into a disc. The spectra of absorption were recorded.

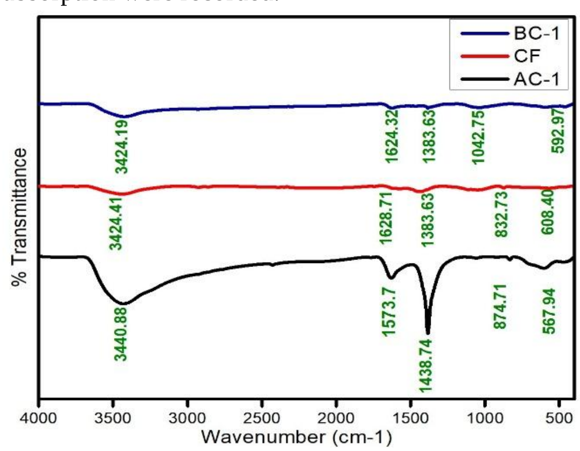


*Figure.1 FTIR of Activated Carbon, Metal ferrite and bionanocomposite*

Seed of (*Azadirachta indica*) neem is a heterocyclic compound containing many functional groups. The absorption band at 3400 show the polymeric OH stretch and at 1430 show the presence of methyl group, at 2860-2924 show symmetric and asymmetric stretching of CH of $CH_2$, at1573 show presence of carboxylate salts, at 874 show C-C stretching, at 567 show OH out of plane .Ferrite is a spinal structured material with which is iso structural with mineral $MgAl_2O_4$ and have general formula ($M^{2+}$) [$Fe_2^{3+}$]$O_4^{2-}$ ,with $M^{2+}$ is divalent and $Fe^{3+}$ is trivalent cation. The absorption bands around 3400cm$^{-1}$ and

1600cm$^{-1}$ are ascribed to the stretching mode of O-H and H-O-H bending vibrations. At 3424 .19 cm$^{-1}$ show polymeric OH stretching. It also shows the presence of intramolecular hydrogen bonds in the molecule. At 1624.32 cm$^{-1}$ show C=C stretching vibrations in which double bond non- conjugated to each other. At 1383.63 cm$^{-1}$ show the presence of methyl group in the bionanocomposite – 1. At 1042 cm$^{-1}$ show the presence of secondary cyclic alcohols.An absorption peak at 592 cm $^{-1}$ show stretching vibrations of metal .

*2.3.2 Field Emission Scanning Electron Microscope*

FESEM is Field Emission Scanning Electron Microscope. It is an advanced technology used to capture microstructure image of the materials. It provides topographical and elemental information at high magnifications. FESEM is a microscope that works with electrons not with light. These electrons liberated by an emission source.

The Nova NanoSEM ™ electron microscope scanner delivers excellent class thinking and analytical performance with a single, easy-to-use tool. Improved optics and acquisition, which includes beam reduction, in lens ETD (SE), TLD (custom), DBS lens and LVD offer the most excellent assortment of information and image processing. Provides 1.6 nm correction at 1 kv and <1 nm at 15 kv.

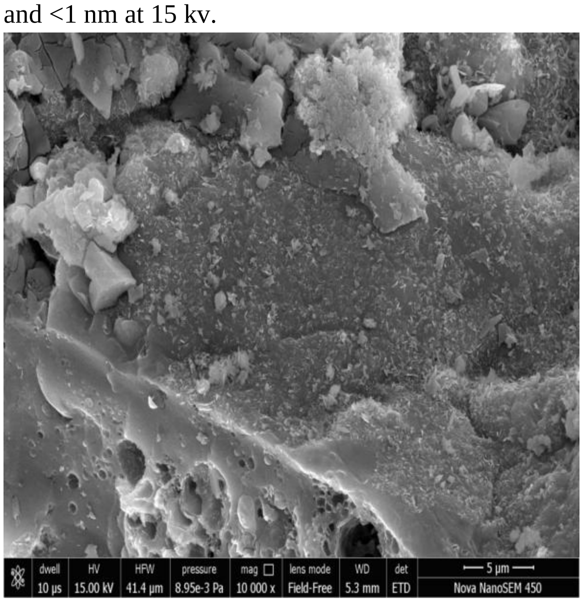


*Figure.2 FESEM of activated carbon ‘of seeds of Azadirachta indica*

2.4 Batch Experiment

Using batch adsorption experiments adsorption of Metformine on to activated carbon -1 was evaluated. Erlenmeyer flasks having capacity of 100ml was used for performing these experiments. The Erlenmeyer flasks contained essential quantity of adsorbent and MET solution (50 ml) with different starting or initial concentrations. On adsorption of contaminants, consequence of altered reaction parameters has given below:

*2.4.1 Effect of  initial pollutant concentration:*

In adsorption process initial pollutant concentration played a noteworthy role. On the sorption process, effect of initial pollutant concentration was investigated. Highest sorption was found at 0.9mg/L.

*2.4.2Effect of sorbent dosage:*

Sorbent dosage determines the aptitude of adsorbent for initial concentration of the adsorbate given. At optimized pollutant concentration the effect of activated carbon-1dose on adsorption of pollutant was studied. Maximum adsorption was found at 50 mg for activated carbon -1. After 100 mg/L no noteworthy exclusion was observed. The reason responsible for this was accumulation of particles of adsorbent, because of no noteworthy increase in useful surface area [10]. So 50mg/L was selected as best possible adsorbent dose for advance study.

*2.4.3 Effect of contact time*

Under optimized conditions, with a pollutant concentration of 0.9 mg/L and a biosorbent dosage of 50 mg/L, the effect of contact time on sorption was evaluated. The maximum adsorption efficiency was achieved after 60 minutes of contact time.

*2.4.4 Effect of pH*

The pH of the pollutant solution is a critical factor influencing the overall adsorption of the pollutant onto Activated Carbon-1. The effect of pH on the adsorption process was investigated, and the results indicated that Activated Carbon-1 exhibited maximum adsorption efficiency at pH 2.

*2.4.5 Effect of temperature*

For overall adsorption of pollutant on to activated carbon -1 the temperature of pollutant solution is an important factor. Temperature has a prominent effect on sorption of pollutant since equilibrium capacity of adsorbent changes with temperature. Ongoing adsorption process is endothermic or exothermic was determined by the results of factor temperature. Under optimized condition of pollutant concentration (0.9mg/L),sorbent dosage(50mg),contact time (60 min.) and pH of solution (2) the effect of temperature on adsorption process was studied. It was found that with increase of temperature the elimination of pollutant increased and maximum adsorption or elimination observed at 55$^0$C.

## 3. CONCLUSION

Water pollution remains a significant global challenge, affecting both developed and developing countries. Contaminants such as heavy metals, pharmaceutical chemicals, dyes, and pesticides have been a primary concern over the past decade. As the consumption of these pollutants continues to rise, they have become major contributors to water contamination. Despite the existence of regulatory policies aimed at controlling the use of these contaminants, they persist in water resources. Consequently, agro-waste materials have been explored as potential adsorbents for the removal of these pollutants from wastewater. In this study, low-cost adsorbents, particularly agro-waste materials, were utilized for the removal of contaminants. The removal efficiency of these adsorbents was evaluated by plotting various graphs, which demonstrated a high level of efficacy in eliminating these toxins from wastewater. Additionally, further research on the preparation of activated carbon from horticultural and household waste could provide valuable insights into the development of cost-effective solutions for wastewater treatment.

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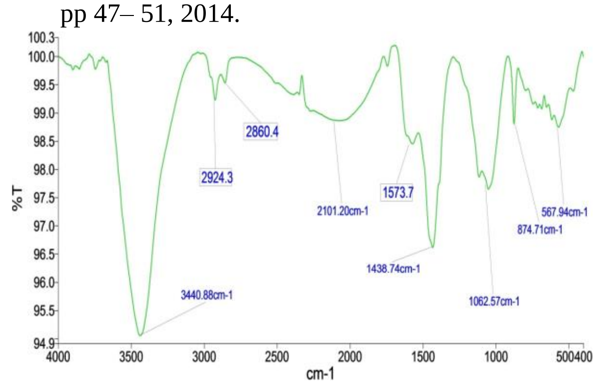

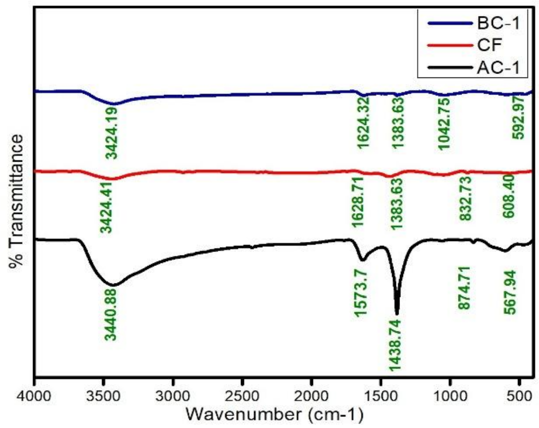
BC-1
CF
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% Transmittance
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4000
3500
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1383.63
1042.75
592.97
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1628.71
1383.63
832.73
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3440.88
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1438.74
874.71
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