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# Synthesis Characterization and application of Resins Obtained from Agricultural Waste to Remove Cu (II) from Waste Water

**Mohammad Azam** **Dr.Gulrez Nizami** **Nida Tanveer** **Mohammad Arshad** **Sheela**
Department of chemistry
Faculty of Science
Mohammad Ali Jauhar University
Rampur , UP, India
Mohammadazamazm@gmail.com

***Abstract-*** **The objective of the work is to a removal of the heavy metal ion of Cu(II) from the wastewater of different industries such as mining and, smelting, plating, brass manufacture, petroleum refining, electroplating industries and Cu- based agrichemicals. In this work, some waste agriculture materials are used as the adsorbent for the elimination of copper particle from the wastewater of Copper plants. Some agricultural materials like rice straw, rice bran, rice husk, rice hyacinth roots, coconut shell and, neem leaves were crushed in roll crusher and then grinded to prepare powdered material and then dried in an oven, then it was used as a copper adsorbent in different pH solutions of copper wastewater. Their resulted solutions were measured by FTIR and some other techniques.**

***Keyword s-*** **Adsorbent, Copper ion, rice straw, rice bran, rice husk, rice hyacinth roots, coconut shell, neem leaves etc.**

## I. INTRODUCTION

The toxic heavy metals ions are present in an environment it is a big problem with the whole world. The aqueous solution isolated of Copper (II) metal ions [B.S.S.K. Das. *et al.*, 2013]. Copper does not smash behind in the atmosphere. The fertilization industry, municipal and stormwater are the possible sources of copper manner with both plantings. [I.A. Sengil. *et al.*, 2009]. A large amount of copper access into the body several diseases was developed such as irritation, corrosion, widespread capillary damage, liver infection, kidney damage, renal damage, central nervous system irritation, and depression [Anees Ahmad[a*] et al., 2009]. Drinking water contains the maximum agreeable concentration of Cu (II) ions is 1.3 mg/L [ATSDR. et al., 2004].

The minimum limit of copper release from industrial wastewater into water is finite to 0.25mg/L [B. Zhu *et al.*, 2008]. The Cu (II) metal ions are efficiently removed by several adsorbents generally; some show a various harmful effect for the low metal concentration such as poor adsorption capacity, low efficiency/cost ration, and ineffectiveness.

Cu (II) metal ions are effectively removed from wastewater by use of natural agriculture yields such as coconut shell, neem leaves, hyacinth roots, rice straw, rice bran, and rice husk's [S. Larous A.H. *et al.*, 2005]. In this investigation, both experimentations were approved that the Cu (II) metal ion removed by each yield of aqueous solution on the basis of adsorption quality of the adapted waste product. The doses of eliminated Cu (II) ion were calculated such as adsorptions isotherms, thermodynamic, parameter, kinetics, an effect of PH solution, temperature, contacts time and adsorbent [H. Agarwal *et al.*, 2011]. The sorption method was chemical in nature it is proofed by the average sorption energy was considered the use of Dubinin Radushkevich isotherm model.

Some energetic functional groups were accountable for Cu (II) ion adsorption method; these functional groups were recognized by FTIR process. [B. singha. *et al.*, 2013]. It reduces the residues whose disposal becomes a major, costly problem. It converts the wastes into useful and inexpensive sorbents for water purification [Anees *et al.*, 2009].

## II.MATERIA AND METHOD

1. **Collected of Six natural agricultural waste material-** All the waste material such as rice straw, rice bran, rice husk, rice hyacinth roots, coconut shell, and neem leaves were conjunct from the local area of Moradabad (U.P).
2. **Adsorbent manufacture method-**Cu (II) metal ions obtained by the drain of adsorbent it was used ago for adsorption studies. The powder forms of adsorbents were saved in an airtight container.

Fig.1 Coconut Shell.

Fig.2 Neem Leaves.

Fig,3 Rice Straw.

Fig.4 Rice Straw.

Fig. 5 Rice Roots.

Fig.6 Rice Husk.

**3. Synthesis Of Cu (Ii) Solution**

For the synthesis of Cu (II) solution, to take 3.292 gm $CuSO_4.5H_2O$ was dissolved in distilled water to make up the 100ml volume of solution. The required standard solution was achieved by Cu (II) solution because this solution is finally diluted. Cu (II) ions were founded in the industrial wastewater it was achieved from a copper plant unit.

**4. Experimentation of adsorption and analytical Method**

The adsorption investigational was convened by a batch process. The adsorption process was affected by the experimental parameter such as initial pH, Initial Cu (II) ions concentration, adsorbent dosage and temperature than contact time was investigated for exclusive time duration.

## III. RESULT AND DISCUSSION

**1. Analysis by Atomic Absorption Analysis using cathode lamp**

Before analysis, all glass was washed with a di-acid mixture and later placed in potassium dichromate for overnight and were washed and were air or oven dried.

**2. Digestion of samples**

250 ml of waste/groundwater/effluent samples with 5 ml of concentrated HNO3 were heated and finally digested with 5 ml of HClO4 till samples became transparent. If a volume of samples reduces below 5 ml then 250ml samples water was taken again and poured and added 5 ml of concentrated HNO3 and 5 ml of HClO4 Digested samples were filtered (using filtering paper: Whatman no. 42) solution was diluted to 50 ml with bi-distilled water. Suitable blank was used to check for possible contamination during extraction.

**3.Calibration of the machine and methodology using CRM**

The sample preparation and analysis included the usual quality assurance protocols, including repeated measurements of a sample during the analysis, runs reagent blanks and use of in-house or procured certified reference plant material **(**Kolthoff, et al., 1971). Same was procured from Certified Reference Material from National Institute of Standard Technology-Denmark (Reference Sample-SRM 1575 a Pie Needle).

This certified material was used to validate for the results of analysis and for interlaboratory comparison as well as to certify the lab methodology. Certified reference material is intended for use in the evaluation of analytical methods for determination of element.

4. **Analysis of the samples**

Metals in the acid mixture were analyzed using atomic absorption spectroscopy (AAS, Electronic Corporation of India Ltd). Compressed air is sent to nebulizer along with digested filleted samples at contestant flow rate at a specific pressure to the acetylene flame to burn the digested samples. During this a specific Cathode, lamps are used for each specific metal. Emitted wavelength light is allowed to pass through burn samples and changes in emission light and its energy is measured by photovoltaic sensors.

The sample preparation and analysis included the usual quality assurance protocols, including repeated measurements of a sample during the analysis runs, reagent blanks and an in-house certified reference plant material (Kolth off et al., 1971).The analysis was highly reliable with variability between replicates and between measurements of the same sample generally being <5%. The percentage recoveries of the analyzed elements were generally consistent with the test used.

Different metals zinc, copper, cadmium, lead, chromium, nickel, and Hg were analyzed by using respective specific hollow cathode lamps (imported) purchased from the ECIL, Hyderabad. The specific software calculates the values for each metal and stores in the system using its own software for each metal and same is as displayed in the monitor and stored in a hard disk of the attached system which controls the atomic absorption analysis/. A System was washed with acid water (2%) after the analysis is over.

## 5. Analysis of Data

### 5.1. Statistical analysis

Samples were collected in triplicate to conduct the statistical analysis. The value represented as a mean + standard error (Snedecor and Cochran, 1967). Values were represented by the mean± standard error (Snedecor and Cochran, 1967). ANOVA revealed a significant difference in the metal concentration at different seasons ($p<0.01$, $p<0.005$) utilizing Duncan's multiple range test (Karmer, 1956).

### 5.2. Effects of pH

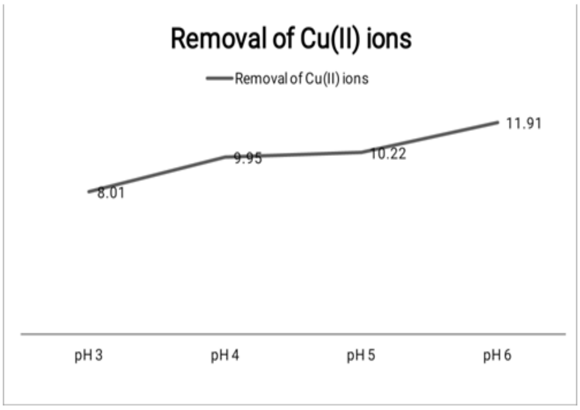


Fig.1 Variation of pH Value.

## 6. Ftir Study

The biosorption capacity of heavy metal onto different natural/ agricultural waste depends upon the presence of several active functional groups at the surface. The Fourier Transform Infrared spectra (FTIR) study of fresh and metal loaded bio-sorbents at optimized batch condition were carried out to identify the functional groups responsible for the adsorption process. Table: 3 indicates the wave number for the different functional groups present in the fresh adsorbents and Cu(II) loaded adsorbents.

Surface –OH group is one of the active functional group responsible for Cu(II) ions resins as the wave number shifts initial pH from 3528.50 cm-1 to 2505.48 cm-1, 2616.28 cm-1,2910.15 cm-1, 3369.05 cm-1, and waste resin as the wave number shift pH 10 from 3095.05 cm-1 to 2915.16 cm-1. Aliphatic C-H stretching may be responsible for Cu(II) adsorption onto resins as the wave number shifts initial pH from 3115.19 cm-1 to 2405.16 cm-1, 2518.19 cm-1, 2717.10 cm-1 and 2924.52 cm-1, and waste resin as the wave number shift pH-10 from 2900.16 cm-1 to 2719.05 cm-1 respectively.

The table also indicated the responsibility of aliphatic acid C=O stretching for resin adsorption process by shifting the wave number initial pH from 2012.15 cm-1 to 1717.73 cm-1, 1704.58 cm-1, 1715.83 cm-1 and 1730.30 cm-1, and waste resin as the wave number shift pH-10 from 1713.44 cm-1 to 1682.01 cm-1 respectively. This assigned the reactivity of unsaturated group like alkenes for the adsorption process.

Table 1 Wave number (cm-1) for the dominant peak from FTIR for Cu (II) adsorption.

| Functional groups | Cu(II) ions loaded | | | | | Waste copper solution with resin |
|---|---|---|---|---|---|---|
| | Fresh resin | PH-3 | pH-4 | pH-5 | pH-6 | pH-10 |
| Surface –OH stretching | 3528.5cm-1 | 2505.48cm-1 | 2616.28cm-1 | 2910.15cm-1 | 3369.05cm-1 | 3095.05cm-1 to 2915.16cm-1 |
| Aliphatic C-H stretching | 3115.19cm-1 | 2405.16cm-1 | 2518.19cm-1 | 2717.10cm-1 | 2924.52cm-1 | 2900.16cm-1 to 2719.05cm-1 |
| Aldehyde C-H stretching | 3016.25cm-1 | 2617.18cm-1 | 2854.13cm-1 | 2858.34cm-1 | 2854.76cm-1 | 2719.92cm-1 to 2696.16cm-1 |
| Phosphate ester group | 2516.19cm-1 | 2216.96cm-1 | 2396.26cm-1 | 2316.76cm-1 | 2359.76cm-1 | 2225.16cm-1 to 2196.16cm-1 |
| Aliphatic acid C=O stretching | 2012.15cm-1 | 1717.73cm-1 | 1704.58cm-1 | 1715.83cm-1 | 1730.;3cm-1 | 1713.44cm-1 to 1682.02cm-1 |

| | | | | | | |
|---|---|---|---|---|---|---|
| Unsaturated group like alkene | 1829.92cm-1 | 1644.02cm-1 | 1645.47cm-1 | 1644.09cm-1 | 1634.25cm-1 | 1534.15cm-1 to 1532.01cm-1 |
| Aromatic C-NO2 stretching | 1612.10cm-1 | 1540.42cm-1 | 1515.35cm-1 | 1520.57cm-1 | 1507.14cm-1 | 1556.75cm-1 to 1539.69cm-1 |
| Alkane group | 1592.15cm-1 | 1472.30cm-1 | 1435.25 m-1 | 1425.10cm-1 | 1410.01cm-1 | 1225.10cm-1 to 1215.12cm-1 |
| Carboxilate anion C=O stretching | 1350.2 cm-1 | 1316.00cm-1 | 1321.00cm-1 | 1300.10cm-1 | 1300.00cm-1 | 1300.00cm-1 to 1296.15cm-1 |
| -SO3 stretching | 1295.10cm-1 | 1225.10 cm-1 | 1250.00cm-1 | 1245.01 cm-1 | 1240.25cm-1 | 1235.16cm-1 to 1225.10cm-1 |
| Si-O stretching | 1110.33cm-1 | 1010.33cm-1 | 1032.25cm-1 | 1033.14cm-1 | 1072.66cm-1 | 1076.94cm-1 to 1098.26cm-1 |
| Sulphonic acid S=O stretching | 1196.3cm-1 | 1031.04cm-1 | 1031.37cm-1 | 1025.06cm-1 | 996.72cm-1 | 1012.72cm-1 to 1000.00cm-1 |

Aromatic NO2 group also responsible resins which is inferred from the shift of the peak more than 10 $cm^{-1}$ FTIR spectrum of resins also showed the intense bands at 1412.30 cm−1 which shifted to 1435.25 cm−1 that indicated the responsibility of alkanes group for the adsorption process. Table 3 also indicated the Si O stretching onto resins and which is active for the adsorption process.

Wave number of initial pH from 1010.33 cm−1 shifted to 996.72 cm−1. Which assigned for sulphonic acid S O stretching for Cu(II) adsorption. Ionic radius of Cu (II) is 0.73 ˚A which is smaller ion, therefore it can enter into smaller pore and surface of the binding site of the natural adsorbents and bind to several group present in the adsorbents simultaneously.

**6.Designing of single-stage batch adsorption system from equilibrium data**

The single-stage batch adsorption system can design by using the adsorption isotherm data. The initial Cu(II) ion concentration was reduced from C0 to Ct of these solution volume V at time t. Cu(II) ion loading into W mass of adsorbents was changed from q0 to qt. At time t = 0, q0 = 0, hence as time proceeds the mass balance equates the removal of Cu (II) ion from the aqueous solution by the adsorbents used. So the mass balance equation for batch adsorption system can be given as,

$$V(C_0 - C_t) = W(q_t - q_0) = Wq_t \qquad (3)$$

When adsorption process was in equilibrium then $C_t = C_e$ and $q_t = q_e$. Freundlich isotherm model was the best fit isotherm model for all the adsorbent used for this study. So this model was used in the batch adsorption design system. So one can write the above equation as Fig . indicates the plot of amount of resin required to removal the Cu(II) ions from aqueous solution at the initial concentration of 3 mg/L for 60%, 70%, 80% and 90% removal at different.

Table 2 Cu(II) ions from aqueous solution.

| S.No. | Cu(II) solution volume | pH | Mass of adsorption required for 80% Cu(II) ions removal | Waste copper solution 80% Cu(II) ions removal |
|---|---|---|---|---|
| 1. | 1000ml | | 21.3 | |
| 2. | 100ml | 3 | 8.01 | |
| 3. | 100ml | 4 | 9.95 | |
| 4. | 100ml | 5 | 10.22 | |
| 5. | 100ml | 6 | 11.9 | |
| 6. | 100ml | | | 16.17 |
| 7. | 100ml | 10 | | 15.59 |

## IV.CONCLUSION

In this work, agricultural waste were used as the adsorbent to remove the Cu (II) ions from the waste water of copper plant unit of Moradabad (Utter Pradesh) India. This way of using agricultural waste material, removed the Cu (II) ion from the waste water and to balance to concentration of Cu(II) ions in the water. Natural adsorbents are suitable for Cu (II) ion removal from aqueous solutions as well as from industrial waste water.

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