

A New Approach for Extraction of Heavy Metal Ion from Industrial Effluents by Silver Nanoparticles Coated With N-(2-Hydroxybenzylidene)-2-Amino Pyridine Schiff Base

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ABSTRACT: Extraction of heavy metal ions from industrial effluents is a matter of great importance because of their toxic effects on human health and on the natural environment in general. The Schiff base named N-(2-hydroxybenzylidene) - 2- amino pyridine [HBAP] formed from condensation of equimolar (1:1) quantity of salicylaldehyde and 2- amino pyridine in alcoholic medium. The prepared Schiff base was characterized by elemental analysis, FTIR, UV-VIS, NMR spectral studies. Synthesis of silver nanoparticles followed by complexation of silver nanoparticles with above mentioned Schiff base. The structure of silver nanocomplex of Schiff base is determined by UV-VIS spectra. Analysis of metal ion done by AAS as well as by spectrophotometrically. Sugarcane bagasse was used as solid phase for the extraction of heavy metal from industrial effluents. The above mentioned silver nanocomplex of Schiff base N-(2-hydroxybenzylidene) -2-amino pyridine is adsorbed on the sugarcane bagasse which is used as solid phase in the extraction process of heavy metal ion from industrial effluent. The solid phase is treated with acid as well as with base to remove all impurities and to activate its surface before extraction. The effects of several parameters that affect the adsorption of metal ion including initial metal ion concentration, contact time, Schiff base weight and pH were examined.

KEYWORDS: Heavy metal, extraction, nanoparticles, parameters.

I. INTRODUCTION

A group of metal or metal like element which having density greater than 5g/cm^3 and atomic number above 20 are termed as heavy metal. Heavy metal causes toxic effect on human health and on natural environment, so extraction of heavy metal from industrial effluent is matter of great importance. Heavy metal releases from various industries such as electroplating, mining and leather industries, pigments, metallurgical process, and textile industries. Industrial effluents were contained many metal ions such as cadmium, chromium, copper, lead, zinc, manganese and iron. Extraction of heavy metal done by many methods such as chemical precipitation, flocculation, filtration, chemical coagulation, reverse osmosis, ion exchange, solvent extraction, adsorption and membrane technologies[1-2]. But these methods are limited by high cost or may be insufficient in removal of heavy metal ion at trace level. Schiff base known as azomethine derivative and it is an important class of ligand that coordinated with metal ion via nitrogen of azomethine. Schiff base was versatile ligand and shows antibacterial, antifungal, anticancer and diuretic activities [3]. For showing these properties Schiff base is widely used in food industry, dye industry, analytical chemistry, catalysis, fungicidal, agrochemical and biological activities [4]. Nanotechnology is the study of materials smaller than 100 nanometres (nm). Silver nanoparticles have been used since ancient times for jewellery, utensils, monetary currency, dental alloy, photography, explosives, etc. Until the introduction of antibiotics, it was also used for its antiseptic activity, specifically in the management of open wounds and burns. Silver has also been used in filters to purify drinking water and clean swimming pool water due to its antimicrobial properties. In the present work, The Schiff base named N-(2

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hydroxylbenzylidene) - 2- amino pyridine [HBAP] formed from condensation of equimolar (1:1) quantity of salicylaldehyde and 2- amino pyridine in alcoholic medium. The prepared Schiff base was characterized by elemental analysis, FTIR, UV-VIS, NMR spectral studies. Silver Nanoparticles were synthesized by chemical reduction method. Silver nitrate used as a metal precursor and sodium borohydride as a reducing agent. Synthesis of silver nanoparticles followed by complexation of silver nanoparticles with above mentioned Schiff base. The structure of silver nanocomplex of Schiff base is determined by UV-VIS spectra. Analysis of Cu (II) and Fe (II) metal ion done by AAS as well as spectrophotometrically. Sugarcane bagasse was used as solid phase for the extraction of heavy metal Cu (II) and Fe (II) from industrial effluents. Adsorption is physico-chemical treatment processes and cost effective method. Sugarcane bagasse is a cheap adsorbent and abundant in nature. It is a by product of waste material from waste industry.

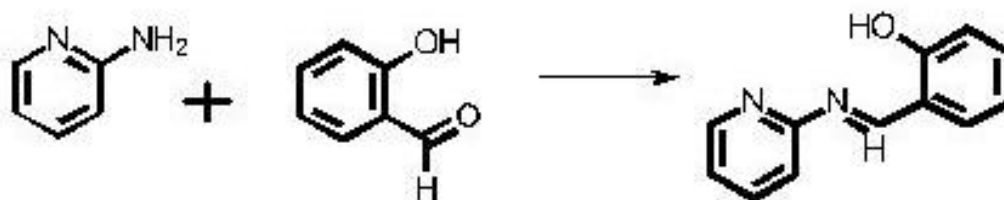
II. LITERATURE SURVEY

According to survey of literature from last few years special attention has been given to the environmental contamination due to heavy metals. Heavy metals are highly toxic and non biodegradable, so removal of heavy metal ion from industrial effluents is a matter of great importance. Many techniques used for extraction of heavy metal such as precipitation, ion exchange, adsorption, dialysis, osmosis. Adsorption technique has received much attention because many low cost and environment friendly material used as adsorbent[5-8]. Schiff bases are very important class of ligand which were widely used in medicinal and pharmaceutical fields because of their wide spectrum of biological activities [9-11]. Nanoparticles have been studied extensively due to their high specific area and unique physicochemical properties [12-15]. Silver nanoparticles have been used from ancient times for purification of water and disinfection of clothes. A silver nanoparticle exhibits a broad spectrum of antimicrobial, antifungal and also an antiviral activity [16-18]. Chemical reduction method is used for fast synthesis of metal nanoparticles. So, using nanoparticles, nanofiltration or other product resulting from progression of nanotechnology could be resolved water quality issues. Magnetite nanoparticles functionalized with carboxyl amine and thiol groups were used for removal of toxic metal ions and bacterial pathogens from water [19].

III. METHODOLOGY

SYNTHESIS OF SCHIFF BASE: A solution of salicylaldehyde in ethanol mixed with solution of 2-amino pyridine and adds few drops of formic acid. Mixture is stirred and thereafter refluxed for 7-8 hrs, the precipitate collected by filtration and recrystallized from alcohol.

Yellow-orange crystal; yield 57%; mp 62-64°C. IR (cm⁻¹): 3434, 1614, 1588, 1277, 1257, 1149, 992, 914, 847, 789, 738, 577. ¹HNMR (CDCl₃, 400 MHz): 6.93-8.48(m, 8H), 9.41(s, 1H), 13.40(s, 1H). Anal.calcd for C₁₂H₁₀N₂O: C, 72.75, H, 5.02, N, 14.14. Found: C, 72.30, H, 5.05, N, 14.1.



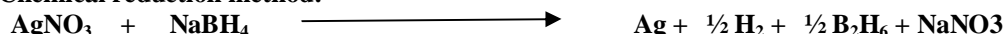
2-amino pyridine

salicylaldehyde

N-(2-hydroxybenzylidene) pyridin-2-amine

SYNTHESIS OF SILVER NANOPARTICLE: Fixed volume of silver nitrate solution added drop wise to ice chilled sodium borohydride solution. The reaction mixture was stirred vigorously on magnetic stirrer. The entire addition took about three to four minutes, after which the stirring was stopped and the stir bar removed. The colloidal solution of nanoparticles was formed.

Chemical reduction method:



SYNTHESIS OF SILVERNANOCOMPLEX OF SCHIFF BASE: Synthesis of the silver nanocomplex was done by anchoring the ligand as prepared above on the synthesized silver nanoparticles [20]. Schiff base was successively added to the nano silver colloidal solution in and stirred for 10-15 minutes.

ACTIVATION OF SOLID PHASE: sugarcane bagasse is used as solid phase in adsorption process. SCB was first washed with distilled water to remove the dust particles, then soaked overnight in NaOH solution and again washed well with DDW. SCB was then soaked in CH₃COOH for 3 hrs to remove the traces of NaOH [21]. It was washed again with DDW till the wash; water became colourless and then filtered, well dried, powdered and sieved before use.

IV. RESULTS AND DISCUSSION

CHARACTERIZATION OF SCHIFF BASE:

UV-VIS Spectral studies: Four bands were showed in ethanol medium. Band A and B at 207 nm and at 268nm wavelength respectively due to transition in aromatic ring [22]. Band C appeared at 305nm wavelength due to transition in between Π orbital localized on central azomethine (-CH=N-) bond [23]. Band D appeared at 347nm wavelength due to charge transfer within the entire Schiff base molecule this bond is commonly observed in o-hydroxy Schiff base [24] and is based on strong intramolecular H-bonding between the hydroxyl group of the salicylidine and the azomethine nitrogen [25].

FTIR Studies: The ν (C=N) absorption band were observed at 1614 cm^{-1} . The stretching frequency observed at 3434 cm^{-1} shows the presence of OH-----N intramolecular H-bond.

¹H NMR Studies: ¹H NMR data shows the tautomeric equilibrium favours the phenol amine in CDCl₃. $\delta=13.40$ ppm, singlet for OH, $\delta=9.41$ ppm, singlet for (-CH=N-).

CHARACTERIZATION OF SILVER NANOPARTICLE:

UV-VIS Spectral studies: The specific colour of colloidal silver nanoparticle solution is due to Plasmon absorbance and produces peak at 405 nm (λ_{max}) wavelength. Incident light produces oscillations in conductive electrons on the surface of nanoparticles and electromagnetic radiation is absorbed so peak is observed.

SEM Studies: The scanning electron microscope (SEM) image shown in figure no.1. The synthesized silver nanoparticle shows the particle size is 50nm which confirms nanostructure.

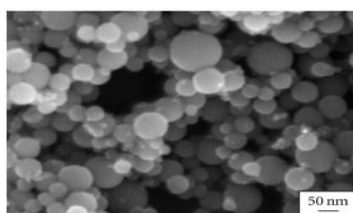


Figure no.1- SEM image of silver nanoparticles

Comparison of UV-VIS Spectra of AgNps and Schiff base complex of AgNps: Comparative Study shown from the figure no.2.

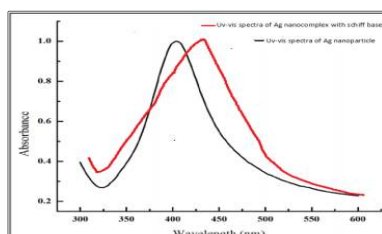


Figure no.2-UV-VIS of AgNps and Ag nanocomplex of Schiff base

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The UV-Vis spectra of silver nanoparticle (AgNps) shows λ_{max} at 405 nm and Schiff base complex of AgNps shows λ_{max} at 450 nm. A spectrum shows shifting at higher wavelength or red shift.

EXTRACTION OF Cu (II) AND Fe (II):

Silver nanocomplex of Schiff base is attached to be on solid phase. Metal ion extracted from industrial water samples by continuous adsorption method. Column method used for determining the effect of various parameters on % removal of Cu (II) and Fe (II) metal ion from industrial effluents.

A glass column of 30 cm length and 2 cm internal diameter was used. Sugarcane bagasse was put between two layers of glass wool, the first at the bottom to avoid loss of sorbent when the sample solution passes through the column and the second at the top to retain it. Then, water samples containing silver nanocomplex of Schiff base passes through the column with flow rate 1.0 ml/ min.

Heavy metal Cu (II) and Fe (II) detected by flame AAS (Atomic absorption spectroscopy) as well as spectrophotometrically.

The percentage of metal ion removal was calculated from following equation:

$$\% \text{ Removal} = (C_0 - C_e) / C_0 \times 100,$$

Where C_0 is the initial concentration of metal ion and C_e is the metal ion concentration at equilibrium.

Effect of initial concentration of metal ion: Effect of initial metal ion concentration on % removal of Cu (II) and Fe (II) metal ion by silver nanocomplex of Schiff base from industrial effluent was investigated.

Table -1 Effect of initial concentration of metal ion on % removal of heavy metal:

Initial concentration of Cu(II) and Fe(II) ppm in sample 1	% Removal of Cu(II)	% Removal of Fe(II)
30	72%	74%
25	88%	89%
20	83%	84%
15	75%	76%
10	68%	69%
5	66%	64%

Table no. 1 shows the effect of initial metal ion concentration on % removal of heavy metal in water sample-1 Effect of metal ion concentration is calculated at constant pH, biomass and weight of silver nanocomplex of Schiff base. Maximum % removal of heavy metal takes places at concentration 25 ppm of Cu (II) and Fe (II) in water sample-1.

Table-2 Effect of initial concentration of metal ion on % removal of heavy metal:

Initial concentration of Cu(II) and Fe(II) ppm in sample 2	% Removal of Cu(II)	% Removal of Fe(II)
32	70%	71%
27	88%	89%
22	84%	86%
17	77%	79%
12	70%	76%
7	68%	70%

Table no. 2 shows the effect of initial metal ion concentration on % removal of heavy metal in water sample-2 at constant pH, biomass sugar cane (bagasse) and weight of silver nanocomplex of Schiff base. Maximum % removal of heavy metal takes places at concentration 27 ppm of Cu (II) and Fe (II) in water sample-2.

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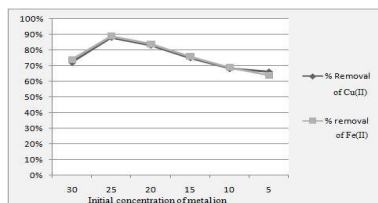


Figure no. 3

As concentration of metal ion increases in water sample-1 the % removal of Cu (II) and Fe(II) increases due to increasing of binding site and at specific concentration of metal ion all the binding sites becomes saturated but after reaching at saturation point as the concentration increases % removal decreases as in figure no.3.

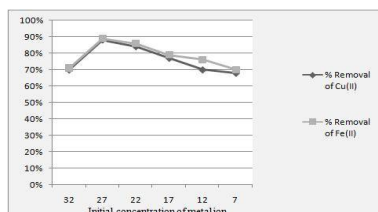


Figure no.4

Same as in figure no.4 as the concentration of metal ion increases % removal of heavy metal ion increases but after saturation of all the binding sites % removal of heavy metal ion decreases in water sample 2. Maximum % removal of heavy metal takes places at concentration 27 ppm of Cu (II) and Fe (II) in water sample-2.

Effect of pH: The pH was varied from 2 to 12 for Cu (II) and Fe (II) and the pH of the solutions was adjusted to different values using suitable buffers.

Table-3 Effect of pH on % removal of heavy metal:

pH	% Removal of Cu (II) in water sample 1	% Removal of Fe (II) in water sample 1	% Removal of Cu (II) in water sample 2	% Removal of Fe (II) in water sample 2
2	44%	48%	47%	48%
4	80%	83%	72%	85%
6	83%	88%	77%	88%
8	86%	88%	89%	89%
10	87%	72%	89%	74%
12	70%	65%	73%	66%

The removal efficiency was calculated at constant Schiff base weight, biomass and concentration of metal ion. The table -3 shows that maximum absorption takes place at pH 10,10,6, 8 for Cu (II) and Fe (II) respectively in water sample 1 and 2.

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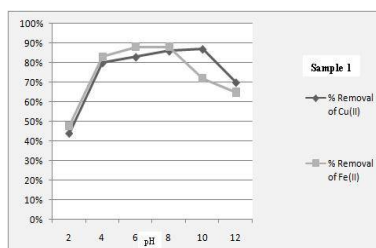


Figure no.5

A figure no-5 shows the uptake of Cu (II) and Fe (II) which is depends greatly on pH. At low pH mostly functional group were ionized and presented in form of positively charged protons so, electrostatic repulsion in between metal cations and the protonated groups of the silver nanocomplex of Schiff base. The metal ions are nearly stable at pH 10 and 6-8 in Cu (II) and Fe (II) respectively due to stability of complexation of metal ion with silver coated Schiff base.

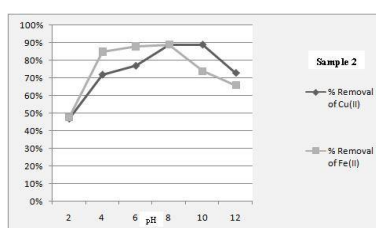


Figure no.6

A figure no-6 shows the uptake of Cu (II) and Fe (II) in water sample 2 which is greatly depends on pH. The removal efficiency was calculated at constant Schiff base weight, biomass and concentration of metal ion. Maximum absorption takes place at 10 and 8 in Cu (II) and Fe (II) respectively.

Effect of Schiff base weight: Different amount of Schiff base complex was added in industrial water samples at constant pH, constant concentration of metal ion and at constant biomass.

Table-4 Effect of weight of Schiff base on % removal of heavy metal:

Weight of Schiff base in gm	% Removal of Cu (II) in water sample 1	% Removal of Fe (II) in water sample 1	% Removal of Cu (II) in water sample 2	% Removal of Fe (II) in water sample 2
0.1	68%	67%	70%	68%
0.25	79%	75%	78%	79%
0.5	90%	88%	91%	89%
0.75	90%	89%	91%	90%
1	90%	89%	91%	90%

According to table 4 maximum % removal of Cu (II) in both samples takes place at 0.5 gm weight of Schiff base due to saturation of absorption active site at 0.5 gm and maximum % removal of Fe (II) in both samples takes place at 0.75 gm weight of Schiff base.

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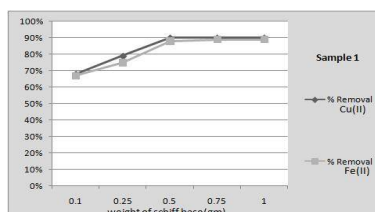


Figure .no 7

Figure no.7 shows as the amount of Schiff base is increases % removal increases and after reaching at 0.5 gm, the % removal is constant due to excess of sorbent quantity inhibiting the approach of adsorbed ions to the active site of the sorbent. Maximum % removal of Cu (II) and Fe (II) in sample 1 is at 0.5 gm and 0.75gm weight of silver nanocomplex of Schiff base respectively.

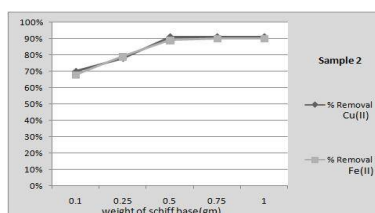


Figure no.8

Figure no.8 shows as the amount of Schiff base is increases % removal increases and after reaching at 0.5 gm, the % removal is constant due to excess of sorbent quantity inhibiting the approach of adsorbed ions to the active site of the sorbent. Maximum % removal of Cu (II) and Fe (II) in sample 2 is at 0.5 gm and 0.75gm weight of silver nanocomplex of Schiff base respectively.

Effect of contact time: The effect of contact time on % removal of Effect of Cu (II) and Fe (II) metal ion by silver nanocomplex of Schiff base from industrial effluent was investigated and result obtained in table no.5.

Table-5 Effect of contact time on % removal of heavy metal:

Contact time(minutes)	% Removal of Cu (II) in water sample 1	% Removal of Fe (II) in water sample 1	% Removal of Cu (II) in water sample 2	% Removal of Fe (II) in water sample 2
5	66%	68%	63%	69%
10	72%	74%	70%	74%
15	78%	76%	76%	78%
20	86%	84%	84%	85%
25	87%	88%	88%	89%
30	87%	88%	88%	89%

Effect of contact time is calculated at constant pH, biomass, and weight of silver nanocomplex of Schiff base and constant initial metal ion concentration. Table 5 shows maximum% removal of heavy metal at 25 minutes in both samples.

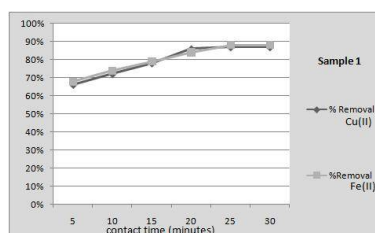


Figure no.9

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Figure no.9 shows the value of % removal of Cu (II) and Fe (II) in sample-1 increased steadily and after 25 minutes the adsorption quantity of metal ion showed nearly no change. This behaviour shows slow equilibrium.

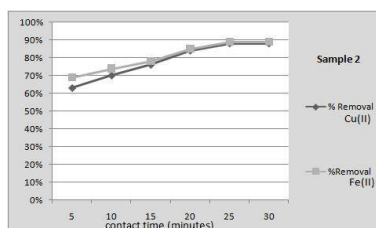


Figure no.10

Figure no.10 shows the value of % removal of Cu (II) and Fe (II) in sample-2 increased steadily and after 25 minutes the adsorption quantity of metal ion showed nearly no change. It describes the sorption sites affected with contact time.

V. CONCLUSIONS

1. The silver nanocomplex of Schiff base attached with solid phase have high surface area in low volume is effectively removed Cu (II) and Fe (II) metal ion.
2. Continuous adsorption method using silver nanocomplex of Schiff base attached with Solid phase sugarcane bagasse is cost effective method.
3. The graph of initial metal ion concentration Vs % removal concluded effective % removal of Cu (II) and Fe (II) 64% to up to 89% as concentration of heavy metal increases 5 ppm to 27 ppm by Silver nanocomplex of Schiff base.
4. The graph of pH Vs % removal of heavy metal ion concluded that maximum % removal of Cu (II) and Fe (II) takes place at pH 10 and 6-8 respectively.
5. The graph of weight of Schiff base Vs % removal of heavy metal ion concluded that maximum % removal of Cu (II) and Fe (II) takes place at 0.5 gm and 0.75 gm weight of Schiff base respectively.
6. The graph of contact time Vs % removal of heavy metal ion concluded that maximum % removal of Cu (II) and Fe (II) takes place at after 25 minutes due to time is much needed to reach equilibrium during adsorption process.
7. From all graphs and tables it is concluded that silver nanocomplex of Schiff base attached with sugarcane bagasse is new and cost effective approach for removal of heavy metal ion from industrial effluents.

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