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Synthesis, characterization and chelating studying of some heavy metals with poly (Furfural-catechol)

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Abstract

Resins derived from the condensation of furfural-phenols are highly efficient in recovering trace metal ions, a process vital for environmental green chemistry. This study addresses the need for novel coordination polymers that can be applied to environmental challenges, such as the removal of toxic heavy metals from water resources. The aim of this study is to synthesize and characterize a new furfural-catechol resin and evaluate its efficiency in the uptake of heavy metal ions, specifically Cd (II), Cu (II), Hg (II), Ni (II), and Pb (II), from water samples. New chelating polymers were synthesized via the condensation of furfural and catechol. The resulting resin and its metallic complexes were identified using UV-VIS and IR spectroscopy, magnetic moment measurements, electrical conductivity, and flame atomic absorption spectroscopy (F. A. A. S). Ion-exchange properties were evaluated using the batch equilibrium method by determining metal ion concentrations before and after adsorption. The study confirmed the synthesis and characterization of the chelating polymer and its complexes. The stability of the polymer chelates in solution was determined using the Ostwald dilution method. Optimum conditions for maximum separation were achieved in acidic media at pH 4. 0-6. 5, particularly for cadmium (II) and mercury (II) at 5-10 ppm uptake.

Conclusions: The synthesized furfural-catechol resin serves as an effective tool for the separation of heavy metals from aqueous solutions. These findings highlight the potential of these coordination polymers for high-efficiency water purification and environmental remediation.

Keywords: Furfural resins, chelation properties of resins, uptake properties of heavy metals, green chemistry, adsorbent resin

Introduction

The resins derived from condensation of furfural-phenols and their substituents have been interesting in the field of green chemistry due to high efficiency in recovering the trace metal ions [1-3]. The bis-phenol A-resin have wide applications in the chelation of transition metals as well as their spectroscopic characterization that have encouraged great researchers to afford novel types of coordination polymers for solar cells manufacturing [4-6]. Recently many researchers have recorded new routes for the synthesis and characterization of poly (5, 5-methylene-bis (8-hydroxy quinoline)-7, 7-dylethylene in which-CH₂- and -CH₂CH₂- groups are attached alternately to quinoline units in the polymer chain [7, 8]. The heavy metal ions uptake properties of polystyrene incorporated with active sites like isomethine and azo dyes were described in the recent years and attracted considerable attention from many researchers [9, 10]. However, in our current work it has included a study on the preparation of a new resin from condensation furfural-m-cresol with a new modification of the optimal methods and their metal complexes with copper (II), nickel (II), cadmium (II), mercury (II) and lead (II) ions. As well as the important and purposeful part of our work is the uptake these mentioned metal ions from water samples by chemical adsorption to state a high efficiency method for resolution the toxic metal ions like Cd (II) and Hg (II).

Materials and Methods

Poly (furfural-catechol) was prepared according to the paper published in literature survey [12]. The reactions and the purity of the prepared resin and complexes were checked by thin

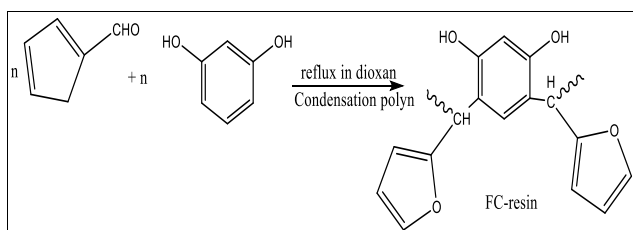
layer chromatography (T. L. C) on using chloroform and ethyl acetate as eluent with 2:3 volume ratios. The decomposition points, solubility and infrared spectroscopy table (1). All other chemicals used were of analytical grade, pbCl_2 , CdCl_2 , H_2O , HgCl_2 , $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ and $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ solutions were prepared and standardized by conventional methods as described by Vogel [13]. The PH of the metal ions solutions were maintained using sodium acetate-acetic acid buffer couple. The batch equilibration method developed [11] was adopted for increasing the chelating ion-exchanging properties of the m-cresol-furfural resin. This comprises (a) a study of the effect of pH on adsorption of metal ions by polymer sample (b) estimation of the time required to reach, the state of equilibrium under given experimental conditions and (c) measurement of the distribution ratio of metal ion between the polymer phase and the solution phase over a wide PH-range.

Table 1: Some physical properties of the adsorbent FC

Solubility	water (-), acetone (+), chloroform (+), methanol (-)
Viscosity	$6.018 \times 10^{-3} \text{ dL} \cdot \text{g}^{-1}$
Conversion	65%
Softening Point	90-115 °C
Color	Dark yellow

Preparation of furfural-catechol resin (FC)

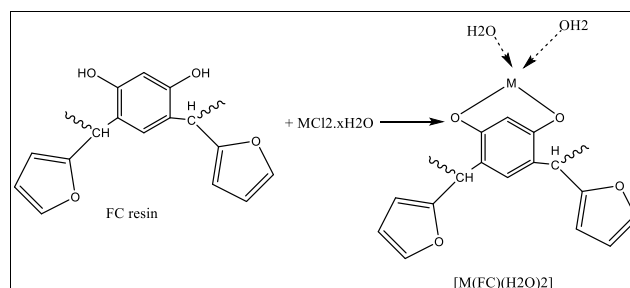
The starting materials furfural (0.02 mole, 1.92 g), catechol (0.011 mole, 1.77 g) were mixed in around bottle flask 100 ml containing 50 ml of dioxan solvent and 2M HCl that is slowly added to the reaction mixture and the content reaction was refluxed at 135 °C temperature for 12 hours on oil bath variable shaking in order to reach homogeneous reaction. The mixture was poured in ice cold water, filtered and then washed with hot water to remove unreacted starting materials. Then the product was washed with cold dioxan and methanol and dried in vacuum, Scheme 1. The dark yellow precipitate of resin was obtained to be about 80-88% then characterized by NMR, IR, UV-Visible spectrum and crostini viscosity, Table 1.



Scheme 1: Preparation of resin (FC)

Preparation of Polymeric Metal Chelates (FC)

A solution of (1.35 g, 0.01 mole), adsorbent copolymer in 25 mL of 2:1 volume ratio of acetone: chloroform mixture was gradually poured to a solution of (0.160 g, 0.011 mole) of $\text{CdCl}_2 \cdot \text{H}_2\text{O}$ or (0.231 g, 0.011 mole) of $\text{CoCl}_2 \cdot \text{H}_2\text{O}$, or (0.312 g, 0.021 mole) of HgCl_2 or (0.267 g, 0.011 mole) of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ and (0.710 g, 0.011 mole) of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ in 25 mL of distilled water at room temperature with good stirring. The reaction mixture was heated on a water bath for 50-65 minutes, precipitates were filtered off, then washed with ethanol and dry ethyl ether, Scheme (2).



Scheme 2: Preparation of metal complexes $[\text{M}(\text{FC})(\text{H}_2\text{O})_2]$

Measurements

Analysis for metal content in the adsorbed complexes were measured by flame atomic absorption technique via Shimadzo-AA-670. The electron ionization mass spectrum for the new adsorbent copolymer MC-F was measured by impact 10 eV MS spectrometer. The vibration spectra (IR) of all polymeric chelates were scanned in CSI on F. T. I. R-8300 Shimadzu spectrophotometer. The electronic spectra for the free adsorbent copolymer and its metal chelates were measured in dimethyl sulfoxide (DMSO) of 10^{-3} M concentration using Shimadzu UV-Vis. 160 A, Ultra-Violet visible spectrophotometer in the range 200-800 nm. The effective magnetic moments of copper(II) and nickel(II) chelates were made at room temperature by the Gouy' method and copper (II) chloride dehydrate was used for calibration. The effective magnetic moments for copper(II) and nickel(II) chelates were measured on Sherewood Magnetic balance at Mustansiriyah University, College of Science, Chemistry Department. The pH of the solutions was adjusted to about 5, 0-8.5 by addition of 3 mL of sodium acetate-acetic acid buffer couple [12]. The polymeric chelate which separated out as a suspension was digested for about 90 minutes on water bath and was then allowed to settle. The solid was filtered, washed with acetone, water and then with ethanol, and finally with petroleum ether (25 mL), Table 2.

Table 2: Some physical properties and some spectral data of adsorbent and its metal chelates

Adsorbent Resin	M% Calc. (found) ^(b)	I. R (v cm^{-1}) ν_{NH} , ν_{OH} , ν_{CO} , $\nu_{\text{M-L}}$	λ_{max} nm (ϵ_{max})	μ_{eff} (B. M)	Λ_{m} $\mu\text{S} \cdot \text{mol}^{-1}$	Suggested structure
FC		3500-3400(m), 1100, 1600	250 (20700) 315(1090)	-	-	-
$[\text{Cu}(\text{FC})(\text{H}_2\text{O})_2]$	7.44 (9.9)	3200(br.), 400-500(Cu-O)	560(190) 450(1400) 350(20110)	1.77	19	Distorted octahedral
$[\text{Cd}(\text{FC})(\text{H}_2\text{O})_2]$	12.31 (12.77)	3180, 400, 245(Cd-Cl)	215, 550, 700	0.00	30	Tetrahedral
$[\text{Ni}(\text{FC})(\text{H}_2\text{O})_2]$	8.801 (9.66)	3200(br.), 400-500, 390(w) (Ni-Cl)	270, 390, 415	2.82	19	Octahedral
$[\text{Hg}(\text{FC})]$	5.99 (8.0)	3200, 1190, 470, 3422 (w) (Hg-Cl)	290 (29070) 385(18900)	0	23	Tetrahedral
$[\text{Pb}(\text{FC})_2(\text{H}_2\text{O})_2]$	6.91 (5.97)	3200(br), 1130, 490(Pb-O), (Pb-Cl)	275(23000) 385(13900)	0.00	25	Tetrahedral

$b = M\%$ was calculated by flame atomic absorption technique via standard addition method.

$c = \Lambda_m$ conductivity of chelate solutions were recorded in DMSO solvent

Results and Discussion

Characterization of polymeric chelates

The MS spectrum of the new resin showed $m/e=374$ molecular ion for the repeated unit *m*-cresol-furfural chelating resin adopting the units of (n) was approximately equal to (2. 5-3. 25) and this new data might agree with the methodology of the most units of furfural-catechol, Figure 1.

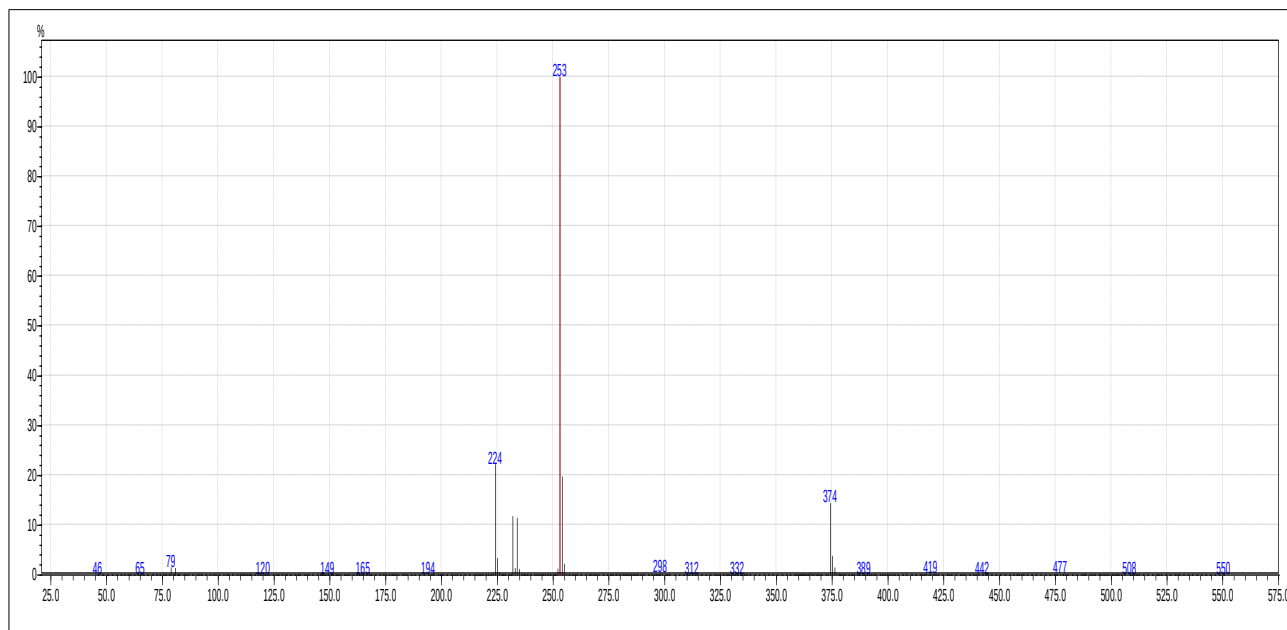


Fig 1: MS spectrum of repeated unit resin FC

All the polymeric chelates are insoluble in common organic solvents and are decomposed by mineral acids affording the original polymeric ligand. The results of metal analyses suggest that the metal: ligand ratio is 1:1 for all complexes, Table 1. As well as the new FC resin was identified on the basis of ¹H NMR spectrum, Figure 2. The ¹H NMR spectrum of the prepared FC- resin in DMSO-*d*₆ solvent showed de

shielded peaks at 12-13 ppm that is associated with the nuclear spin of acidic phenolic hydroxyl groups. Furthermore the spin-spin coupling at (6.95-7.6) ppm and (7.88-8.22) ppm are assigned to nuclear spin of aromatic protons of phenyl catechol moiety and furan ring respectively [14].

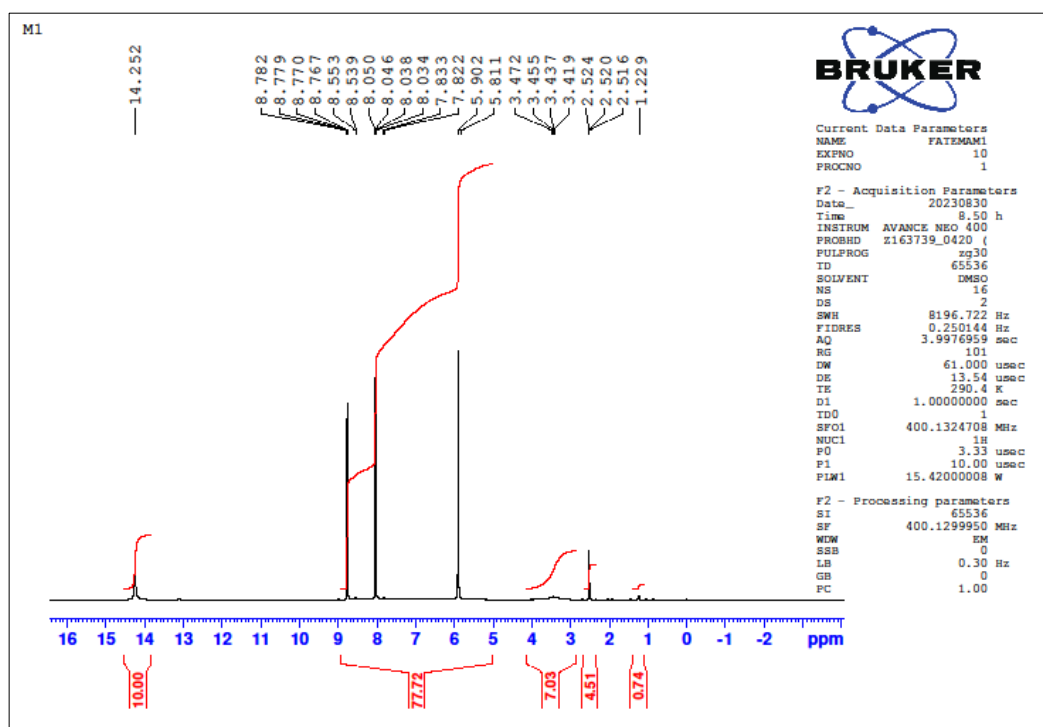
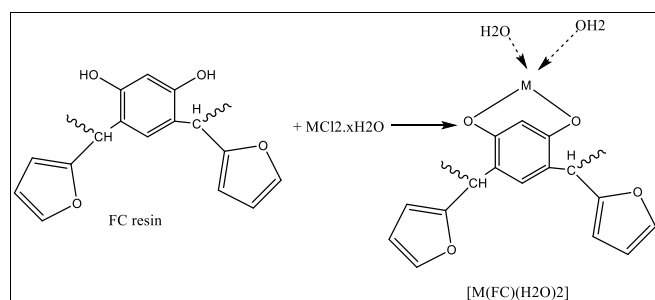


Fig 2: ¹H NMR spectrum of FC resin in *d*₆-DMSO

The IR spectrum of the adsorbent resin, FC and its metal complexes were measured in the range 400-4000 cm^{-1} to assign their functional groups and to give evidence for the active sites of coordination behavior. The comparison of the absorptions for the free adsorbent copolymer and its metal complexes exhibited remarkable changes in the C-O bonds, therefore indicates the bidentate behavior of this system to afford five-membered stable ring with all selected metalions [13-15]. The medium bands at around 3200-3430 cm^{-1} and (1250-1150) cm^{-1} reveals the stretching of (OH), (C-O) and O-H of hydrated water molecules respectively [18]. Furthermore the appearance of bands in the region 600-200 cm^{-1} in the metal polymeric chelates supports the Furthermore, the weak bands in the regions (350-490) cm^{-1} are consistent with the formation of (M-O) coordination bonds [18, 19].



Scheme 3 Coordination sites of FC with the metal ions

Electronic spectra of the adsorbent resin and its metal complexes

The adsorbent copolymer exhibited a high intensity band in the U. V-region is splitted into two components with maximum absorption at wave lengths 250 nm and 315 nm. This band may be attributed to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions of aromatic and furan system [15, 18]. The pale green solution of Cu-FC chelate in DMSO displayed two weak absorptions at 560 and 450 nm which are assigned to spin-allowed transitions $B_{1g}^2 \rightarrow B_{2g}^4$ and $B_{1g}^2 \rightarrow E_g$ respectively. The effective magnetic moments for the solid copper(II) complex recorded 1.78 BM value indicating the distorted structure of copper (II) complex [19, 20]. The dark brown solution of nickel(II) adsorbent complex showed weak bands at 410 and 390 nm assigning to ${}^3T_1 \rightarrow {}^3T_2$ and ${}^3T_1 \rightarrow {}^3A_2$ respectively. Furthermore the high-intense peak at around 270 nm is attributed to LMCT. The observed magnetic

moments of the solid complex at 2.90 BM values confirms the tetrahedral geometry [20, 21].

Effect of pH and time on uptake of metal ion

Based on the data provided in the table and the generated bar chart, Figure 3 here is a professional scientific. The removal of heavy metal ions (Hg, Pb, Ni, Cu, Cd) from aqueous solutions is highly dependent on the pH of the solution. This dependency is primarily due to the surface charge of the adsorbent and the speciation of the metal ions in the water. At pH 2, the removal efficiency for all metals is at its lowest (ranging from 10% to 40%). Mechanism: In highly acidic environments, there is a high concentration of hydrogen ions (H^+). These protons compete strongly with the positively charged metal ions (M^{2+}) for the available active sites on the adsorbent surface. Surface Charge: Additionally, the adsorbent surface becomes protonated (positively charged), leading to electrostatic repulsion between the surface and the metal cations, which hinders adsorption. As the pH increases to 4 and 6, the removal efficiency improves significantly for most metals. Mercury (Hg) and Nickel (Ni): These metals reach their maximum removal efficiency at pH 6 (80% and 70%, respectively [20, 21]. At this range, the competition with H^+ ions decreases, and the surface charge of the adsorbent becomes more neutral or slightly negative, favoring the binding of cations. Continued Increase (Pb, Cu, and Cd): The removal of Cadmium (Cd) reaches a peak of 95% at pH 9, while Lead (Pb) and Copper (Cu) also show their highest values. Precipitation: Beyond adsorption, the dominant mechanism at high pH is often chemical precipitation. The abundance of hydroxide ions (OH^-) leads to the formation of insoluble metal hydroxides, such as $Cd(OH)_2$ or $Pb(OH)_2$. Sharp Decline (Ni and Hg): Interestingly, the removal of Nickel(II) drops drastically from 70% (at pH 6) to only 15% (at pH 9). Soluble Complexes: This decline is likely due to the formation of soluble anionic hydroxy complexes (e. g., $[Ni(OH)_2]$ or $[Ni(OH)_4]^{2-}$ at high pH, which causes the metal to remain in the solution rather than being removed [22]. The selectivity patterns of the metal ions by the resin are as follows:

pH = 6: $Cu(III) > Ni(II) > Hg(II) > Pb(II) > Cd(II)$
 pH = 8: $Cd(II) > Cu(II) > Ni(II) > Ni(II)$
 pH = 10: $Pb(II) > Cd(II) > Ni(II) > Cu(II) > Cu(II)$

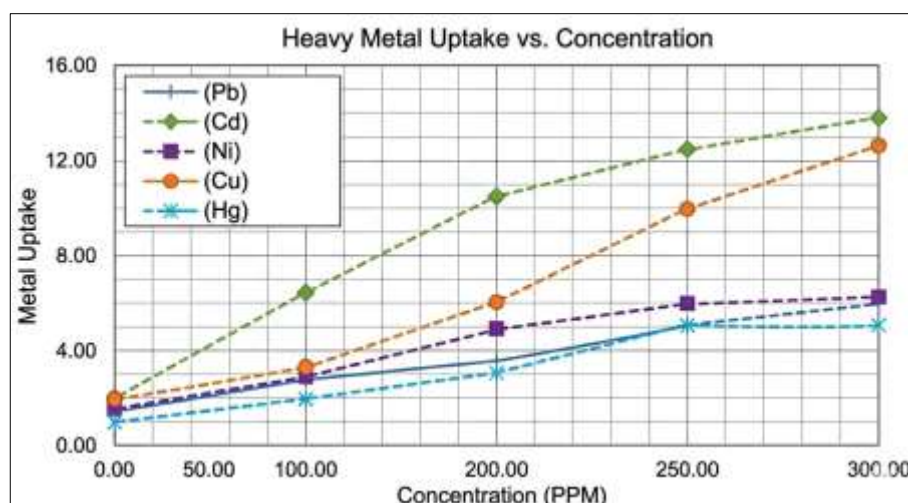


Fig 3: variation of uptake of metal with concentration of Metal Uptake Variation with Concentration

The experimental data demonstrates a positive correlation between the initial metal concentration and the uptake capacity of the adsorbent. As the concentration of metal ions (Pb, Cd, Ni, Cu, Hg) increases from 50 to 300 PPM, the uptake values (measured in mg/g or equivalent units) exhibit a consistent upward trend. This behavior is primarily attributed to the increased driving force provided by the concentration gradient, which overcomes the mass transfer resistance between the aqueous and solid phases. The results indicate a clear hierarchy in the adsorption capacity among the studied heavy metals:

Highest Affinity (Cadmium - Cd): Cd ions (represented by the diamond symbols) showed the highest uptake across the entire concentration range, reaching approximately 13.8 at 300 PPM. This suggests that the adsorbent surface has a higher density of functional groups with a specific affinity for Cd(II) ions [21-23].

Intermediate Performance (Copper - Cu and Nickel - Ni): Cu (II) and Ni (II) showed significant increases, with Cu notably surpassing other metals at higher concentrations (250-300 PPM), suggesting a multi-layer adsorption mechanism or higher saturation limit for copper. **Lowest**

Affinity of Hg(II) consistently remained at the lower end of the uptake spectrum, which may be due to its larger ionic radius or lower electronegativity compared to the other metals in this specific system. At lower concentrations (50-100 ppm), the slopes of the curves are relatively steep, indicating a high availability of vacant active sites on the adsorbent surface. However, as the concentration progresses toward 300 PPM, some curves (like Pb and Hg) begin to show a slight decrease in the rate of uptake (a flattening of the slope). This phenomenon signifies that the active sites are becoming increasingly saturated, leading to a plateau effect where further increases in concentration result in diminishing returns in uptake capacity [11, 22]. The variation in uptake between the metals can be explained by several factors, including: **Ionic Radius:** Smaller ions often penetrate the adsorbent pores more effectively. **Hydration Energy:** Metals with lower hydration energy are more easily dehydrated and adsorbed onto the surface. **pH of the Solution:** The speciation of these metals Pb(II), Cd(II) and the surface charge of the adsorbent are highly dependent on the competitive environment shown in the multi-component system, Figure 4.

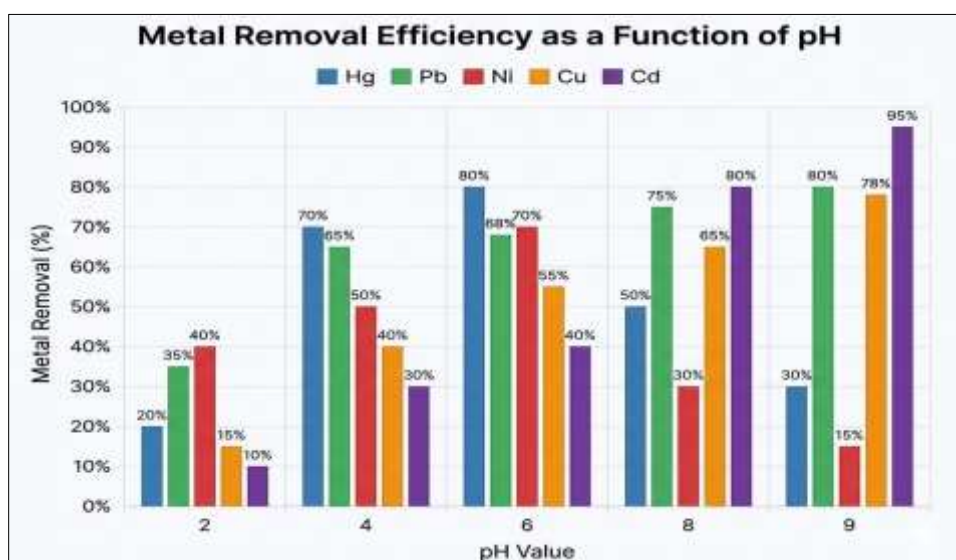


Fig 4: variation of removal metal% with pH variation

Effect of initial concentration of transition elements is an important variable, which determine the maximum uptake capacity of the polymer, Table 3 shows the uptake capacities of MFC resin for the single metal aqueous solution under study. From the plots obtained for each metal ion, the metal capacities of the present resin are quite comparable to those of resins employed earlier [24, 25].

Table 3: Comparison of the rates of metal ion up take: percentages attainment of equilibrium state

Time (hr)	Cu (II)	Ni (II)	Cd(II)	Hg(II)	Pb(II)
0.5	40	35	40	50	20
1	60	65	60	70	35
3	70	75	78	85	40
5	90	95	95	97	65

* $\text{MCl}_2 \cdot x\text{H}_2\text{O}$ or $\text{CuCl}_2 \cdot 2\text{H}_2\text{O} = 0.01\text{mol. lit}^{-1}$, volume = 5mL, pH = 4.0-8.5, temperature = 298 K, wt. FC = 20 mg

Conclusion

The results observed from elemental analyses, IR spectra and MS spectrum investigated the condensation of furfural

aldehyde with catechol to afford the new adsorbent resin, FC. As well as the spectroscopic data of UV-Visible-NI, molar conductivity and magnetic moments measurements confirmed the chelation of the new MFA with all metal ions undertaken through amino and hydroxyl groups with six-membered ring that is so stable according to the thermal and kinetic principle. However, the recovery and uptake metal ions study of copper(II), cadmium(II), lead(II), nickel(II) and mercury(II) in their aqueous solution with optimum conditions of temperature at 298 °K and PH of 3.5-8.0 provided new route to adsorb these toxic heavy metals from the water and encourage us to keep on the future study for others factors like interferences of mixed ligands and separation effect of mixtures for others metal ions. **Optimal pH for Overall Removal:** A pH range of 6.0 to 7.0 appears to be the most effective sweet spot for removing a broad spectrum of these metals simultaneously. **Targeted Removal:** If the specific goal is to remove Cadmium or Lead, a highly alkaline environment (pH 9) is superior. However, for Nickel or Mercury, neutral to slightly acidic conditions are preferred to avoid the formation of soluble

complexes. In case of Cu (II), Hg (II) and Ni(II), the plateau is observed at (pH = 6. 5).

Author Contributions

The contributions of authors for this new work involved the preparation, purification and characterization of the metal complexes with the resin of furfural-catechol by Miss Bayader F. Abbas. As well as the design of the resin isolation with its environmental structures of complexes were designed and fully discussed by Dr Mahmoud Al-jibouri.

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Conflicts of interest

Authors declare that no conflict of interests for publication the present work.

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