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Biosorption of Cu (II), Ni (II) and Pb (II) from synthetic waste water using *Asphodelus tenuifolius* Biomass: A wheat weed as biosorbent

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Abstract: Asphodelus tenuifolius (AST) a wheat weed, at present study, was taken as a biosorbent for the removal of heavy metals Cu (II), Ni (II) and Pb (II) ions from synthetic wastewater. The effect of variation of contact time, pH, adsorbent dose, concentration of metal ions and the temperature was studied. Optimal conditions for maximum adsorption were found, pH 5, initial metal ion concentration 10 mg/L, contact time 120 min, temperature 27 °C and 0.5 g adsorption dose. Equilibrium adsorption data were confirmed with Langmuir, Freundlich and Temkin isotherm models. Langmuir isotherm was found a better model for biosorption of metal ions. The value of Δ H° for metal ions was found in favour of chemisorptions and endothermic The outcomes indicate that AST is effective biosorbent and maximum removal of Cu (II), Ni(II) and Pb (II) ions by AST was found 90% 91% 93% respectively. A good biosorption capacity of AST biomass Qmax (mg/g) 38.46, 12.82 and 11.49 for Cu (II), Ni (II) and Pb (II) ions was achieved respectively. The metal binding capacity of *Asphodelus tenuifolius* for the removal of heavy metal ions from the synthetic wastewater is as the order of Cu (II) < Ni (II) < Pb (II).

Keywords: Biosorption, biosorbent, Asphodelus tenuifolius (AST), synthetic waste water, heavy metals.

1.0 Introduction: Due to the snappy flourish of many industries, laboratories, metallurgical process, explosive manufacturing, electroplating, painting wastes containing heavy metals is directly or indirectly liberated into the water system causing serious environmental pollution and affecting human health [1]. The metal elements having a density greater than 5g /cc, high atomic weight and non-degradable are considered heavy metals. The important toxic heavy metal elements are Cu, Ni, and Pb heaving densities 8.96, 8.9 and 11.34 g/cc in that order Copper (Cu), Nickel (Ni) and Lead (Pb) are three priority pollutants out of 129 listed by the Environmental Protection Agency (EPA) [2].

Copper is more toxic due to being non-biodegradable and carcinogenic [3]. Excess of copper in the body causes Wilson's disease [4]. A high dose of nickel is carcinogenic and causes a headache, chest pain rapid respiration, dizziness, cyanosis, extreme weakness and a very painful disease "nickel-itch" [5, 6]. Lead toxicity can result in encephalopathy neurobehavioral, hypertension, brain damage, kidney damage, liver damage and reproductive system. At even minute concentrations of Pb (II) ions may cause diseases such as anaemia, encephalopathy, hepatitis and nephritic syndrome [7]. The presence of lead in drinking water causes no relaxation according to the central pollution control board (CPCB). So it is important to remove the Pb (II) ions from drinking water.

The various methods have been used to remove heavy metal ions from synthetic waste water recently such as chemical precipitation, ion exchange, membrane process, reverse osmosis, electro-dialysis and solvent extraction [8]. These methods are non-economical and have many disadvantages such as cost, low removal efficiency, high energy consumption and generation of toxic sludge or other waste products that require disposal or treatment.

Biosorption is possessed having various advantages over conventional methods like low cost, high removal efficiency, minimization of biological and chemical sludge, regeneration of biosorbent, the possibility of metal recovery and no additional nutrient required [9, 10]. Recently, various biosorbent have been used for the removal of toxic heavy metal ions from wastewater like Bacteria: Gram-positive bacteria (*Bacillus* sp., *Corynebacterium* sp., etc.), Gram-negative Bacteria (*Escherichia* sp., *Pseudomonas* sp., etc), cyanobacteria (*Anabaena* sp., *Synechocystis* sp., etc), Fungi such as Molds (*Aspergillus* sp., *Rhizopus* sp.), mushrooms (*Agaricus* sp., *Trichaptum* sp.,) and yeast (*Saccharomyces* sp., etc). Industrial wastes such as Fermentation wastes, food/beverage wastes, activated sludge, anaerobic sludge is used as an adsorbent [11]. Adsorption onto activated carbon is a well-known method for removing toxic metal ions, but the high cost of activated carbon restricts its use in developing countries [12]. The main objective of the present work is to investigate a biosorbent for the removal of Pb (II), Cu (II) and Ni (II) ions from synthetic waste water. AST is a very bad and nocuous weed found in wheat, mustard, linseed, chickpea and lentil field in India and Pakistan [13]. AST is found in almost seventeen countries around the world [14]. In the present study, *Asphodelus tenuifolius* is a highly noxious wheat weed used as an effective biosorbent for the removal of copper, nickel and lead from synthetic wastewater.

Materials and methods:

Biosorbent:

AST biomass was collected from the wheat field of Kaneri Mahoba Bundelkhand area of Utter Pradesh. The Biomass was then washed with double distilled water, dried overnight at 70° C in a hot air oven (Popular Traders S.N.-1680). The dried biomass was then ground in an electric grinder. The powdered biomass was activated with 0.1N HNO₃.at room temperature for 24 hours. The obtained biomass was dried in a hot air oven at 333 K for 2 days and then subjected to pass through the 63 microns (170 BSS) sieve. The sieved biomass was then stored in an airtight bottle. The image of the plant and the powder is shown in Figure 1.



Figure 1: (a) Asphodelus tenuifolius plant (b) the powder form of Asphodelus tenuifolius

Adsorbate: CuSO4.5H2O, NiSO4.6H2O and PbSO4 were obtained in analytical grade (Merck Co.) and used withoutfurther purification synthetic 1000ppm stock solution was prepared for each metal.

$$1 gram metal equivalent = \frac{molecular weight \times 100}{atomic weight \times n \times Purity}$$
(1)

Where 'n' is the number of metal atoms in a one-mole substance

Copper solution: 3.927 grams of CuSO4.5H2O was added to the 100ml of distilled water in a 1000ml volumetric flask. It was dissolved by shaking and the volume was made up to the mark. The Cu(II) ion concentration of this solution was 1000 mg/L.

Nickel solution: 4.477 grams of NiSO4.6H2O was added to the 100ml of distilled water in a 1000ml volumetric flask. It was dissolved by shaking and the volume was made up to the mark. The Ni (II) ion concentration of this solution was 1000 mg/L.

Lead solution: 2 gm of lead sulphate was dissolved in distilled water and make a solution of 1000ml. The concentration of this solution was 1000 mg/l.

Batch mode of Biosorption Studies:

To investigate the effect of various parameters such as adsorbate concentration (10-100 ppm), biosorbent dosage (0.1-1 gm in 100ml solution), agitation time (40 -180 min), pH (2-9) and temperature (17-47^oc) batch mode adsorption studies for individual metal ions were carried out. The solution containing adsorbate and biosorbent was taken in 100 ml conical flasks and agitated at 120 rpm in a thermal shaker at predetermined time intervals. The solution was then filtered with Whatman No-42 filter paper.

Metal ions analysis:

After adsorption, the final residual metal ion concentration is measured by an Atomic Absorption spectrophotometer (AAS). To estimate the percentage removal of metal ions from an aqueous solution, the following equation is used.

% removal of metal ion =
$$\frac{(C_{initial} - C_{final})}{C_{initial}} \times 100$$
 (2)

Where $C_{initial}$ and C_{final} are the concentration (mg/l) of adsorbate before and after adsorption, respectively. The adsorption capacity and the quantity of metal adsorbed at equilibrium is calculated by using the equation (3):

$$Q_e = \frac{(C_i - C_e) \times V}{m} \tag{3}$$

'V' is the volume of solution in (L), 'm' is the mass of biosorbent in (g) and Q_e is the metal quantity adsorbed at equilibrium in (mg/g)

Results and discussion:

Functional Group Characteristics of the AST biomass:

To determine the main functional groups present in the AST biosorbent the FT-IR spectra were recorded after treatment of biosorbent with 0.1N HNO₃. The functional groups of AST biosorbent corresponding to infrared absorption bands are shown in Figure 2. In Figure 2, the sharp absorption band at 3328 cm⁻¹ and 3113cm⁻¹ indicates the presence of the -OH group in the biosorbent. The presence of a strong band at 1640, 1590 cm⁻¹ indicates the presence of the (C=O) group in the biosorbent. IR absorption bands near 1300cm⁻¹ indicate the presence of (C-O) of –COOH group. These all the functional groups are responsible for the biosorption of Cu (II), Ni (II), and Pb (II) onto AST



Figure 2: FT-IR spectrum of activated Asphodelus tenuifolius biomass before adsorption

Effect of various parameters Effect of Contact Time:

The variation in the percentage removal of heavy metals with contact time using 0.5g/100ml of AST biosorbent at pH 5.0 with varying initial metal ions concentration ranging from 10 ppm to 100 ppm is shown in Figure 3, 4 and 5. For Pb (II) Metal ions, the percentage removal is observed nearly 92% at 120 minutes. The percentage removal in all cases is observed comparatively lower for 40 min. The removal efficiencies are increasing at higher contact time up to 120 min and then gradually decrease at 180 minutes, maximum removal obtained at 120 minutes. In the case of Cu (II) and Ni (II) ions, the removal efficiency is also increased with time up to 120 minutes. The maximum percentage removal of copper and Nickel ions is 87% and 89% respectively at 120 minutes. The graph indicates that on increasing concentration the percentage removal of metal ions decreases. This is due to the concentration gradient which acts as an increasing driving force to overcome the resistance to mass transfer of metal ions between the aqueous and solid phases [15, 16].





Figure 5: Effect of contact time on percentage removal of Pb (II) ions onto AST Biomass

Effect of pH:

pH is one of the most important parameters and affects the surface charge of the biosorbent, degree of ionization and specification of adsorbate [17, 18]. Figures 6, 7 and 8, show the effect of pH on heavy metals removal efficiencies of AST biomass. The study is conducted at an initial metal ions concentration of 10, 30, 50 and100ppm in 100ml solution, constant biosorbent dose 0.5g/100ml. For all the heavy metal ions, the pH ranges from 2.0 to 9.0. The percentage adsorption increases with pH to attain a maximum at pH 5.0 and thereafter it decreases with a further increase in pH. The maximum removal of Cu (II), Ni (II) and Pb (II) at pH 5.0 is found to be nearly 90%, 90% and 93%, respectively. At a low pH range, metal is present predominantly as metal ions in the adsorptive solution, there is a competition between H⁺ and M⁺² ions for adsorption at the ion exchangeable sites, leading to a low removal of metal. The extensive repulsion of metal ions due to protonation of the biosorbent surface at lower pH may be another reason for the decrease in adsorption of metal in the lower pH range [19-22]. This results in a decrease of metal ions due to the formation of soluble hydroxides of metal ions [23].



Figure 8: Effect of pH on percentage removal of Pb (II) ions onto AST Biomass.

Effect of biosorbent Dose:

Figure 9, 10 and11 shows the adsorptive removal of heavy metal as a function of biosorbent dose ranging from 0.1 to 1gm. On increasing the biosorbent dose the percentage removal of heavy metals is also increasing. Fig. 9, 10 and 11 showed that the maximum removal of Cu (II), Ni(II) and Pb (II), are 87%, 91% and 87% respectively at 0.5 gm dose of the biosorbent. This result can be explained by the fact that the biosorption sites remain unsaturated during the biosorption reaction whereas the number of sites available for biosorption sites increased by increasing the biosorbent dose [24-26].



Figure 9: Effect of Adsorbent dose on percentage removal of Cu (II)ion Figure 10: Effect of Adsorbent dose on percentage Onto AST Biomass.





Effect of initial metal ion concentration:

The effect of heavy metal ion concentration at 0.5 gm biosorbent dose, pH 5.0, 120 rpm and 120 minutes contact time on the percentage removal of heavy metals onto AST biomass is shown in Figure 12. The figure reviles that the percentage removal decreases with the increase in initial metal ion. High removal efficiency is achieved at 10mg/l of metal ion concentration; this is because, at lower initial metal ion concentrations, sufficient adsorption sites are available for the adsorption of metal ions [16, 27]. The maximum removal of copper and nickel ions is found to be 90% and 91% respectively at 10 ppm. The maximum removal of Pb (II) ion is found to be 93 % at 10 ppm. Hence, the percentage removal of heavy metals depends on the initial metal ions concentration and decreases with an increase in initial metal ions concentration.



Figure 12: Effect of concentration on percentage removal of metal ions onto AST biomass

Adsorption isotherms:

An adsorption isotherm is characterized by certain constant values, which can be used to describe how solutes interact with biosorbent and so is critical in optimizing the use of biosorbent [28]. Three kinds of adsorption isotherm equations such as Langmuir, Freundlich and Temkin isotherms have been applied in the present study. Langmuir adsorption isotherm:

Langmuir's adsorption isotherm model describes quantitatively the formation of a monolayer of adsorbate on the surface of the biosorbent sites and thereafter no further adsorption takes place at that site. The Langmuir isotherm is valid for monolayer adsorption onto a surface of biosorbent having a finite number of identical active sites. To analyze the adsorption study, Langmuir represented the following equation:

$$Q = Q \frac{K_L C_e}{\max 1 + K_I C_e}$$
(4)

Langmuir adsorption parameters were determined by transforming the Langmuir equation (4) into the linear form: [29].

$$\frac{\underline{C_e}}{Q_e} = \frac{1}{Q_{max}K_L} + \frac{\underline{C_e}}{Q_{max}} \tag{57}$$

Where Qe and Ce correspond to the mg of metal adsorbed per gm of biosorbent and residual metal concentration in the solution when in equilibrium. KL (L/mg) and Qmax are Langmuir constant and a maximum capacity of adsorption (mg/g) respectively. Values of Langmuir parameters Qmax and KL were calculated from the slope and intercept of the linear plot of Ce/Qe versus Ce for Cu (II), Ni (II) & Pb (II) Figure 13[30]. The values of Qmax, KL, and correlation coefficient R² are listed in Table 2.



Figure 13: Langmuir isotherm for the biosorption of Cu (II), Pb (II) and Ni (II) ions onto AST biomass.

R_L Parameter based on Langmuir isotherm:

The essential features of the Langmuir isotherm may be expressed in terms of equilibrium parameter R_L , which is a dimensionless quantity referred to as separation factor or equilibrium parameter [31]:

$$\mu = \frac{1}{1 \pm K_{\rm eff}} \tag{6}$$

Where C_{i} = initial concentration, K_{L} = the constant related to the Kenfergy of adsorption (Langmuir constant), R_{L} value indicates the adsorption nature to be either unfavorable if $R_L > 1$), linear if $R_L = 1$, favorable if $0 < R_L < 1$ and irreversible if $R_L = 0$. Table 1 confirms that the R_L is greater than 0 but less than 1 indicating that Langmuir isotherm is favorable.

C _i (mg/L)	Cu (II)	Ni (II)	Pb (II)	
10	0.952	0.420	0.322	
30	0.869	0.194	0.137	
50	0.800	0.126	0.087	
100	0.666	0.067	0.045	

Table 1: R_L values based on the Langmuir isotherm

Freundlich isotherm:

The Freundlich model is used to estimate the adsorption intensity of metal ion relapse on a heterogeneous adsorption surface by multilayer adsorption [32]. For the analysis of the present study, the linear form of the Freundlich equation is used;

$$logQ_e = logK_f + \frac{1}{n}logC_e$$
(7)

Where K_f and 'n' are Freundlich constants represent the adsorption intensity and adsorption capacity, respectively. The Freundlich constants K_f and 'n' are obtained from the slope and intercept of the plots of log Q_e versus log C_e (Figure 14). It gives a straight line with a slope of 1/n and intercept of log K_f . The calculated values of K_f and 'n' are given in Table 2. The literature reviles that values of 'n' in the range of 2-10 exhibit well adsorption, 1-2 exhibit moderately difficult and less than 1 implies low adsorption [33]. The values of 'n' are 2.132, 2.43 and 0.8163 respectively for Ni (II), Pb (II) and Cu (II). The R^2 values are0.993, 0.976 and 0.962 for Cu (II), Ni (II) and Pb (II) ions respectively.



Figure 14: Freundlich isotherm for the biosorption of Cu (II), Pb (II) and Ni (II) ions on to AST biomass

Temkin isotherm:

The Temkin isotherm model is used for the study of biosorption equilibrium. The adsorption is characterized by a uniform distribution of binding energies and adsorbate-biosorbent interaction [34]. The linear form of the Temkin isotherm model is expressed as Eq. (8) [35].

$$Q_e = b_T \ln A + b_T \ln C_e \quad (8)$$

Where br is the Temkin constant related to the heat of sorption (J/mole) and A is the adsorption isotherm constant in (L/g). The values of br and A are calculated from the slope and intercept of the linear plot of Q_e versus ln C_e (Figure 15) and are given in Table 2.



Figure 15: Temkin Isotherm for the biosorption of Cu (II), Pb (II), and Ni (II) ions on to AST biomass

	Langmuir		Freundlich			Temkin				
Metal2+	Q _{max}	K _L	R ²	K _f	1/n	n	R ²	b _T	А	R ²
Cu	38.461	0.004703	0.999	-0.468521	1.225	0.816327	0.993	12.32	1.039646	0.936
Ni	12.82	0.137809	0.995	-0.916876	0.469	2.132196	0.976	5.448	1.360705	0.982
Pb	11.494	0.200461	0.997	-0.417936	0.413	2.421308	0.962	4.697	1.671162	0.995

Table 2: Biosorption isotherm constants for sorption of Cu (II), Ni (II) and Pb (II) ions onto AST biomass

The maximum adsorption capacity (Q_{max}) for the biosorption of Pb (II), Ni (II), Cu (II) ions onto AST and the other constant values calculated from the Freundlich and Temkin models are listed in Table 2. From Table 2, it is clear that the Langmuir isotherm gives a better fit with the experimental data (correlation coefficient R²> 0.99) in the adsorption of metal ions onto AST. The higher correlation coefficient values confirm the monolayer coverage of metal ions onto the adsorbent [36]. The Freundlich constant 'n' is found to be greater than one and indicates that the adsorption of metal ions onto adsorbent is favorable.

Thermodynamic Studies:

From Eq. 10 an

Adsorption of Cu (II), Ni (II) and Pb (II) ions onto AST biomass is investigated at five different temperatures from 290 to 320 K. The initial concentration of metal²⁺ ions is 10 mg/L. The biosorption efficiency as a function of temperature is shown in Figure 16. Figure 17 indicates that an increase in temperature results in an increase in the biosorption until a peak is reached on a further increase in temperature leads to a decrease in the biosorption efficiency. The optimum temperature for maximum biosorption is obtained 27^{0} C. The binding of metal²⁺ ions with AST biomass is considered a reversible reaction. The equilibrium constant (K_{eq}) is calculated using the following equation (9).

$$X_{eq} = \frac{L_{ad}}{C_e} \qquad (9)$$

Where the C_{ad} (mg/l) and the C_e (mg/l) are the concentration of solute adsorbed at equilibrium and the solute concentration in solution at equilibrium respectively. The K_{eq} is related to the change in free energy by Eq. (10). The relationship of Gibb's free energy change (ΔG°) to enthalpy change (ΔH°) and entropy change (ΔS°) of adsorption is expressed as (11).

$$\Delta G^{\circ} = -RT ln K_{eq}$$
(10)
$$\Delta G^{\circ} = \Delta H^{\circ} - T \Delta S^{\circ}$$
(11)
$$ln K_{eq} = \frac{\Delta S^{\circ}}{R} - \frac{\Delta H^{\circ}}{RT}$$
(12)

The values of ΔH° and ΔS° are determined from the slope and intercept of the linear plot of ln K_{eq} versus 1/T of Eq.12 (Figure 17). The calculated thermodynamic parameters are listed in Table 3. Literature reviles That the high enthalpy values of ΔH° (80-240 kJ/mole) indicate that chemisorptions adsorption is embraced during the process of adsorption [37]. In the present study the change in enthalpy (ΔH^{0}) of Cu (II), Ni (II) and Pb (II) is found to be +127.703, +130.222 and +146.243kJ/mole respectively. Thus values of ΔH° indicated that the adsorption is chemisorptions process is endothermic. A positive value of ΔS° indicates the increasing randomness at the solid-liquid interface during the adsorption of all three metal ions on the biosorbents. The negative values of ΔG^{0} for all the metal ions indicate the process is spontaneous.

Metal ions	Temperature(K)	ΔG° (KJ/mole)	Keq	ΔH ^o (KJ/mole)	ΔS ^o (KJ/mole)	
Cu (II)	290	-1.292	1.709			
	295	-3.313	3.861		+0.444	
	300	-5.743	10.000	+127.703		
	310	-2.461	2.597			
	320	-2.252	2.331			
Ni (II)	290	-1.462	1.834			
	295	-3.510	4.184		+0.453	
	300	-6.005	11.111	+130.222		
	310	-2.676	2.824			
	320	-2.372	2.439			
Pb (II)	290	-1.535	1.890			
	295	-3.946	4.997			
	300	-6.632	14.285	+146.243	+0.509	
	310	-2.936	3.125			
	320	-2.645	2.702			

Table 3: Calculated thermodynamic parameters of Cu (II), Ni (II) and Pb (II) ions onto AST Biomass



Figure 16: Effect of temperature on Biosorption of Cu (II), Ni (II) & Pb (II) ions onto AST



Comparison with other used biosorbent:

Table 4 represents the comparison of biosorption capacity (Q_{max}) of AST biomass for Cu (II), Ni (II) and Pb (II) ions with that of various biosorbents reported in the literature. It is seen from the table that AST has a good adsorbent capacity with Q_{max} (mg/g) 38.46, 12.82 and 11.49 for Cu (II), Ni (II) and Pb (II) ions respectively compared with other biosorbents. Therefore, *Asphodelus tenuifolius* biomass as biosorbent has important potential for the removal of Cu (II), Ni (II) and Pb (II) ions from an aqueous solution.

	The second se			
Adsorbents	$Q_{max}(mg/g)$			references
	Cu (II)	Ni (II)	Pb (II)	
Artemisia vulgarise	0.88	-	-	[25]
Gossypium hirsutum L.	-	-	2.78	[38]
Sophora japonica	35.48	-	-	[39]
Rubus ellipticus	4.48	-	3.38	[16]
Pyrus pashia	4.73	-	5.73	[18]
Waste pomace of olive oil	-	10.64	-	[40]
Chitosan	16.8	2.4	-	[41]
Chitosan acetate crown ether	31.3	4.1	-	[42]
Asphodelus tenuifolius	38.46	12.82	11.49	Present
				study

Table 4: comparison between low-cost adsorbents and Asphodelus tenuifolius in the aspect of adsorption capacities (Q_{max})

4.0 Conclusions:

AST is a waste wheat weed that is an effective biosorbent for the removal of Cu (II), Ni (II) and Pb (II) ions from wastewater. Experimental result shows that maximum removal of Lead ion by *Asphodelus tenuifolius* waste wheat weed at optimum condition (pH 5.0, 120 min. contact time, 0.5g/100ml biosorbent dose and 10 ppm concentrations) is 93%. The maximum removal efficiency for Cu (II) & Ni (II) ions is 90% & 91% respectively at optimum condition (pH 5.0, 120 min.

contact time and 0.5g/100ml biosorbent dose, 10 ppm concentration). The adsorption process results showed that the Langmuir isotherm model is best fitted for the adsorption of Cu (II), Ni (II) and Pb (II) ions onto AST biomass. The negative value of ΔG^0 indicates that the adsorption of Cu (II), Ni (II) and Pb (II) ions onto AST is spontaneous and feasible.

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