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Removal of Traces of Silver Ions from Aqueous Solutions Using Coconut Husk as a Sorbent

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ABSTRACT

Sorption of silver ions onto coconut (*Cocos nucifera*) husk has been studied in detail using radiotracer technique. Maximum sorption (98%) of Ag ions (8.23×10^{-4} M) onto sorbent surface is achieved from 4.5 mL of 0.1 M HNO_3 solution within 20 min using 200 mg of coconut husk. The sorption data follow the Freundlich and Dubinin-Radushkevich (D-R) isotherms. The values of Freundlich characteristic constants $1/n = 0.98 \pm 0.09$ and of $A_m = 0.35 \pm 0.05$ m mole g^{-1} and D-R constants $\beta = -0.006207 \pm 0.000439$ kJ^2 $mole^{-2}$, $X_m = 0.57 \pm 0.08$ m mole g^{-1}, and $E = 9.0 \pm 0.3$ kJ $mole^{-1}$ have been computed. The variation of sorption with temperature yields thermodynamic parameters $\Delta H = 61.3 \pm 5.4$ k J $mole^{-1}$, $\Delta S = 241.9 \pm 18.7$ J mol^{-1} K^{-1},

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and $\Delta G = -10.6 \pm 0.09\,\mathrm{kJ\,mole^{-1}}$ at 298 K. The positive enthalpy and negative value of free energy reflect the endothermic and spontaneous nature of sorption respectively. Sulphate, Al(III), Ni(II), and Ba(II) enhance the sorption significantly whereas Ce(III) and citrate cause its suppression appreciably. To check the selectivity of the coconut husk, the sorption of a number of metal ions on the sorbent surface has been measured under optimized conditions. The results indicate that coconut husk can be used to separate Ag ions from Cs(I), Zn(II), Cr(III), Co(II), and Se(IV).

Key Words: Ag(I) ions; Sorption; Radiotracer technique; Thermodynamics; Sorption isotherms; Coconut husk.

INTRODUCTION

Silver, a heavy, noble, and precious metal has the highest thermal and electrical conductivities among all the metals. Silver, its compounds and alloys have wide applications and usage in jewelry and silverwares, as oxidation catalyst in the production of certain organic materials, in corrosion-resistant storage batteries, in dental amalgams and dentistry, in brazing alloys, in fuses, in electrical and electronic contact points in electrical devices and in medical instruments, in special military devices, in medicines, pharmaceuticals, paints, inks, in silver mirrors, glass polishers and colors in electroplating, in photographic industry and processes, in engine pistons, heavy-duty bearings in aircraft engines, electrical windings in motors operating at high temperatures, in electrical coatings to ceramic, in ceramic capacitors, in chemical industry, in autoclave piping and similar equipment.[1,2]

The recovery of silver from scrap produced by arts and industry is important from economical and environmental points of view. A large number of cheaper materials including agricultural waste namely maize cob,[3] pine,[4] neem and mango[5,6] bark, onion skins,[7] apple waste,[8] groundnut husk,[9] rice husk,[10] peanut hulls, rice straw and sugarcane bagasse,[11] sawdust[12] and coconut husk[13,14] have been exploited for the recovery of metal ions from aqueous solutions. This communication presents the results of our investigation using coconut husk as a sorbent for the accumulation of traces of silver ions from aqueous solutions. The main objective of this study is to check the efficacy of coconut husk, a cheaper commodity abundantly available, for the recovery of silver ions from aqueous media.

EXPERIMENTAL

Radiotracers

The radiotracer ^{110m}Ag ($t_{1/2} = 249.8$ d) and other radionuclides used in this study, i.e., ^{51}Cr, ^{60}Co, ^{65}Zn, ^{75}Se, ^{99m}Tc, ^{115m}Cd, ^{124}Sb, ^{134}Cs, ^{147}Nd, $^{152+154}$Eu, ^{170}Tm, and ^{203}Hg have been prepared by irradiating specpure metals or their appropriate compounds in 10 MW swimming pool-type research reactor of this institute for suitable period at a neutron flux of $5 \times 10^{13}\,\mathrm{n\,cm^2\,s^{-1}}$. After appropriate cooling the irradiated metal was dissolved in concentrated nitric acid. To remove excessive acid from the sample, it was diluted with deionized water and heated to dryness. This procedure was repeated thrice and the sample was diluted with deionized water. The radiochemical and radionuclidic purity of the tracers was monitored on a 4k series of 85 Canberra multichannel analyzer coupled with a 25 cm^3 Ge(Li) detector.

Reagents

The chemicals employed in the present investigation were of analar or equivalent grades and all solutions were made in doubly distilled deionised water. Buffer solutions having ionic strength of 0.1 M were prepared by mixing appropriate volumes of 0.1 M solutions of HNO_3 and KNO_3, CH_3COOH and CH_3COONa; and H_3BO_3 and NaOH for pH 3, 4–6 and 7–10 respectively.

Coconut Husk

Coconut husk, a by-product of coconut fruit, has been procured from the local market and washed extensively with deionized water. It was dried overnight at 105°C. Later, it was split into threads and barks. The thread-like portion was heated overnight at 105°C and ground in a pestle and mortar up to 40 mesh size (0.42 mm ϕ) and used as a sorbent for Ag(I) ions. The BET surface area using nitrogen as a standard has been assessed to be $<2\,\mathrm{m^2\,g^{-1}}$.

Sorption Measurements

Radiotracer and batch techniques were employed to measure sorption of silver ions. 5 cm^3 of an electrolyte of specific pH or acid concentration was taken in a glass culture tube and known concentration of Ag(I) tracer was added to it and mixed uniformly. An aliquot of 0.5 cm^3 was withdrawn for gross gamma counts (A_o). The remaining solution was shaken with 100 mg of coconut husk for 30 min on a Stuart scientific wrist-action shaker. The phases were separated by centrifugation for 3 min. Later, 0.5 cm^3 of aliquot again was taken out for radioassay on a Tennelec gross gamma counter equipped with a 30 cm^3 well-type NaI(Tl) crystal.

The sorbed concentration of Ag(I) ions was computed by the difference in the activity of aliquots before (A_o) and after shaking (A_e). The percent sorption and distribution coefficient (R_d) were calculated as

$$\% \text{sorption} = \frac{A_o - A_e}{A_o} \times 100 \tag{1}$$

$$R_d = \frac{\text{Amount of metal on husk}}{\text{Amount of metal in solution}} \times \frac{\text{Volume of solution (V)}}{\text{Mass of dry husk (W)}} = \text{cm}^3\,\text{g}^{-1} \tag{2}$$

The percent sorption and the distribution coefficient are correlated as

$$\% \text{sorption} = \frac{100\ R_d}{R_d + (V/W)} \tag{3}$$

All the experiments were performed at controlled temperature 28 ± 2°C or at temperatures specified otherwise. Linear regression analysis was carried out to evaluate slopes and intercepts of the plots and for statistical analyses of the data. The correlation coefficients for all regression analyses were in the range of 0.9785–0.9903. The results were the average of at least triplicate independent measurements and were reproducible to within ± 3% in most cases.

RESULTS AND DISCUSSION

The distribution of metal ions between solid-liquid phases is influenced by cation-anion exchanger, chemical and physical sorption, precipitation and self-diffusion in the solid phase. Laboratory scale studies of sorption have the advantage that various factors controlling the sorption process may be varied systematically to ascertain their part in the mechanism.

Selection of Electrolyte

The composition of sorptive solution and nature of electrolyte is an important factor which influences the sorption of trace elements onto sorbent surface.[15] Therefore, the sorption of traces of silver ions has been investigated to find out the best electrolyte giving maximum sorption. A number of sorptive solutions like deionized water, nitric acid (0.001–1.0 M), and buffers of pH 3–10 were tested. The results are given in Table 1 along with sorption edges, i.e., sorption percent or distribution coefficient vs. pH. The dilute nitric acid solution (0.1 M) has been found to be the most suitable electrolyte giving maximum accumulation of Ag(I) ions along with pH 6 buffer. Similarly for quantitative sorption of silver onto thiol cotton fiber is shown in the pH range of 1–8[16] and on chelating resin containing thiol and amine groups at pH 6.[17] At pH 6 more than 90% sorption of Cu(II) ions onto byospsis plant[18] is also reported, which can be compared with similar extent of silver ions sorption onto coconut husk. However, for further studies 0.1 M nitric acid solution has been selected. The influence of pH may cause changes of the chemical state and of elemental species in solution, variation of potential between solid and liquid phases, electrostatic attraction or repulsion between charged surface and elemental species in solution and the competition of H_3O^+ ions with these species in solution in ion-exchange processes.

Another important parameter, i.e., of the agitation time between sorbent and sorbate was varied from 1 to 60 min. Fig. 1 shows sorption profile of silver ions as a function of time. The percent sorption increases from 75 to

Table 1. Sorption of Ag (I) ions (2.74×10^{-6} M) onto coconut husk (100 mg) after 20 min agitation time.

Electrolyte	R_d ($cm^3\,g^{-1}$)	%Sorption
Deionized water	187	80.6
1.00 M HNO_3	287	86.5
0.5 M HNO_3	306	87.2
0.2 M HNO_3	350	88.6
0.1 M HNO_3	366	89.1
0.01 M HNO_3	310	87.3
0.001 M HNO_3	47	51.1
pH 3 buffer	349	88.6
pH 4 buffer	365	89.0
pH 5 buffer	368	89.1
pH 6 buffer	434	90.6
pH 7 buffer	131	80.5
pH 8 buffer	150	77.2
pH 9 buffer	207	77.6
pH 10 buffer	171	78.5

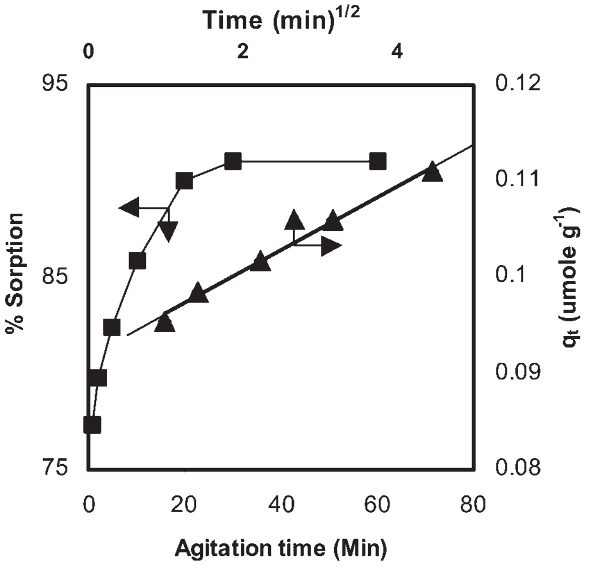

Figure 1. Sorbed concentration of Ag(I) ions onto coconut husk as a function of shaking time.

almost 90% from 1 to 20 min and remained constant up to 60 min. Therefore, 20 min agitation time between sorbate and sorbent has been employed for further investigations. According to this figure, equilibrium is achieved after 20 min. This can be compared with 40 min agitation time for two coinage metals, i.e., Cu(II) and Au(III) sorption onto blue algae and other bio materials.[18]

Kinetics of Sorption

The kinetics of sorption of Ag(I) ions onto coconut husk surface has been monitored applying different equations. For example Morris-Weber equation in the following form:[19]

$$q_t = R_D\sqrt{t} \tag{4}$$

where q_t = sorbed concentration at time ‘t’ and R_D is intraparticle diffusion rate. The plot q_t vs. $\sqrt{t}$ is given in Fig. 1, which yields a straight line. From the slope of this linear plot intraparticle diffusion rate, R_D, has been computed to be 4.4 n mole g^{-1} $min^{-1/2}$. The data have been subjected to Reichenberg relationship, where

$$F = \frac{q_t}{q_e} \tag{5}$$

is the ratio between q_t and q_e representing sorption concentrations at time 't' and at equilibrium. This value of F can be related to

$$F = \left(1 - \frac{6}{\pi^2}\right)e^{-Bt} \tag{6}$$

where Bt is a mathematical function which can be calculated at each value of F. The value of Bt can be more simplified[20] as

$$Bt = \ln 0.39256 - \ln F \tag{7}$$

The plot of Bt vs. time is depicted in Fig. 2, which is a straight line and does not pass through the origin. In other words, the rate of sorption is not controlled by particle diffusion but may be film diffusion or chemical control.

Lagergren equation[21]

$$\log(q_e - q_t) = \log q_e - \frac{kt}{2.303} \tag{8}$$

where q_e, q_t and t have usual meanings and k is the first-order rate constant, has been tested. The plot of log $(q_e - q_t)$ vs. time is given in Fig. 2. The rate constant k, from the slope of the plot comes out to be 0.12 min^{-1}.

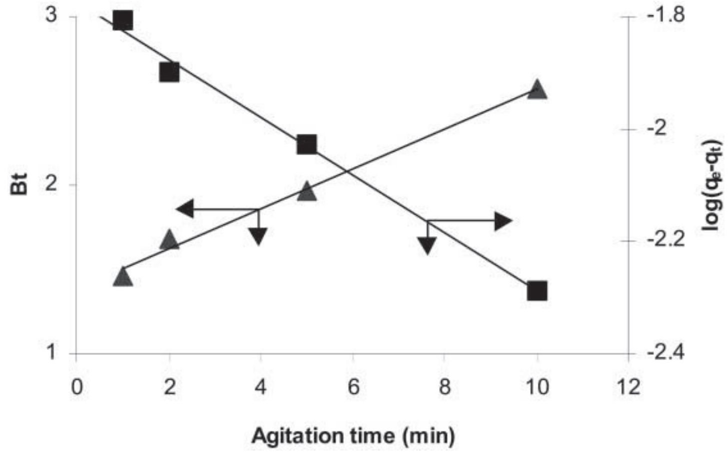

Figure 2. Reichenberg and Lagergren plots of Ag(I) ions sorption onto coconut husk.

This can be compared with the value of 0.177 min^{-1} evaluated for Ag(I) sorption onto polyurethane foam (PUF).[22]

The accumulation of traces of silver ions onto sorbent surface was also investigated as a function of sorbent dosage from 10–300 mg. The results are shown in Fig. 3. Percent sorption increases with an increase in the amount of sorbent and attains a value around 90% showing maximum sorption at 100 mg and then remains constant up to 300 mg. 200 mg of coconut husk was used for further experiments.

Sorption Isotherms

The concentration of silver ions in sorptive medium also influences its sorption.[15] The effect of sorbate concentration was monitored over 300-fold from 2.74×10^{-6} to 8.23×10^{-4} M. The results are given in Fig. 4. The distribution coefficient registers an increase with increasing silver concentration and attains almost a constant value around 5×10^{-4} M. The sorption data was subjected to different sorption isotherms in linear form to evaluate constants. The Langmuir model did not fit the data to an acceptable degree and gave higher errors than Freundlich and D-R. It is envisaged that Freundlich and D-R isotherms are most suitable for our sorption

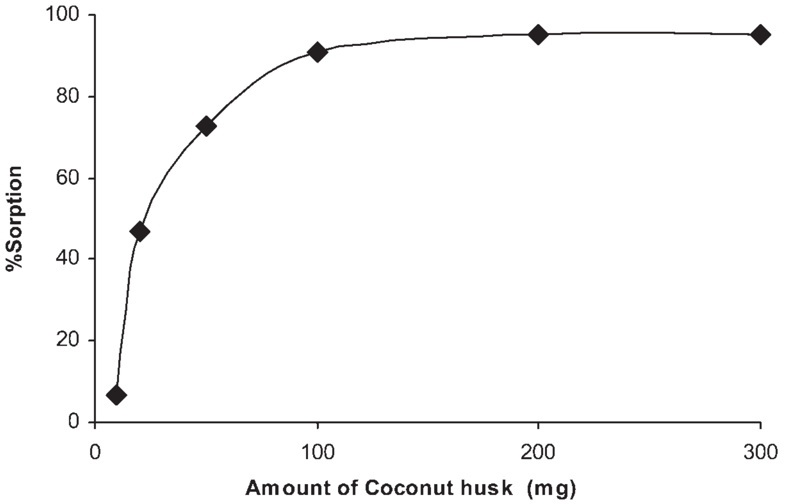

Figure 3. Effect of the amount of sorbent on the sorption of Ag(I) ions onto coconut husk.

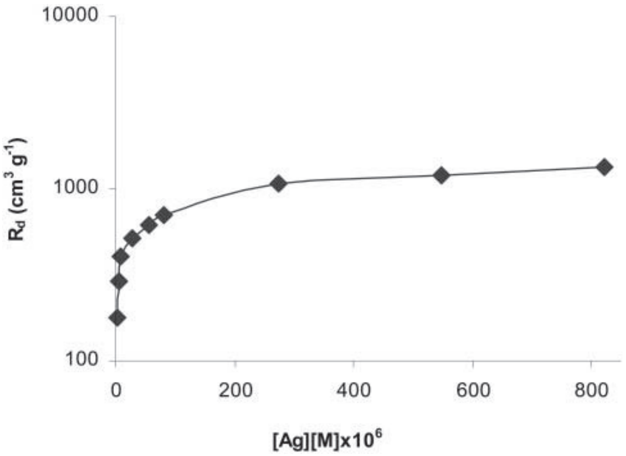

Figure 4. Effect of the concentration of Ag(I) on its sorption onto coconut husk from 0.1 M HNO_3.

system. The soption follows both Freundlich and Dubinin-Raduskevich (D-R) isotherms. Fig. 5 illustrates Freundlich sorption isotherm in the following linearized form.[23]

$$\log C_{ads} = \log A + \frac{1}{n} \log C_e \tag{9}$$

where C_{ads} represents adsorbed concentration of silver ions onto sorbent surface and C_e is the equilibrium concentration of silver in solution and

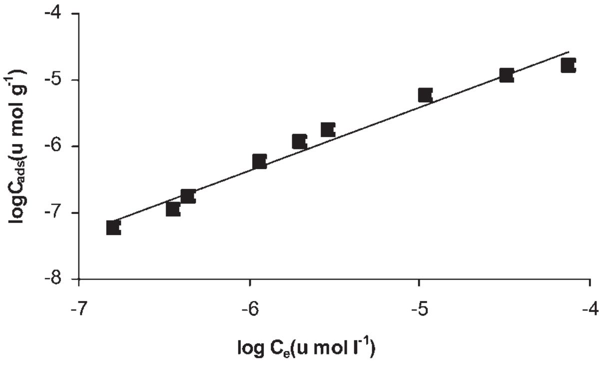

Figure 5. Freundlich sorption isotherm of Ag(I) ions onto coconut husk.

A gives the value of sorption capacity and $1/n$ is a Freundlich constant which is always less than unity. The plot of log C_{ads} vs log C_e is given in Fig. 5 which is a straight line over the entire concentration range of Ag(I) ions investigated. This isotherm assumes heterogeneity of the surface and envisages exponential distribution of sites and their energies.[24] Steep slope, i.e., $1/n$ close to unity indicates higher sorptive capacity at higher concentration. From the slope and intercept of the linear plot the values of Freundlich constants, i.e., $1/n = 0.98 \pm 0.09$ and of sorption capacity, $A_m = 0.35 \pm 0.05\,\mathrm{m\,mole\,g^{-1}}$ have been estimated. This value of A_m is of the same order of magnitude as of $0.15 \pm 0.03\,\mathrm{mmol\,g^{-1}}$ computed for Ag(I) sorption onto (PUF)[22] and of $0.3\,\mathrm{m\,mole\,g^{-1}}$ for Ag(I) on thiol cotton fiber[16] and of 0.73 and $0.54\,\mathrm{m\,mole\,g^{-1}}$ on black oak bark and on milorganite, respectively,[25] and $1.2\,\mathrm{m\,mole\,g^{-1}}$ on chelating resin of thiol and amine groups.[17]

The sorption data were also subjected to test Dubinin-Radushkevich (D-R) isotherm in the following linearized form.[26]

$$\ln C_{ads} = \ln X_m - \beta\varepsilon^2 \tag{10}$$

where compose absorbed concentration, C_{ads}, the sorption capacity X_m, are in $\mathrm{mole\,g^{-1}}$. β is a D-R characteristic constant, which is related to Polanyi potential, ε

$$\varepsilon = RT\ln\left(1 + \frac{1}{C_e}\right) \tag{11}$$

where R is a gas constant in $\mathrm{kJ\,mole^{-1}}$ and T is temperature in Kelvin. The value of β can be correlated to mean free energy of sorption, E, by the following relationship,[27] i.e.,

$$E = \sqrt{\frac{1}{-2\beta}} \tag{12}$$

If ln C_{ads} is plotted against ε^2, a straight line may yield the values of β and X_m from the slope and intercept of the resultant plot. The value of E, can be evaluated using equation (12). The D-R plot is shown graphically in Fig. 6, which is a straight line over the entire concentration of sorbate employed. The values of $\beta = -0.006207 \pm 0.000439\,\mathrm{kJ^2\,mole^{-2}}$, $X_m = 0.57 \pm 0.08\,\mathrm{m\,mole\,g^{-1}}$, and of $E = 9.0 \pm 0.3\,\mathrm{kJ\,mole^{-1}}$ have been estimated for the system. Polanyi potential, ε, is the work to remove a molecule to infinity from its location in the sorption space and is independent of temperature. The D-R isotherm envisages heterogeneity of the sorption energies or sorption space. This isotherm is applicable from trace to saturation values and the sorbate properties do not differ from the corresponding bulk phase except for its lower energy.

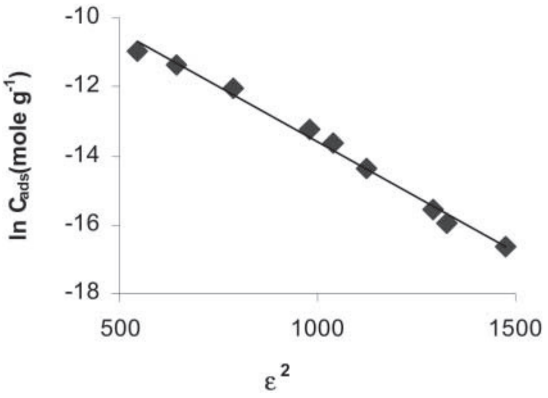

Figure 6. D-R sorption isotherm of Ag(I) ions onto coconut husk.

Thermodynamics of Sorption

The effect of temperature variation on the sorption has also been studied under optimized conditions selected for Ag(I) ions from 5 to 35°C. The sorption has shown an increase with raising temperature. If sorption decreases with increasing temperature it may be indicative of physical sorption, and the reverse is generally true for chemisorption. The thermodynamic parameters ΔH, ΔS, and ΔG can be computed using following relationships:

$$\log K_c = \frac{-\Delta H}{2.303RT} + \frac{\Delta S}{2.303R} \tag{13}$$

$$\Delta G = -RT \ln K_C \tag{14}$$

where $K_C = F/1 - F$, where F is the fraction sorbed at equilibrium; R, is a universal gas constant and T is temperature in Kelvin.

If Equation (13) holds good it yields the value of ΔH and ΔS from the slope and intercept of the linear plot, which is represented in Fig. 7. The values of $\Delta H = 61.3 \pm 5.4\,\mathrm{kJ\,mole^{-1}}$, $\Delta S = 241.9 \pm 18.7\,\mathrm{J\,mole^{-1}\,K^{-1}}$, and $\Delta G = -10.6 \pm 0.09\,\mathrm{kJ\,mole^{-1}}$ at 298 K have been evaluated for the sorption system. Relatively higher value of ΔH may be related to large cavities of sorbent as well as smaller ionic radius of Ag(I) ions in a large pore size exchanger. The positive value of enthalpy and negative value of free energy reflect the endothermic and spontaneous nature of sorption, respectively. A value of $\Delta G = -8.68 \pm 0.09\,\mathrm{kJ}$ at 298 K has been estimated for Ag(I) sorption onto PUF.[22]

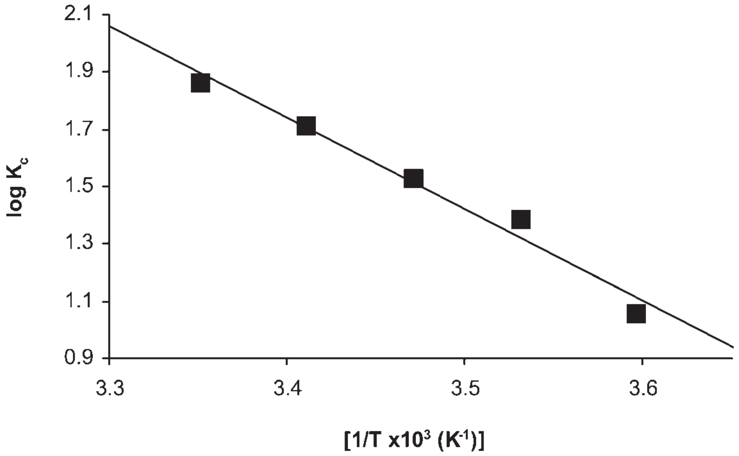

Figure 7. Variation of sorption equilibrium as a function of temperature.

Sensitivity of the Sorbent

Anions, cations, and complexing agents affect the sorption of traces of metal ions. Therefore, effect of common anions and cations on the sorption of silver ions has been monitored. The anions were added as their sodium or potassium salts whereas cations were included as their nitrates. The concentration of these additional ions was kept at least 200 times more than the concentration of Ag(I) ions. The results are recorded in Tables 2 and 3. The distribution ratio, R_d increases in the presence of sulphate and carbonate ions significantly whereas citrate and cyanide ions depress the sorption. Generally, the addition of complexing agents may cause a decrease in the sorption. Our results suggest cosorbed anions may increase the sorption of cations as in the case of sulphate and carbonate. In case of cations Al(III) and Ni(II) register an increase while Ce(III) causes a reduction to some extent in the sorption of Ag(I) ions. Therefore, it is necessary that those ions that reduce the sorption must be absent from the sorptive medium, otherwise low yields would result. Competitive sorption phenomena is of relevance since from a practical point of view several metal ions are expected to be present in real samples of wastewater and industrial effluents.

Selectivity of the Sorbent

The selectivity of the sorbent was tested by measuring the sorption of several metal ions representing different groups and oxidation states of the

Table 2. Influence of anions on the sorption of Ag(I) ions (8.23×10^{-5} M) onto coconut husk 200 mg after 20 min agitation time from 0.1 M HNO_3.

Anions	R_d ($cm^3\,g^{-1}$)	%Sorption
Nil	612	96.5
Sulfate	1,590	98.6
Carbonate	1,113	98.0
Oxalate	975	97.4
EDTA	950	97.7
Tartrate	932	97.5
Nitrate	891	97.3
Chlorate	855	97.4
Thiocyanate	692	96.8
Borate	618	96.5
Acetate	607	96.4
Molybdate	568	96.4
Chromate	348	93.9
Cyanide	264	91.9
Citrate	121	84.3

periodic table under the optimized conditions selected for Ag(I) ions. The findings have been shown in Table 4 in terms of separation factor, distribution ratio, and percent sorption. Cs(I), Zn(II), Cr(III), Co(II), Se(IV), Cd(II), and Tc(VIII) have shown lower sorption affinity for coconut husk surface. These metal ions can be separated from Ag(I) ions using sorbent columns and give separation factors in the order of $\sim 200 > 3000$.

Sorption Mechanism

Ion exchange may be regarded as an important mechanism for Ag(I) ions sorption onto coconut husk. The functional groups[18] like OH^- and fiber carbonaceous C_xOH get dissociated at various pH values depending upon their acidic dissociation constants and consequently taking part in surface complexation/exchange of metal cations

$$OH \rightleftharpoons O^- + H^+$$
$$COOH \rightleftharpoons COO^- + H^+$$
$$C_XOH \rightleftharpoons C_XO^- + H^+$$

Surface complexation is likely to occur during pH variation studies. It is reported that coconut husk contains 38% cellulose, 28% hemicellulose,

Table 3. Effect of cations on the sorption of Ag(I) ions (8.23×10^{-5} M) onto coconut husk (200 mg) after 20 min agitation time from 0.1 M HNO_3.

Cations	R_d ($cm^3\,g^{-1}$)	%Sorption
Nil	612	96.5
Al(III)	1,372	98.4
Ni(II)	1,371	98.4
Ba(II)	1,319	98.3
Sr(II)	1,070	97.9
Mg(II)	1,077	97.9
Co(II)	1,006	97.8
Pb(II)	945	97.7
Fe(III)	808	97.3
Y(III)	568	96.5
Cr(III)	518	95.8
Zn(II)	457	95.3
Hg(II)	296	92.9
K(I)	277	92.5
Ce(III)	179	88.8

and 32.8% lignin.[28] The lignin content may enhance biosorption/bio accumulation of metal ions.[5] Salim and his coworkers[29] attributed the loss of metal ions from aqueous solution in the presence of agricultural materials due to the complexation of constituents of these materials.[29]

Table 4. Selectivity of coconut husk and separation factors (α) of metal ions.

Metal ion	R_d ($cm^3\,g^{-1}$)	%Sorption	$\propto = R_d$ Ag(I)/R_d M
Ag(I)	612	96.5	—
Sb(III)	31	57.8	20
Eu(III)	23	52.3	27
Tm(III)	19	46.4	32
Hg(II)	10	31.4	61
Nd(III)	8	25.4	77
Tc(VII)	3	12.9	204
Cd(II)	3	8.0	204
Se(IV)	2	6.9	306
Co(II)	2	6.3	306
Cr(III)	1	5.0	612
Zn(II)	<1	<3	971
Cs(I)	<1	<1	3,060

The phenolic group in the wood plays an important role through making a chelate complex to metal ions.[30] The fast silver sorption by coconut husk may be ascribed to several hydroxyl and carboxylic groups present in the sorbent matrix.

CONCLUSIONS

The present findings indicate the suitability of economical coconut husk, an agricultural waste, as an effective sorbent for the retention or removal of Ag(I) ions from aqueous solutions especially from wastewater and industrial effluents. The equilibrium isotherm studies reveal that Freundlich and D-R models are most suitable for the present sorption system. The values of sorption capacity and sorption energy have been evaluated along with thermodynamic parameters ΔH, ΔS, and ΔG. The selectivity investigations show that this material can be used to separate Ag(I) ions from Cs(I), Zn(II), Cr(III), Co(II), and Se(IV).

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