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The interaction effects of cypress *(Cupressus sempervirens)*, cinchona *(Eucalyptus longifolia)* and pine *(Pinus halepensis)* leaves on their efficiencies for lead removal from aqueous solutions

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Abstract

Batch and isotherm studies were carried out to compare the effectiveness of decaying leaves of cypress *(Cupressus sempervirens),* cinchona *(Eucalyptus longifolia)* and pine *(Pinus halepensis)* to adsorb lead from its aqueous solution and to study the leaf interaction effects. Lead removal increased with increasing concentrations of both lead ions and the plant leaves employed. Removal efficiency of leaves followed the decreasing order: pine > cypress > cinchona. While cinchona leaves showed an antagonistic effect on the removal efficiencies of cypress, pine, and a combination of cypress and pine leaves, the effect of cypress on the removal efficiency of pine leaves was additive. The adsorption of lead by cypress and cinchona leaves was well defined by both the Freundlich and Langmuir isotherms, but only the Freundlich isotherm was adopted for pine leaves. Desorption of lead from leaves upon standing in deionized water was minimal. © 2002 Elsevier Science Ltd. All rights reserved.

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1. Introduction

Lead and its compounds have often been referred to as common and significant pollutants. Lead is also considered among the most toxic heavy metals due to its bioaccumulation potential and inhibition of the synthesis of hemoglobin (Waldboutt, 1973; Cheremisinoff and Habib, 1982).

Lead can enter and be adsorbed into the human body through inhalation, diet or skin contact, and can produce adverse effects on virtually every system in the body (Zhang et al., 1997; Loghmanadham, 1997). Acute lead poisoning in humans causes severe dysfunction of the kidneys, liver, the central and peripheral nervous system, and the reproductive system, and causes high blood pressure (Casarett and Doulles, 1980; Schwarz, 1977; Mahaffey, 1977; Dinman, 1972; Zielhuis, 1975; Scott, 1977; Tariq and Fatima, 1995; Younas and Shahzad, 1998). Lead is especially harmful to developing brains of fetuses and young children, and may affect children's mental and physical health resulting in learning disabilities, behavioral problems and mental

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retardation. Thus, some countries including the United States have taken measures to reduce lead exposure through comprehensive prevention strategies including environmental standards that removed lead from gasoline, paint, and plumbing. Health-related prevention strategies like screening, physician education and family and community education have also been undertaken (CDC, 1997). Several methods and reagents have been reported for the removal of lead from polluted water. Among these are precipitation, ion exchange, oxidation, cementation, activated carbon, hair, and biomass (Wing and Rayford, 1978; Shambaugh and Melnyk, 1978; Tan et al., 1985; Luo and Hong, 1997; Fane et al., 1992; Grappelli et al., 1992; Hao et al., 1993; Dushenkov et al., 1995; Okieimen et al., 1986; Eromosele and Otitolaye, 1994; Chaudhari and Tare, 1996; Al-Asheh and Duvnjak, 1997; Okieimen et al., 1985; Ho et al., 1995). Decaying plant leaves have also been recommended as a simple inexpensive method to remove heavy metals from solutions (Salim and Robinson, 1985a,b; Salim, 1988; Salim et al., 1992; Sayrafi et al., 1996a,b, 1999; Tee and Khan, 1988).

In a previous work, several factors affecting removal of lead from solutions by cypress leaves were investigated (Salim et al., 1994). The rate and amount of lead removal was highly affected by the acidity of solution and the optimum pH for removal was approximately within neutral pH range. The presence of some competing ions like Ca^{2+}, Zn^{2+} and Ni^{2+} did not affect the rate of lead removal, whereas other ions like Cu^{2+}, Ag^{+} and Cd^{2+} interfered negatively, and ions like Mg^{2+} and Na^{+} showed positive interference. Agitation was found to increase the rate of lead adsorption. The mechanism proposed for lead adsorption on cypress leaves assumed film diffusion as the rate-limiting step.

A literature search indicated that the use of a combination of different plant leaves and their interaction effects to remove lead, or other toxic metals from solutions has not been reported.

The present work was aimed at describing the removal of lead from aqueous solutions using leaves of three different plant species; cypress (*Cupressus sempervirens),* cinchona *(Eucalyptus longifolio)* and pine *(Pinus halepensis)*, and studying the interaction effects of leaves on their efficiencies for lead removal.

2. Materials and methods

2.1. Preparation of lead solutions

Stock solutions of lead were prepared by dissolving 1.60 g $Pb(NO_3)_2$ (99% purity by Merck) in deionized water to a 1-l volume. The required concentrations (500, 750, 1000, 1250, 1500 and 20 000 μg/l) were prepared from the stock solution by dilution.

2.2. Collection of plant leaves

The plant leaves employed were collected from the Jerusalem area. The leaves were cleaned with deionized water and dried in an oven at 80°C for 24 h prior to use.

2.3. Working conditions

Unless otherwise stated, polyethylene containers were used throughout the present work. In order to minimize possible adsorption of lead on the container walls upon introducing the lead solution, the containers were filled with 1.0 M HNO_3 solution for a period of 3 days and then rinsed thoroughly with deionized water prior to use.

To determine the pH of plant leaves suspensions, 12-g/l leaves (one species or more) were soaked in deionized water (250 ml) in a container which was then sealed with parafilm and continuously shaken.

A control, with no leaves, was set to determine the loss, if any, due to adsorption on the container walls.

All experiments were carried out in duplicate at room temperature (22 ± 1°C).

2.4. Lead removal experiments

The desired weight of plant leaves was soaked in a 250-ml solution of the required lead concentration (500, 750, 1000, 1250 and 1500 μg/l). The pH of the resulting suspension was adjusted (throughout the experiment) to a value of 6.5 using dilute NaOH solution, as required. The container was then sealed with parafilm and continuously shaken, to fully expose the leaf surface to the lead solution, until completion of equilibrium was reached (approx. 48 h).

2.5. Effect of plant species on lead removal by 1eaves

To study the effect of plant species on lead removal, 12 g/l leaves (one species or combinations of different species in equal weight ratio) were employed to remove lead from the desired solution (500, 750, 1000, 1250 and 1500 μg/l).

To compare lead removal by the three plant species under assured saturation of adsorption sites on the leaf samples, 13.3-g/l leaves of the desired plant were soaked in a lead solution at initial concentration of 20 mg/l. The suspension was continuously shaken at constant pH of 6.5. The lead that remained in solution was monitored with time (until no change in lead concentration was observed) using a flame atomic absorption spectrometer (Video II aa/ae spectrometer/Instrumentation Laboratory).

The effect of leaf concentration of a certain plant species on the removal of lead (1000 μg/l) was studied by employing 8-, 12-, 16- and 24-g/l leaves.

2.6. Desorption of lead from leaves

To study the desorption of lead from leaves that had been employed for lead removal, the leaves were separated from lead solution by decantation, rinsed with deionized water and then soaked in deionized water using a new container. The container was then sealed with parafilm and shaken continuously for a period of 48 h before the solution was analyzed for desorbed lead.

2.7. Instrumentation

Unless otherwise stated, all samples were analyzed for lead by Anodic Stripping Voltammetry (AVS) using a Polarographic Analyzer/ Stripping Voltammeter Model 264 B, EG & G Instrument. An aliquot of 0.5 ml of the sample solution was added to a volumetric cell that contained 5 ml of 0.2 M HCl. After accumulation of the metal on the HMDE, at a potential of −0.75 V, three measurements were carried out for each sample. The detailed working conditions are shown in Table 1.

The solution pH values were measured using a Corning pH-meter Model 12 that had been calibrated by means of standard buffers prior to each use.

The experimental error in each reading was estimated at 5–7%.

3. Results and discussion

Table 2 gives the pH of the employed plant leaves and their different combinations in deionized water. The most acidic leaves are those of cinchona (pH 4.80) followed by pine (pH 6.00), and cypress was the least (pH 6.34). Cypress–cinchona and cypress–cinchona–pine combinations showed pH readings higher than the average pH of their separate components. However, the pH of the pine–cinchona combination was almost the same as the average pH of the two constituents. Thus, one might conclude that cypress leaves have the highest buffering capacity among the three species of leaves employed.

Fig. 1 shows that the fraction of lead removed by a certain concentration of plant leaves increased with increasing initial concentration of lead in solution.

Table 1
The working conditions used for anodic stripping voltammetry (AVS)

Experimental parameters for low and moderate conc.	
Working electrode	Mercury drop electrode
Drop size	Middle
Reference electrode	Ag/AgCl electrode
Auxiliary electrode	Pt wire
Initial potential	−0.75 V
Final potential	−0.40 V
Deposition potential	−0.75 V
Integration set point	−0.53 V
Purge time	30 s
Deposition time	60 s[a]
Equilibrium time	15 s
Scan rate	10 mV/s
Current	1 μA
Pulse amplitude	25 mV
Recorder *x*-axes scale	500 mV/cm[b]
Recorder *y*-axes scale	250 mV/cm
Supporting electrolyte	0.5 M HCl

[a] Deposition time for high concentration was 40 s.
[b] Recorder *x*-scale for the high concentration was 100 mV/cm.

It is also noticed that increasing the concentration of leaves in a given aqueous solution of lead increased the fraction of lead that could be removed by leaves employed for treatment (Fig. 2).

Among the three plant species employed, pine leaves were the most efficient to remove lead from its solution and cinchona was the least. This could be due, in part, to the individual leaf shape and whether the leaf surface is smooth or rough, as both factors are related directly to the exposed leaf area. Another factor could be the buffering capacity of the leaf suspension, as discussed above.

Results of lead removal from a relatively highly concentrated lead solution (20 mg/l) by the leaves of the three plant species (Table 3) indicates that the above-mentioned order of lead removal efficiency holds even at saturation of adsorption sites on the leaf sam-

Table 2
pH of plant leaves in deionized water after 48-h contact

Plant leaves (g/l)	pH ± 0.01
–	5.78
Cypress (12)	6.34
Cinchona (12)	4.80
Pine (12)	6.00
Cypress (6) + cinchona(6)	5.97
Cypress (6) + pine (6)	6.15
Pine (6) + cinchona(6)	5.46
Cypress (4) + cinchona (4) + pine (4)	6.00

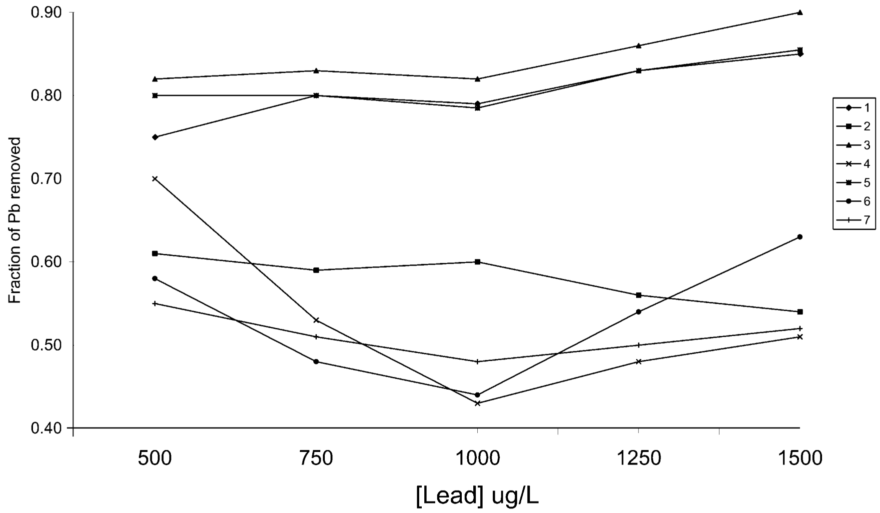

Fig. 1. Effect of initial lead concentration (μg/L) on its removal by plant leaves (g/L): (1) cypress (12); (2) cinchona (12); (3) pine (12); (4) cypress (6) + cinchona (6); (5) cypress (6) + pine (6); (6) pine (6) + cinchona (6); and (7) cypress (4) + cinchona (4) + pine (4).

ples. This order was obvious from the fraction of lead removed from solution after equilibrium had been accomplished (pine 0.76, cypress 0.58, and cinchona 0.38). For almost all concentrations of lead employed in this study, a synergistic (additive) interaction effect was observed between the lead removal efficiency of cypress leaves and that of pine leaves. However, when cinchona leaves were added to a leaf suspension of cypress, pine, or the cypress–pine combination, the lead removal efficiency of the resulting leaf mixture was lower than that of cypress, pine or a combination of cypress and pine, respectively. Thus, the interaction effect between the lead removal efficiency of cinchona leaves and that of the other plant species was antagonistic.

Freundlich and Langmuir isotherms are the most widely applied adsorption isotherms. The Freundlich, though empirical and its application is limited to solutions of moderate concentration, has been found more appropriate to several adsorption processes where non-uniformity of actual surfaces is expected. It can be written as:

$$X/M = kC_e^{1/n}$$

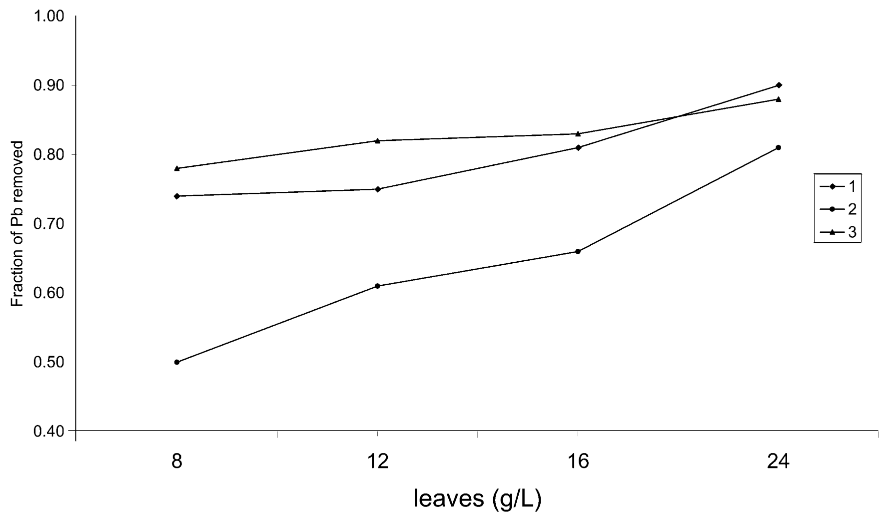

Fig. 2. Effect of concentration of Leaves (g/L) on lead (1000 μg/L) removal: (1) cypress; (2) cinchona; and (3) pine.

Table 3
Effect of different plant leaves (13.3 g/l) on the concentration of lead ions (20 mg/l) at pH = 6.5

Plant leaves					
Cypress		Cinchona		Pine	
t (h)	Lead (mg/l)	t (h)	Lead (mg/l)	t (h)	Lead (mg/l)
0.50	19.8	0.50	20.0	0.50	18.0
1.00	19.6	1.00	20.0	1.00	16.2
1.50	19.4	1.50	20.0	1.50	15.0
2.00	19.0	2.00	20.0	2.00	14.0
2.50	18.8	4.00	20.0	3.00	13.0
3.00	18.5	8.00	20.0	4.00	12.4
4.00	18.0	12.00	20.0	6.00	11.6
5.00	17.6	14.00	19.9	8.00	10.8
6.00	17.1	16.00	19.8	10.00	10.2
8.00	16.3	18.00	19.7	12.00	9.6
10.00	15.4	20.00	19.6	14.00	9.0
12.00	14.8	22.00	19.5	16.00	8.4
14.00	14.0	24.00	19.2	20.00	7.4
16.00	13.4	26.00	16.0	24.00	6.6
20.00	12.0	28.00	13.0	28.00	6.0
24.00	11.0	30.00	12.0	32.00	5.6
32.00	9.7	32.00	12.6	36.00	5.4
40.00	8.8	34.00	12.6	40.00	5.0
48.00	8.4	36.00	12.6	44.00	4.9
52.00	8.3	40.00	12.6	48.00	4.8
56.00	8.3	52.00	12.4	52.00	4.8
60.00	8.3	60.00	12.4	60.00	4.7

where: X/M is the amount of solute adsorbed per unit weight of the adsorbent (μg/g); C_e is the equilibrium concentration of the solute in the bulk solution (μg/l); k is a constant indicative of the adsorption capacity of the adsorbent (l/μg); and n is a constant indicative of the adsorption intensity of the adsorbent.

The Langmuir isotherm, though commonly applied to monolayer chemisorption of gases, is generally applied when no strong adsorption is expected and the adsorption sites are uniform. The following form of the Langmuir isotherm is to be appropriate to adsorption from solution:

$$X/M = abC_e/(1 + aC_e)$$

where X/M and C_e are defined as before for the Freundlich isotherm, a is a constant indicative of the binding energy between the adsorbed species and the adsorbent (l/μg), and b is a constant indicative of the mass of adsorbed solute required to completely saturate a unit mass of a adsorbent and thus, indicative of the monolayer coverage.

The linear regression method was used to determine the Freundlich and Langmuir constants (Figs. 3 and 4, respectively). The applied isotherm equations and the calculated constants for the plant leaves employed are given in Tables 4 and 5.

Adsorption of lead ions from aqueous solution by all species of plant leaves employed is well described by either isotherm with cypress showing the best correlation coefficients (Table 4).

Although applying the Langmuir isotherm to pine leaves resulted in a high correlation coefficient (0.9), the equation was rejected due to the negative values obtained for the constants a and b. The two constants are indicative of the binding energy and the monolayer

Table 4
The Freundlich and Langmuir isotherm equations for lead adsorption by plant leaves from aqueous solutions

Isotherm	Plant leaves	Equation	Correlation coefficient
Freundlich	Cypress	$\text{Log } X/M = 0.9734 \log C_e - 0.3805$	0.97
	Cinchona	$\text{Log } X/M = 0.6054 \log C_e + 0.1295$	0.91
	Pine	$\text{Log } X/M = 1.5772 \log C_e - 1.7194$	0.89
Langmuir	Cypress	$1/(X/M) = 2.6332\ 1/C_e + 0.0007$	0.97
	Cinchona	$1/(X/M) = 3.7945\ 1/C_e + 0.0104$	0.89
	Pine	$1/(X/M) = 4.4068\ 1/C_e - 0.0096$	0.90

Table 5
The Freundlich and Langmuir constants for adsorption of lead by plant leaves

Plant leaves	Freundlich constants		Langmuir constants	
	k	n	a	b
Cypress	0.416	1.027	2.658×10^{-4}	1.428×10^{3}
Cinchona	1.347	1.652	2.741×10^{-3}	96.154
Pine	0.019	0.634	-2.178×10^{-3}	−104.167

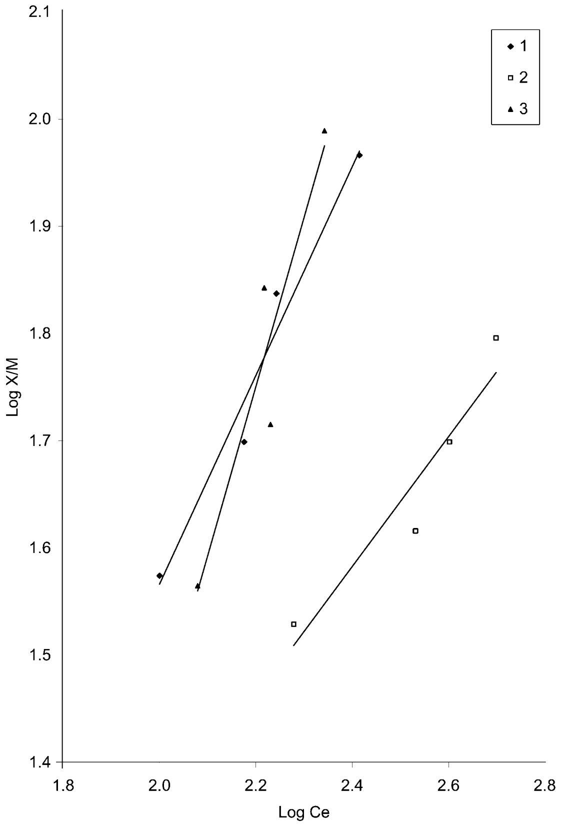

Fig. 3. Linear Freundlich isotherm: (1) cypress; (2) cinchana; and (3) pine.

coverage, respectively. This rejection is confirmed by analyzing the Langmuir constants by means of the separation factor (R), which is defined as (Weber and Chakravorti, 1974; Hall et al., 1966):

$$R = 1/(1 + aC_o)$$

where R is the dimensionless constant separation factor, a is the Langmuir constant (l/μg), and C_o is the initial solute concentration (μg/l).

The type of isotherm is obtained from the R-value as shown in Table 6. Table 7 shows the calculated R-values for the Langmuir isotherm applied to leaves of the three species of plants. The calculated values of R were in the favorable range for leaves of cypress (0.79) and cinchona (0.267), but irreversible for pine leaves (-0.849). The irreversibility of the Langmuir isotherm in the case of pine leaves agrees with the assumption that this isotherm does not fit to cases of strong adsorption (Moore, 1974). The observed low desorption of lead ions from leaves into deionized water ($< 3\%$) indicates good retention capacity of leaves for lead ions. This agrees with an earlier report (Salim et al., 1994), that partial stripping of lead ions from cypress leaves occurs only at a pH that is highly acidic (< 1.5) or highly alkaline (> 11).

4. Conclusions

The following conclusions could be drawn from the results of the present study:

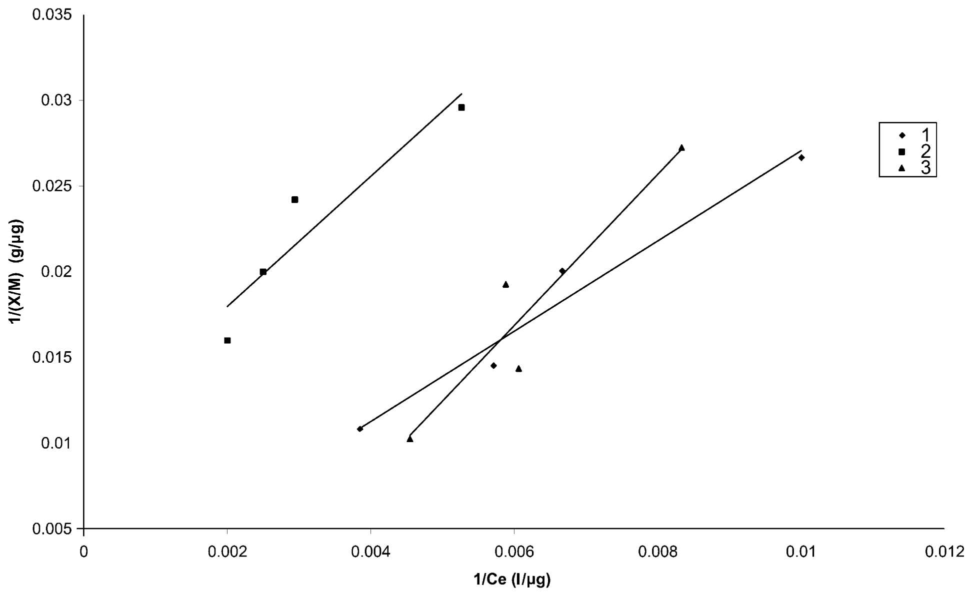

Fig. 4. Linear Langmuir isotherm: (1) cypress; (2) cinchona; and (3) pine.

Table 6
Types of isotherms for different values of R

Value of R	Type of isotherm
$R > 1$	Unfavorable
$R = 0$	Linear
$0 < R < 1$	Favorable
$R < 0$	Irreversible

Table 7
Separation factor R, for the adsorption of lead by plant leaves

Plant leaves	C_0 (μg/l)	Langmuir constant (l/μg)	R
Cypress	1000	2.658×10^{-4}	0.790
Cinchona	1000	2.741×10^{-3}	0.267
Pine	1000	-2.178×10^{-3}	-0.849

1. Lead removal from aqueous solutions by plant leaves increases with increasing initial concentration of lead in solution and with increasing concentration of leaves employed.
2. For the plant leaves employed, the removal efficiency followed the decreasing order: pine > cypress > cinchona.
3. Cinchona shows an antagonistic effect on lead removal efficiency of cypress, pine and their combination, while the effect of cypress on pine leaves efficiency is additive.
4. The adsorption of lead by cypress and cinchona leaves is well defined by either the Freundlich or Langmuir isotherm. However, the Freundlich isotherm is reasonable to define the adsorption of lead by pine leaves.
5. Cypress leaves show the highest buffering capacity among the three species of plant leaves employed.

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