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RECOVERY OF VALUABLE PERFUMERY COMPOUNDS FROM A GERANIUM STEAM DISTILLATION CONDENSATE USING POLYMERIC ADSORBENTS

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ABSTRACT

Geranium steam-distillation condensate water contains a number of valuable perfumery components. This water was characterized and found to contain geraniol, nerol, citronellol, linalool, isomenthone and menthone. Recovery of these components from the condensate water by adsorption on polymeric adsorbents is described. Single solute and multisolute adsorption equilibria for a variety of polymeric adsorbents are reported. A rational explanation is given for the difference in single solute and multisolute equilibria. A column breakthrough study showed that the components can be readily recovered by adsorption.

Key Words: Geranium; Condensate; Water; Polymeric adsorbents; Recovery

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INTRODUCTION

Essential oils obtained from natural sources (flowers, leaves, etc.) are important raw materials for the perfume and flavor industry. The most commonly used processes for essential oil production are steam distillation and solvent extraction (1). During steam distillation, substantial amounts of water-soluble compounds (alcohols, ketones, aldehydes, etc.) present in the essential oil are dissolved in the steam distillation condensate water due to their finite solubilities in water (2–4). The recovery of these compounds becomes very important due to environmental or economic reasons (2,5). Furthermore, the water-soluble compounds are the major aroma-imparting compounds. Thus, if these compounds are recovered and blended with the oil phase, the resulting oil will be similar to the natural oil in its aroma/flavor.

Recovery of these valuable compounds can be completed either by solvent extraction or adsorption. The choice of recovery depends on the concentration of the compounds present in the condensate water. Adsorption is the mostly widely used process for condensates with concentrations less than 2000 ppm due to low solubilities (2).

In recent years, polymeric adsorbents have played a very important role in recovery and removal of dissolved organic compounds from aqueous solution (6–9). Although a great deal of literature exists for activated carbon systems used as adsorbents, the efficiency of polymeric adsorbents, due to their wide range of properties, such as surface area, porosity, and polarity, allows them to replace activated carbon systems for liquid phase adsorption. The adsorption process on polymeric adsorbents is physical, the result of weak van der Waal's forces, which allow the adsorbents to be easily regenerated with a suitable eluant (10). However, regeneration of activated carbon requires more severe conditions than it does for the polymeric adsorbents (11). In addition, at relatively high concentrations, polymeric adsorbents have higher capacities than do the activated carbons (7).

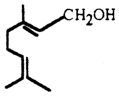
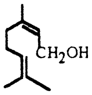
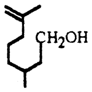
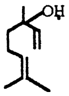
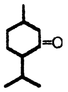
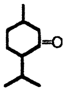
Our study deals with the recovery of dissolved organic (perfumery) compounds from the steam distillation condensate of geranium (*Pelargonium graveolens*) essential oil by adsorption. The adsorption was carried out with polymeric adsorbents (Amberlite XAD-2, Amberlite XAD-4, Amberlite XAD-7, and INDION 1014 MN-2). The geranium-condensate water was obtained from a steam distillation unit in South India. Geranium oil is produced by the steam distillation of the leaves and stalks of *Pelargonium* species. The oil is an amber to greenish-yellow liquid that has a characteristic rose-like odor, which is highly valued. The Bourbon and North African varieties are the 2 main types of oil. The Bourbon quality is more valuable and thus more expensive. Annual worldwide production of Geranium oil is about 500 tonnes. Geranium oil is the most important natural raw material in the perfumery industry (12). The compounds cited most in the literature about geranium oils (from different cultivars) are citronellol, geraniol, linalool, phenyl ethyl alcohol, rhodinol, eugenol, and isomenthone (1). Our work involved the characterization of the geranium- condensate water and equilibrium adsorption studies for single and multisolute systems. Column breakthrough data were obtained with 1 polymeric adsorbent due to a limited supply of the condensate water.

EXPERIMENTAL

Materials

The aqueous solutions for the single-solute adsorption studies were prepared with pure solute samples. The physical properties of the solutes are given in Table 1. All the aqueous solutions were prepared in distilled water. The polymeric adsorbents, Amberlite XAD-2, Amberlite XAD-4, and Amberlite XAD-7 were supplied by the Rohm and Haas Co (France), while INDION 1014 MN-2 was sup-

Table 1. Properties of Solutes

Solute	Structural Formula	Molecular Formula	Density (g/cm^3) (25°C)	Boiling Point (°C)	Aroma Profile
Geraniol		$C_{10}H_{18}O$	0.89	230	Mild and sweet, floral rose-type odor.
Nerol		$C_{10}H_{18}O$	0.88	227	Sweet rosy, refreshing and wet seashore odor of moderate tenacity.
Citronellol		$C_{10}H_{20}O$	0.86	225	Fresh rosy odor, variable according to purity.
Linalool		$C_{10}H_{18}O$	0.86	198	Light and refreshing, floral woody odor with a faint citrus note.
Isomenthone		$C_{10}H_{18}O$	0.9	208	Powerful, refreshing and clean minty odor of moderate tenacity.
Menthone		$C_{10}H_{18}O$	0.9	204	Minty, refreshing and diffusive odor.

Data from (17).

Table 2. Properties of Adsorbents

Adsorbent	Total Area (m^2/g)	Pore Volume (cm^3/g)	Particle Size (mm)	Density (g/cm^3)	Polarity	Polymer Matrix
Amberlite XAD-2	300[a]	0.69	0.4	1.02	Nonpolar	Polystyrene-divinylbenzene
Amberlite XAD-4	750[a]	0.99	0.5–0.7	1.03–1.04	Nonpolar	Polystyrene-divinylbenzene
Amberlite XAD-7	450[a]	1.08	0.45–0.6	1.06–1.08	Intermediate	Acrylic ester
INDION 1014 MN-2	1000[b]	1.08	0.3–1.2	1.04	Nonpolar	Polystyrene-divinylbenzene

[a] Data from (18).
[b] Data from (19).

plied by Ion Exchange Ltd (India). The physical properties of the adsorbents are given in Table 2. The geranium condensate water was supplied by a steam distillation unit in South India. Acetone (Extra Pure) was supplied by S. D. Fine Chem. Ltd, which was used for washing the adsorbent. The ethanol used for regeneration of loaded adsorbent was freshly distilled.

Adsorbent Preparation

Each adsorbent was first washed with 10 bed volumes of acetone and then dried at 60°C at 135 Pa absolute pressure. This process removes any kind of impurities present in the adsorbent. The vacuum-dried adsorbents were kept in sealed flasks to prevent ingress of moisture.

Equilibrium Adsorption Studies

Aqueous solutions of 1000 ppm concentration of all the solutes were prepared with distilled water. As the maximum solubilities of the solutes were 1000 ppm, higher concentration solutions could not be prepared. These solutions were used for single solute adsorption studies. Adsorbents (0.05 g, 0.1 g, 0.2 g, and 0.4g) were added to 50 mL aqueous solutions in conical flasks. These flasks were sealed and kept 25°C until equilibrium was attained. The aqueous solutions were then analyzed for their equilibrium concentrations.

The multisolute equilibrium adsorption studies were conducted through the same procedure as that for single solute adsorption. The geranium steam-distillation condensate water was used in the mixture.

Column Exhaustion Studies

A glass column, 1500 mm long and 14 mm i.d., was used for the exhaustion runs. The column was packed up to 70 mm with a slurry of the adsorbent with water. Acetone, followed by distilled water, was passed through the column. The geranium condensate water was then passed in down-flow mode through the column. The flow rate for the breakthrough runs was adjusted to 10 mL/min with a Millipore peristaltic pump. Samples were collected periodically and analyzed. The flow rate was adjusted to 2 mL/min for regeneration runs with an ethanol eluant. Samples were collected and analyzed.

ANALYSIS

Gas Chromatography

The analysis of all the samples was done through gas chromatography (GC) with a Chemito 8510 model GC. The glass GC column was 2 m long and 3 mm i.d., and it was packed with Carbowax 20M supported on 80/100 mesh Chromosorb T. The injector and detector were each kept at 235°C. The oven temperature was adjusted to 125°C and the nitrogen flow rate was 50 mL/min. Three microliters of sample injected into the GC.

Gas Chromatography–Mass Spectroscopy

The details of the gas chromatography–mass spectroscopy (GC-MS) study are as follows:

Gas chromatograph: Hewlett-Packard HP-5890 Series II Plus
Mass selective detector: Hewlett-Packard MSD 5972
Capillary column: Alltech AT-1000, length = 60 m, i.d. = 0.25 mm
Injector temperature: 250°C
Oven Programming: Initial Temperature = 40°C; rate = 4°C/min; oven temperature = 210°C; and
and oven time = 15 minutes
Carrier gas: Helium
Carrier gas flow rate: 0.5 mL/min
Split ratio: 1:250

The loading capacity, c (mmol/g) was determined from a solute mass balance equation (5,13). Knowing the initial and final bulk concentrations,

$$c = \frac{(C_o - C_e)\cdot V}{m} \tag{1}$$

where C_o is the initial concentration of solute (mmol/L); C_e is the final equilibrium concentration of solute (mmol/L); V is the volume of the solution (L), and m is the mass of the adsorbent (g).

The equilibrium adsorption data for single- and multisolute adsorption were fitted to the Freundlich and Langmuir isotherms (14–15). The Freundlich isotherm for single solute adsorption is

$$c = m_F C_e^{1/n} \tag{2}$$

where c is the loading capacity (mmol/g); C_e is the final equilibrium concentration of solute (mmol/L); and m_F and n are constants. The higher the value of m_F and n, the better is the adsorption capacity of the adsorbent.
The Langmuir isotherm for single solute adsorption is,

$$\frac{c}{c^*} = \frac{KC_e}{1 + KC_e} \tag{3}$$

where c is the loading capacity (mmol/g); c^* is the maximum loading capacity (mmol/g); K is the adsorption equilibrium constant (l/mmol); and C_e is the final equilibrium concentration of solute in the liquid phase.

RESULTS AND DISCUSSIONS

Characterization of Geranium Condensate Water

The characterization of the geranium condensate water was done through G C. The compounds detected in the geranium condensate water were geraniol (60 ppm), nerol (50 ppm), citronellol (100 ppm), linalool (12 ppm), isomenthone (70 ppm), and menthone (40 ppm). These results were confirmed by GC-MS analysis. However, the analysis of the samples obtained after column elution with ethanol indicated the presence of rose oxide, α-terpineol and menthol. Apparently these compounds were present in extremely low concentrations (probably below the detection threshold) in the original geranium condensate water. However, once they were concentrated on the adsorbent and eluted, the concentrations were much higher and thus detection was possible. Rose oxide is a highly valuable compound, which can impart a sweet fruity odor at extremely low concentrations.

EQUILIBRIUM ADSORPTION STUDIES

Single Solute Adsorption

The sorption isotherms were plotted in the conventional manner of loading an adsorbent based on the concentration of the solute in the aqueous phase. Typi-

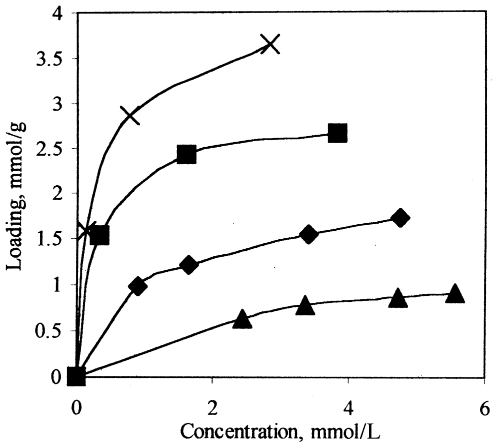


Figure 1. Single-solute adsorption isotherms (loading versus concentration) for geraniol on polymeric adsorbents at 25°C. ◆ = Amberlite XAD-2; ■ = Amberlite XAD-4; ▲ = Amberlite XAD-7; × = INDION 1014 MN-2.

cal isotherms for 2 solutes, geraniol and isomenthone, are shown in Figs. 1 and 2, respectively. Alcohols and ketones were also present in the aqueous phase. The isotherms shown in the figure are either of Type I or Type II, depending upon the adsorbent (14). Isotherms for Amberlite XAD-7 were of Type I (geraniol) or Type III, indicating that it was unfavorable. This result is due to the relatively polar surface of Amberlite XAD-7. The equilibrium adsorption isotherms for the single-solute adsorption data were fitted based on the Freundlich and Langmuir models

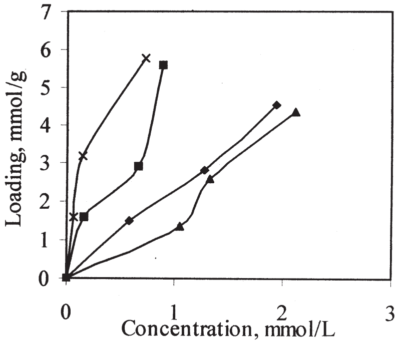


Figure 2. Single-solute adsorption isotherms (loading versus concentration) for isomenthone on polymeric adsorbents at 25°C. ◆ = Amberlite XAD-2; ■ = Amberlite XAD-4; ▲ = Amberlite XAD-7; × = INDION 1014 MN-2.

Table 3. Parameters for Freundlich Isotherm (Single Solute Adsorption)

	Adsorbent											
	XAD-2			XAD-4			XAD-7			INDION 1014 MN-2		
Solute	n	m_F	R^2	n	m_F	R^2	n	m_F	R^2	n	m_F	R^2
Geraniol	2.99	1.03	0.99	3.20	1.94	0.95	2.24	0.69	0.97	2.93	2.83	0.97
Nerol	1.29	1.25	0.95	2.28	1.74	0.98	1.47	0.47	0.74	4.33	2.84	0.93
Citronellol	1.47	2.82	0.95	1.3	3.83	0.99	0.56	1.36	0.85	1.95	5.9	0.97
Linalool	1.32	1.77	0.92	1.83	3.12	0.95	0.59	1.1	0.96	2.99	3.99	0.95
Isomenthone	1.31	2.5	0.98	1.91	4.54	0.93	0.73	1.55	0.96	1.88	7.35	0.98
Menthone	1.27	3.24	0.99	1.84	5.77	0.93	0.4	2.08	0.95	2.2	10.86	0.94

R^2 = correlation coefficient.

(14). The values for the Freundlich and Langmuir constants are given in Tables 3 and 4. The values the Freundlich isotherm exponents show that the INDION 1014 MN-2 has the best adsorption capacity for all the solutes. The capacity decreased in the order of decreased adsorbent surface area; i.e., INDION 1014 MN-2 > Amberlite XAD-4 > Amberlite XAD-2 > Amberlite XAD-7. Although the surface area of Amberlite XAD-7 is greater than that of Amberlite XAD-2 the adsorption capacity is less for Amberlite XAD-7 because of its intermediate surface polarity. The values of the equilibrium adsorption constants for the Langmuir isotherm were also the highest for INDION 1014 MN-2 for all the solutes, indicating that this compound has the best adsorption capacity. The order of adsorbent capacity

Table 4. Parameters for Langmuir Isotherm (Single Solute Adsorption)

	Adsorbent											
	XAD-2			XAD-4			XAD-7			INDION 1014 MN-2		
Solute	K	X^*	R^2	K	X^*	R^2	K	X^*	R^2	K	X^*	R^2
Geraniol	1.85	1.77	0.97	5.67	2.58	0.99	0.38	1.36	0.99	10.34	3.14	0.98
Nerol	0.41	4.6	0.96	2.41	2.76	0.96	0.28	2.17	0.8	36.01	3.12	0.99
Citronellol	1.75	4.13	0.96	0.61	10.34	0.96	—	—	—	2.2	11.49	0.98
Linalool	0.73	4.29	0.93	4.56	3.31	0.93	—	—	—	45	3.13	0.91
Isomenthone	0.78	5.62	0.97	9.24	3.49	0.93	—	—	—	15.37	4.14	0.96
Menthone	0.87	6.52	0.98	11.08	3.77	0.94	—	—	—	26	6.75	0.93

R^2 = correlation coefficient.

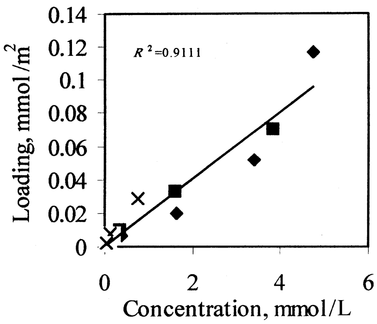


Figure 3. Single-solute adsorption isotherms (surface concentration versus concentration) for geraniol on polystyrene-divinylbenzene adsorbents at 25°C. ◆ = Amberlite XAD-2; ■ = Amberlite XAD-4; × = INDION 1014 MN-2.

was as follows: INDION 1014 MN-2 > Amberlite XAD-4 > Amberlite XAD-2 > Amberlite XAD-7.

The single-solute adsorption isotherms were also plotted on the basis of surface concentration (mmol/m^2) versus concentration of solute in the aqueous phase (7,16). The plots for 2 solutes, geraniol and isomenthone, are shown in Figs. 3 and 4, respectively, for the polystyrene-divinylbenzene adsorbents INDION 1014 MN-2, Amberlite XAD-4, and Amberlite XAD-2. According to the plots a good correlation exists between surface and solute concentrations, and data for all the adsorbents can be represented by a single line when plotted in terms of surface concentration. Some scatter is seen with regard to ketone plots but a trend of correlation with surface area is evident (Fig. 4). These plots indicate that the adsorption capacity is related to the adsorbent surface area.

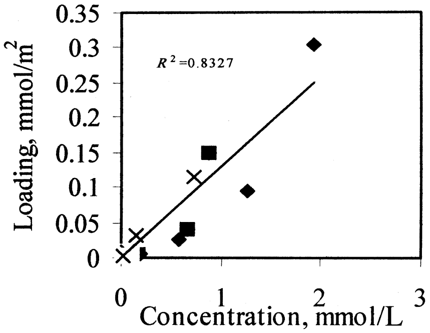


Figure 4. Single-solute adsorption isotherms (surface concentration versus concentration) for isomenthone on polystyrene-divinylbenzene adsorbents at 25°C. ◆ = Amberlite XAD-2; ■ = Amberlite XAD-4; × = INDION 1014 MN-2.

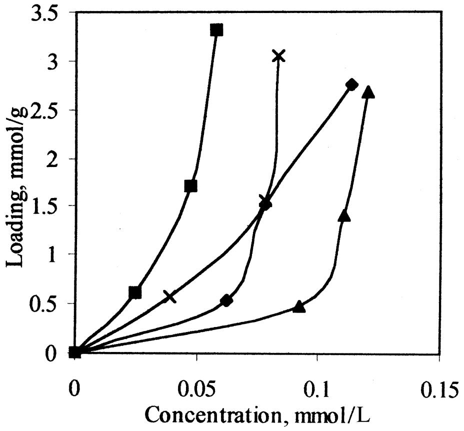


Figure 5. Multisolute adsorption isotherms (loading versus concentration) for geraniol on polymeric adsorbents at 25°C. ◆ = Amberlite XAD-2; ■ = Amberlite XAD-4; ▲ = Amberlite XAD-7; × = INDION 1014 MN-2.

Multisolute Adsorption

The sorption isotherms of multisolutes were totally different from single solute adsorption isotherms for all the adsorbents. The multisolute isotherms were of the Type III and unfavorable (14). The isotherms for multisolute adsorption of geraniol and isomenthone are shown in Figs. 5 and 6. An attempt was made to fit

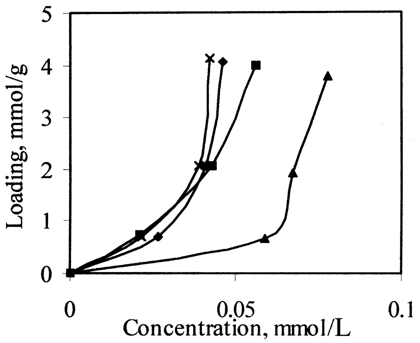


Figure 6. Multisolute adsorption isotherms (loading versus concentration) for isomenthone on polymeric adsorbents at 25°C. ◆ = Amberlite XAD-2; ■ = Amberlite XAD-4; ▲ = Amberlite XAD-7; × = INDION 1014 MN-2.

Table 5. Experimental and Calculated Loading Values for Citronellol onto Polystyrene-Divinylbenzene Adsorbents (Multisolute Adsorption)

XAD-2		XAD-4		INDION 1014 MN-2	
Experimental	Calculated	Experimental	Calculated	Experimental	Calculated
4.8	0.71	5.46	0.19	4.79	0.52
2.51	0.67	2.82	0.18	2.53	0.49
0.91	0.48	1.01	0.11	0.95	0.43
0.58	0.33	0.61	0.11	0.53	0.44

Loading values were calculated through the use of the multisolute Langmuir equation (14).

the multisolute adsorption data to the multisolute Langmuir isotherm (14). The multisolute Langmuir equation yielded poor correlation with the data. The loading values obtained experimentally and by the multisolute Langmuir equation for citronellol are shown in Table 5. The experimental values were consistently higher than the predicted values. This difference can be explained on the basis of interaction between adsorbed solutes. All the solutes in the mixture have functional groups capable of hydrogen bonding (Table 1). Thus, an adsorbed solute can hydrogen bond with another solute in the bulk liquid and hold it. Although the hydrogen-bonded solute is not directly attached to the adsorption surface, it is evidently removed from the bulk liquid and is counted as adsorbed, increasing the experimental values of amount adsorbed. The overall value of adsorbed concentration of any solute from the mixture was always lower than that for the single-solute experiment. This result is likely due to competitive adsorption and also to low solute concentrations in the mixture. A surface site occupied by one solute is not available to other solutes. Thus, the overall surface availability for any given solute is reduced in the case of adsorption from a mixture. The extent of adsorption is approximately proportional to the surface area available. The positive effect of hydrogen bonding is still not sufficient to raise the amount adsorbed to the level of single solute adsorption. Thus, the unavailable area (competitive adsorption) has a dominant effect.

Therefore, in the case of multisolute adsorption, the extent of adsorption decreases. All the isotherms for multisolute adsorption are of Type III and unfavorable.

The isotherms of surface concentration versus concentration of solute in the aqueous phase in multisolute adsorption cases did not give a favorable fit to the linear model, indicating that the adsorption process was totally different in the mixture of solutes due to competitive adsorption. The plots for surface concentration are shown in Figs. 7 and 8 for geraniol and isomenthone.

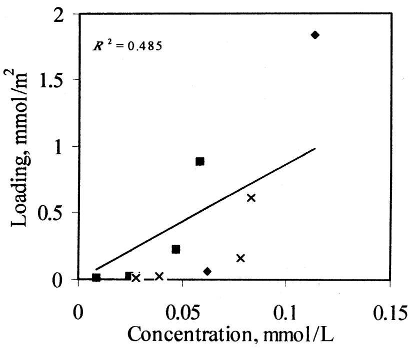


Figure 7. Multisolute adsorption isotherms (surface concentration versus solute concentration) for geraniol on polystyrene-divinylbenzene adsorbents at 25°C. ◆ = Amberlite XAD-2; ■ = Amberlite XAD-4; × = INDION 1014 MN-2.

COLUMN EXHAUSTION STUDIES

Breakthrough

The results of the multisolute breakthrough studies are shown for geraniol and isomenthone in Figs. 9 and 10. The plots show a smooth S-shaped breakthrough curve for both the solutes. The capacities at breakpoint and saturation point are given in the Table 6. Normally, fixed-bed adsorption is used in at least a 2 bed in series configuration with a third regeneration column. In such a mode, the first column can be used to exhaustion because the second column is still fresh and capable of trapping the solute leaking from the first column. The column exhaus-

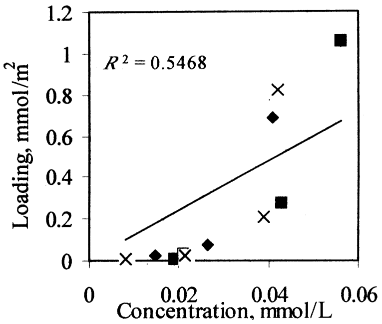


Figure 8. Multisolute adsorption isotherms (surface concentration versus concentration) for isomenthone on polystyrene-divinylbenzene adsorbents at 25°C. ◆ = Amberlite XAD-2; ■ = Amberlite XAD-4; × = INDION 1014 MN-2.

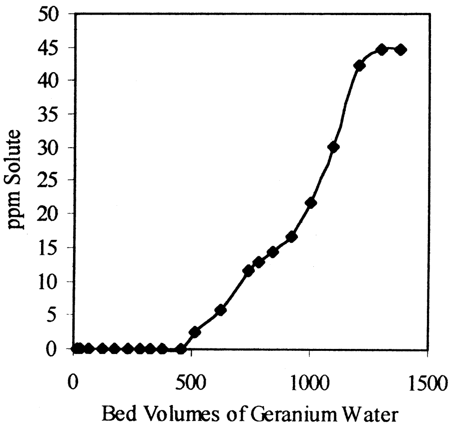


Figure 9. Breakthrough curve for geraniol adsorption on INDION 1014 MN-2 at 25°C; concentration = 60 ppm; mass of adsorbent = 10.5 g.

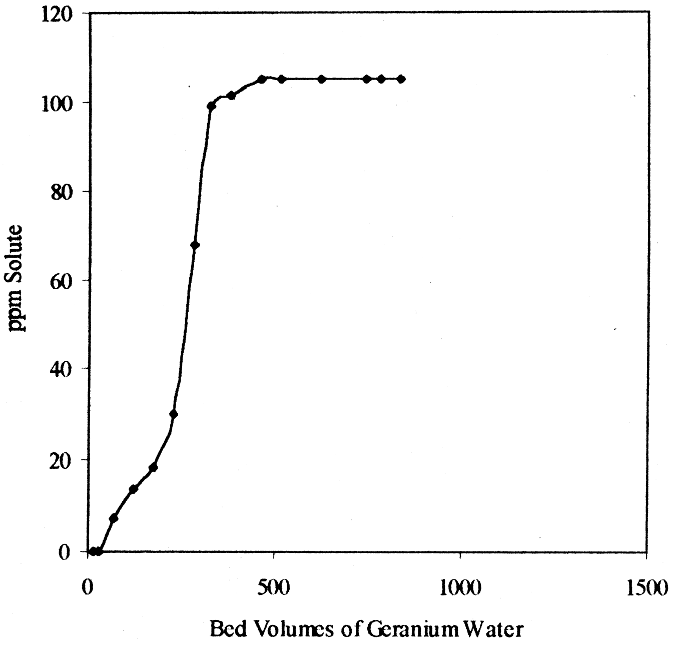


Figure 10. Breakthrough curve for isomenthone adsorption on INDION 1014 MN-2 at 25°C; concentration = 70 ppm; mass of adsorbent = 10.5 g.

Table 6. Capacity at Break Point and Saturation Point for Adsorption

Solute	Capacity at Break Point (g/g of resin)	Capacity at Saturation Point (g/g of resin)
Geraniol	0.022	0.040
Nerol	0.014	0.019
Citronellol	0.024	0.054
Linalool	0.0007	0.005
Isomenthone	0.006	0.024
Menthone	0.003	0.016

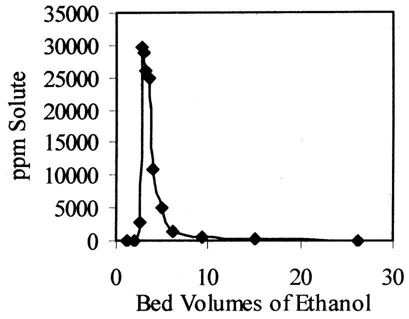


Figure 11. Regeneration curve for geraniol on INDION 1014 MN-2 at 25°C.

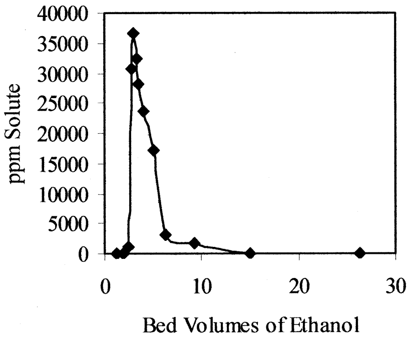


Figure 12. Regeneration curve for isomenthone on INDION 1014 MN-2 at 25°C.

Table 7. Values of Initial Load, Rest Load, and Mass of Adsorbent Used for Desorption

Solute	Initial Load, g	Rest Load, g	Mass of Adsorbent, g
Geraniol	0.45	0.0	11.20
Nerol	0.21	0.0	11.20
Citronellol	0.61	0.0	11.20
Linalool	0.05	0.0	11.20
Isomenthone	0.27	0.0	11.20
Menthone	0.17	0.0	11.20

tion study was carried out for only one polymeric adsorbent (INDION 1014 MN-2) because the supply of the geranium condensate water was limited. The column exhaustion studies used 11.2 g of adsorbent.

REGENERATION

The regeneration plots for geraniol and isomenthone are shown in Figs. 11 and 12. The complete regeneration of the exhausted bed was possible through the use of 6–7 bed volumes of ethanol. The initial load, rest load, and mass of adsorbent used are given in the Table 7. The compound concentrations in the regeneration samples were relatively high. Thus, for citronellol, the original water concentration was 100 ppm whereas the regeneration sample showed a concentration of 9.6×10^4 ppm. This result indicates that the adsorbents are separating the organic compounds from water and concentrating them simultaneously. The extract was evaporated and the oil recovered. A material balance calculation indicated that the recovery of the components was in excess of 95%. The recovered oil had the typical pleasant geranium aroma.

CONCLUSIONS

Characterization of the geranium condensate water was completed. The polymeric adsorbent INDION 1014 MN-2 had greater adsorption capacity than the other samples in the single solute experiments. The single solute adsorption was totally dependent on the surface area of the adsorbent. However, in cases of multisolute system, competitive adsorption affected the experimental outcomes. We present evidence that increased adsorption from mixtures is due to hydrogen bonding of the solutes. The GC-MS results of the regeneration samples detected 3 important perfumery/flavor compounds, rose oxide, menthol, and α-terpineol that could not be detected in the condensate water due to their very low concentrations. The recovery of the compounds was also high (95%) indicating the feasibility of an adsorptive recovery process for these valuable solutes.

NOMENCLATURE

C_e	equilibrium concentration of solute (mmol/L) or (ppm)
C_o	initial concentration of solute (mmol/L) or (ppm)
K	Langmuir equilibrium adsorption constant (L/mmol)
m	mass of the adsorbent (g)
m_F	Freundlich constant
n	Freundlich constant
V	volume of solution (mL) or (L)
c	loading of the adsorbent (mmol/g)
c^*	maximum loading of the adsorbent (mmol/g)

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