Food Chemistry 82 (2003) 195–202

Food Chemistry

www.elsevier.com/locate/foodchem

Recovery of volatile aroma components from aqueous waste streams using an activated carbon column

Amr E. Edris[a,*], Badie S. Girgis[b], Hoda H.M. Fadel[a]

[a]*Aroma and Flavor Chemistry Department, National Research Centre, Dokki, Gizza, 12622, Egypt*
[b]*Surface Chemistry Laboratory, National Research Centre, Dokki, Gizza, 12622, Egypt*

Received 17 September 2002; accepted 22 October 2002

Abstract

The solid phase adsorption of aroma components depleted from essential oils and drained with the condensed water water during the distillation of aromatic plants onto granular activated carbon (GAC) is reported. A mini-column packed with laboratory-prepared activated carbon, derived from pistachio shells, which were previously treated with H_3PO_4 and pyrolysed at 500 °C, was used. Three aromatic waste waters (peppermint, lemongrass, and spearmint) discharged from a distillation plant, were chosen and allowed to pass through the column. GC and GC–MS techniques were employed to detect and quantify the selected key aroma components in the waste waters, and in the effluent waste water after passing through the carbon column. The high surface area GAC ($\approx$1300 m^2/g) proved highly efficient in retaining most of the selected aroma components of the waste waters up to 90 bed volumes of treatment without reaching exhaustion. The uptake of the aroma components by activated carbon was demonstrated to be dependent on their chemical structure and oxygen-functional groups. Hydrophilic aroma components were retained more selectively than hydrophobic ones, due to the acidic nature of the carbon surface, which had good affinity for water. Recovery of the aroma from the loaded carbon column, with diethyl ether, resulted in general quantitative recovery of the adsorped components and the degree of recovery (70–98%) depended on the chemical structure. Open chain components were partially entrapped within the internal porosity, which inhibited their complete recovery. The reported process of solid-phase separation appears useful for the recovery of water-soluble aroma components without affecting their physical or chemical characteristics and without need to raise the temperature.

Keywords: Activated carbon; Recovery; Volatile aroma; Essential oils; waste water

1. Introduction

Solid-phase extraction techniques (SPE), involving adsorbents, are nowadays extensively employed in various industrial processes, for example, purification, separation and recovery. The essential forces of adsorption are physical in nature, so that the physical and chemical properties of the sorbed compounds are not affected. Different types of sorbents have been used in industrial applications, e.g. zeolite, alumina, silica and activated carbon. The latter is an amorphous solid which is exploited in many industrial and environmental applications due to its large internal surface area (Kirk-Othmer, 1999) and its unique mesoporosity and microporosity (Wartelle, Marshall, Toles, & Johns, 2000). The adsorption characteristics of activated carbon depend on the type of starting material, methods and conditions of preparation (Wartelle et al., 2000). Hutta, Simunicova, Kaniansky, Tkacova, and Brtko (1989) have described a carbonaceous sorbent as a solid-phase material to recover short-chain fatty acids in drinking water. Jyun, Shinnyow, and Chou (2000) investigated the feasibility of removal of chlorinated volatile organic compounds from groundwater by activated carbon fibre adsorption.

The SPE technique plays an important role in isolation and recovery of volatile food flavour components (Coulibaly & Jeon, 1992). Deeth, Fitz-Gerald, and Snow (1983) used neutral alumina as a sorbent to isolate volatile free fatty acids from milk and dairy products. Matsukura, Takaahashi, Ishiguro, and Matsuchita (1984) studied the adsorption and recovery of 176 volatile aroma compounds from roasted tobacco plant on an activated carbon surface. Takacs (1989) isolated short-chain fatty acids and neutral volatile compounds

---

[*] Corresponding author.

doi:10.1016/S0308-8146(02)00503-4

from (UHT) processed milk, using C_{18} Sep-Pak materials. Sakano, Yonamura, Toma, Miyakara, and Okajai (1996) studied the improvment of coffee aroma by removal of pungent volatiles, using zeolite as adsorbent. Suryadi and Suresh (2000) reported the adsorption of flavour esters onto activated carbon. Wartelle et al. (2000) investigated the possibility of using activated carbons from nutshell to adsorb and recover volatile organic compounds for the purge-and-trap gas chromatographic analysis of flavours.

The application of activated carbon in the essential oil extraction industry seems scarce. Essential oils are highly concentrated flavouring agents. They are extracted from aromatic plants, mainly by steam- or water-distillation. During this process, the essential oil and the condensed steam (distillation waste water) are brought together in intimate contact for a long time. As a result, some of the essential oil components (mainly the oxygenated ones) are partitioned into the water phase. Partition of the polar volatile components from essential oils into the water phase during distillation results in an unbalanced aroma and flavour profile of the oil compared with that of the original plant, which leads to an inferior oil quality as well as financial loss in dumping the distillation water rich in expensive aroma flavouring (Fleisher, 1991). It is reported that the ratio of drained water-soluble aroma components to their parent essential oil could be, 1:1 in the case of basil essential oil (eugenol type) (Zukov, Andreevick, & Chipiga, 1979), 1:2 in the case of dittany oil (*Dictamnus gymnostylis* Stev.) (Kozhin, Fleisher, & Smirnov, 1972) and 1:3 in caraway oil (Fleisher and Fleisher, 1988).

For this reason, some techniques have been developed to regain the water-soluble aroma components, in order to refortify the parent oils. One of the oldest techniques being used is called “cohopation” or redistillation of the waste water, Gokhale (1959). The disadvantage of this technique is that it leads to only a partial and disproportional recovery of oil constituents (Zukov et al., 1979). A more advanced technique for recovery of aroma from distillation waste water is known as the “Poroplast” extraction technique, which shows both technical and economic efficiency (Fleisher, 1990).

To our knowledge, there are no reports discussing the utilization of activated carbon for extracting the water-soluble aroma volatile components of waste water from industrial essential oil distillation. Thus, this investigation examines the possibility of adsorbing these valuable aroma components, from the waste water of essential oil distillation, on the surface of activated carbon prepared from an agricultural waste product (pistachioshell), followed by their recovery using a low-boiling organic solvent. The extraction efficiency and recovery percentage of the key aroma components of each waste water were determined.

2. Materials and methods

2.1. Activated carbon

This was prepared and characterized at the Laboratory of Surface Chemistry, The National Research Centre, Cairo, Egypt.

2.2. Chemicals

Authentic samples of menthol (99.0%), menthone (90.0%), citral (95.0%), Myrcene (commercial, 89.0% by GC), l-carvone (98.0%), d-limonene (97.0%), 1,8-cineole (99.0%) and 3-nonanone (97.3%), Aldrich Chemical Co. Wisconsin, USA were used in this investigation. Their purities were re-checked by GC.

2.3. Aromatic waste waters of distillation

These were provided by the Horticultural Research Institute, Medicinal and Aromatic Plant Research Section, El Kanater El Khairya, Egypt. The aromatic waste waters were obtained as by-products of the steam-distillation of the fresh plant parts of peppermint (*Mentha piperita*), spearmint (*Mentha spicata* L.) and lemongrass (*Cimombogon citratus* L.). The aromatic waters proved to be free from any oil droplets, as revealed by the light microscope.

2.4. Preparation of activated carbon

Tested activated carbon was prepared from Pistachio shells; a discard, low-value agricultural waste. The water-washed dried solid was crushed and ground to a size of 0.5–2.0 mm, then soaked in 50% (volume) H_3PO_4 and left for 3 days. The acid-impregnated mass was pyrolysed in a stainless steel tube in a tube furnace, at 500 °C for 2 h. The cooled product was thoroughly washed with hot water, and finally dried in an air oven at 110 °C to constant weight. The produced carbon exhibited an acidic effect of pH 4.8 and total acidity, by sodium hydroxide, as suggested by Boehm (1994), equal to 0.9 mmol/g.

2.5. Characterization of activated carbon

Porosity characteristics were estimated by the standard adsorption of N_2 at 77 K, using a sorptometer of the type Gemini 2375 V3.03 (Micromeritics). A sorption isotherm was obtained to find several pore characteristics: (1) The BET-surface area ($S_{\mathrm{BET}}\mathrm{m}^2/\mathrm{g}$), the total pore volume from the nitrogen held near the saturation pressure (V_{p}, cm^3/g) and average pore radius from $r^{-} = 2\ V\mathrm{p} \times 10^4/S_{\mathrm{BET}}$; (2). More information was obtained by comparison with adsorption data on a non-porous carbon, known as the α_s-plot (Selles-Perez &

Martin-Martinez, 1991). From the slope of the initial linear section the total surface area ($S_t^\alpha m^2/g$) was obtained the slope of later rectilinear section corresponded to the non-microporous surface area (S_n^α, m^2/g) and extrapolation of this latter straight line to the V_a-axis gave an estimate of the micropore volume (V_o^α, cm^3/g). The volume within mesopores was then calculated from $V_{meso} = V_p - V_o^\alpha$ (cm^3/g).

2.6. Extraction and determination of the absolute amount of some key aroma components in aromatic waste water

Four hundred millilitres of each aromatic waste water was extracted four times with a total of 800 ml methylene chloride in a separating funnel (4 extractions×200 ml solvent each). These extractions proved to be enough for collecting all the aroma components from the waste waters, as revealed by the GC for a fifth extraction. The solvents were collected in a 2-l conical flask, dried over anhydrous sodium sulfate, evaporated using a rotary evaporator to ca. 10 ml and transferred into a vial immersed in an ice bath; then the remaining solvent was further evaporated, slowly, under a nitrogen stream to a volume of 1 ml. The earlier experiment was conducted three times. For absolute quantitation of some key aroma components, 3 μl of 3-nonanone was used as an internal standard (IS). The IS was added to each vial just before injection into the GC to give a final concentration of 2.39 mg (IS)/ml solvent/400 ml aromatic waste water. The samples were then quickly injected into the GC. The response factor and relative response factor of each calibrated aroma compound was calculated to determine their absolute amounts.

2.7. Adsorption of aroma components from the waste water on carbon surface

One gram of activated carbon (mesh size 0.5–2.0 mm) was packed into a glass column (20 cm length×8 mm internal diameter) fitted with a stop-cock. The carbon occupied a volume of 4.5 cm inside the column, which will be referred to as the "carbon bed volume". Four hundred millilitres of each of the three aromatic waste waters were allowed to drop from a separating funnel over the carbon bed at a rate of 5 ml/min. The aromatic water current formed a constant height of about 2 cm in the glass column over the carbon bed.

In order to test the change in adsorption efficiency of carbon for the aroma components, at different time intervals, the effluent (400 ml) was collected in eight vials (fractions), each containing 50 ml of the effluent waste water. The absolute amount of aroma component in each effluent fraction was determined.

2.8. Extraction and quantitation of aroma components from each effluent fraction

The earlier-mentioned method was used, as in the determination of the absolute amount of major aroma components in waste water, except for changing the amount of internal standard from 3 μl/ml solvent to only 1 μl/ml solvent to match the small quantities of aroma compounds in 50 ml of effluent.

2.9. Recovery and quantitation of the adsorbed aroma components from the carbon surface

Adopting the procedure of Matsukura et al. (1984), the carbon bed was removed from the glass column, placed over a filter paper and air-dried at room temperature for 1 h then extracted three times with 30 ml of diethyl ether (3 extractions×10 ml for each extraction). The ether was then collected, dried over anhydrous sodium sulphate, transferred to a vial and slowly evaporated to exactly 1 ml in an ice bath using nitrogen. 3-Nonanone was added to the vial as an internal standard for quantitation at a level of 3 μl (2.398 mg)/1 ml solvent/1 g carbon, then injected into the GC.

2.10. GC analysis

GC analysis of the aroma components was conducted using a Perkin-Elmer model system XL equipped with a flame ionization detector (FID). A 60 m×0.32 mm i.d. fused silica capillary column, coated with DB-5, was used. The oven temperature was programmed from 50 to 200 °C at a rate of 3 °C/min. The injector and detector temperatures were 220 and 250 °C, respectively. Helium was used as a carrier gas at a flow rat of 0.8 ml/min. Values reported were averages of two analyses.

2.11. GC–MS analysis

GC–MS analysis was conducted on a Hewlett Packard 5985 coupled with an HP MS instrument system. The ionization voltage was 70 eV and the ion source temperature was 200 °C. Other parameters were the same as in the GC conditions.

2.12. Component identifications

The components under investigation were identified by one or all of the following procedures: (1) matching their mass spectrum with that of (NBS) MS library or other published mass spectra (Stenhagen, 1972), (2) authentic sample injection and retention time matching with the unknown, (3) co-ejection of the authentic with the unknown and (4) matching of the retention indices of the unknowns with reported data (Sadteler, 1986), using standard hydrocarbons (C_8–C_{18}) (Aldrich Chemical Co.).

3. Results and discussion

3.1. General features of the adsorbent and waste waters

Analysis of the N_2/77 K adsorption isotherm resulted in a number of porous characteristics, as detailed in the previous section (Table 1). The employed, laboratory-prepared, activated carbon appeared to be a high quality adsorbing carbon. Thus, its ‘‘apparent surface area’’ was high ($\geqslant 1250$ m^2 g^{-1}), as estimated by both the BET method and the comparison method by the α_s plot. Its internal pore volume was also extensive (0.87 ml mg^{-1}), and contained both micropores and meso/macropores to the same extent. This was clear from the estimated micropore/mesopore volumes (0.464/0.406 ml g^{-1}) as well as the micropore/non-micropore surface areas (563/731 m^2 g^{-1}). It is thus a mixed-porosity carbon and seems suitable for adsorption from solution, where its average pore diameter amounts to 27.

On the other hand, GC analysis of the three aromatic waste waters of distillation from peppermint, lemongrass and spearmint, revealed the presence of numerous components. This investigation focussed only on the key volatile components that give the characteristic aroma and flavour to each corresponding essential oil. These components are hydrophilic due to the presence of oxygen in their structures. The study also focused on some hydrophobic components (straight C–H components), regardless of their scarcity (relative area %), in the waste water, in order to investigate the effect of component polarity on its adsorbability on the carbon surface. These components are: menthol, menthone, 1,8-cineole and myrcene for peppermint waste water, citral (*cis*-neral and *trans*-geranial) and myrcene, for lemongrass waste water and l-carvone, 1,8-cineole and limonene for spearmint waste water. The percentages of these components in the waste water are reported in Table 2. The structures of these aromatic components are schematically shown in Fig. 1, which indicates that these selected components have different polarities, ranging from polar (hydrophilic alcohols, aldehydes, ketones) to non-polar (hydrophobic terpenic hydrocarbon components).

Table 1
Texture characteristics of the activated carbon

Parameter	Value	Parameter	Value
S_{BET} (m^2 g^{-1})	1260	S_t^α (m^2 g^{-1})	1294
V_p (ml g^{-1})	0.870	S_n^α (m^2 g^{-1})	731
r^- (Å)	13.8	V_o^α (ml g^{-1})	0.464
V_{meso} (ml g^{-1})	0.406	S_{mic} (m^2 g^{-1})	563

3.2. Carbon removal of aroma components from peppermint waste water

The waste water of peppermint proved to contain low amounts of the four key aroma components: menthone, menthol, 1,8-cineole and myrcene (total = 39.6 mg l^{-1}), Table 2. These values were lower than those found in a previous study (110 mg l^{-1} for the same four components) [unpublished data]. The low content of aromatics in this waste water could be accidental in this batch, due to the changing quality of the aromatic plant, the season of cultivation or the conditions of the steam-distillation unit.

Table 2
Carbon column performance towards the different waste waters and amounts recovered by ether

Waste water	Component	Relative area % of aroma component in the aromatic waste water	Absolute amount of aroma component in the waste water (mg l^{-1})	% of aroma components retained on carbon column	% of aroma component recovered by ether	Total aroma components adsorbed on carbon mg/g[a]
Peppermint	Menthone	15.8	17.4	96.8	96.2	14.3
	Menthol	0.84	11.0	98.6	90.8	
	1,8-Cineole	9.81	10.7	72.6	100	
	Myrcene	0.44	0.42	49.7	100	
	Total		39.6	90.2		
Lemongrass	Neral	37.1	77.6	92.6	69.4	66.9
	Geranial	43.1	97.6	95.1	65.1	
	Myrcene	1.23	1.92	68.7	100	
	Total		177	93.8		
Spearmint	l-Carvone	81.4	269	78.1	99.2	85.2
	Limonene	0.31	0.647	17.3	100	
	1,8-Cineole	2.57	6.75	42.4	100	
	Total		276	77.3		

[a] Volume of waste water treated with 1 g activated carbon was 400 ml. The values are averages of three experiments, S.D. not more than ±1.

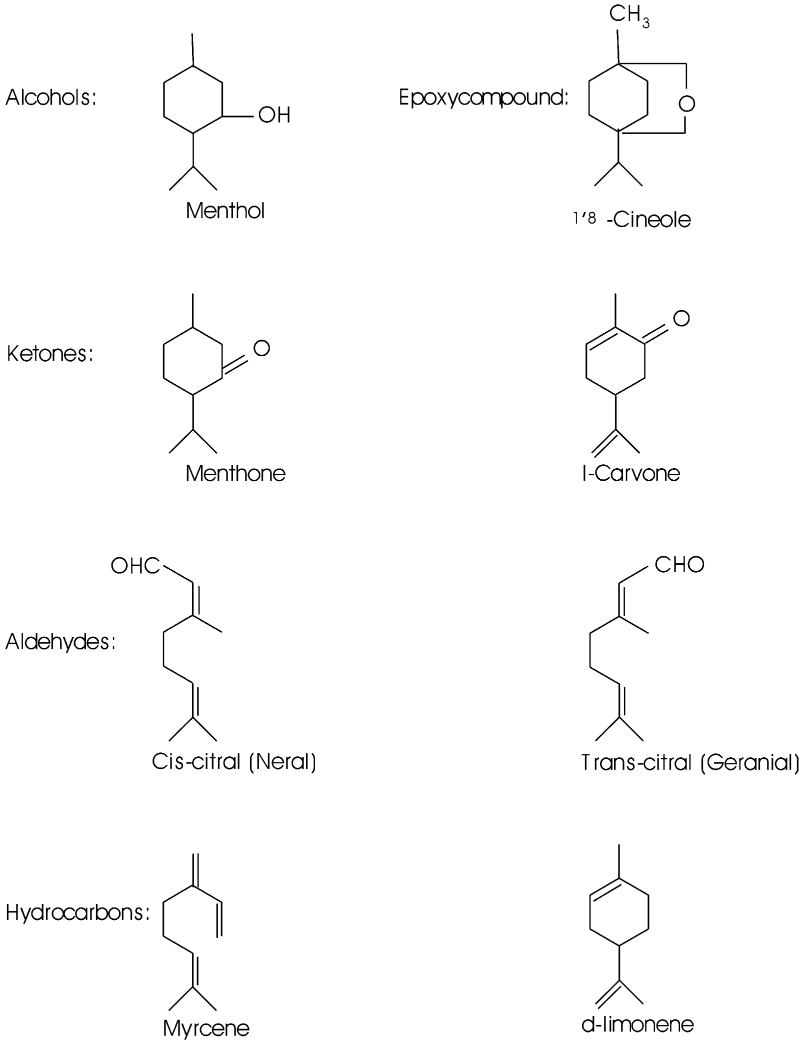

Fig. 1. Chemical structure of the key aroma components in peppermint, lemongrass and spearmint distillation waste waters.

Fig. 2 presents the contents of the four components in the effluent fractions and the change of adsorption efficiency of carbon over time, or the breakthrough curve. This strongly indicates that the carbon-column retains most of the menthone and menthol, with appreciable retention for 1,8-cineol, whereas myrcene is poorly removed, particularly in the early stages of separation. Although the first three aroma components exhibit typical breakthrough curves that either slightly or appreciably increase with time, myrcene shows anomalous behaviour. Thus, it begins with a high content in the effluent (≈90%) which thereafter decreases to a lower content in further fractions (around 40–50%). This means that the tested active carbon selectively captured both menthone and menthol in the early stages. This could be due to the high affinity of the polar surface of carbon for these hydrophilic components, but a much lower affinity for the hydrophobic myrcene.

Table 2 shows the overall carbon column performance for the adsorption and retention of the volatile components, as well as the amounts recovered from the adsorbent by extraction with ether. The data quantitatively indicate the selectivity order and the percents removed appear to be 98.6 for menthol, 96.8 for menthone, 72.6 for cineol and 49.7% for myrcene. Ether recovery of the adsorbed components seems to be quantitative and efficient with respect to the main components, menthol and menthone (⩾90% recovery).

3.3. Carbon removal of aroma components from lemongrass waste water

In this case the three key aromatic components neral, geranial and myrcene represent 177 mg l^{-1}. Fig. 3 shows the carbon column performance and breakthrough curves for the three components in the lemongrass waste water. They seem normal up to the 6th fraction, the concentrations of the aromatic components increasing in the effluent water with time; then the rates of increase suddenly slow down. Such a trend can be accounted for by the slow partial diffusion of the aroma components into the internal porosity of the highly porous carbon

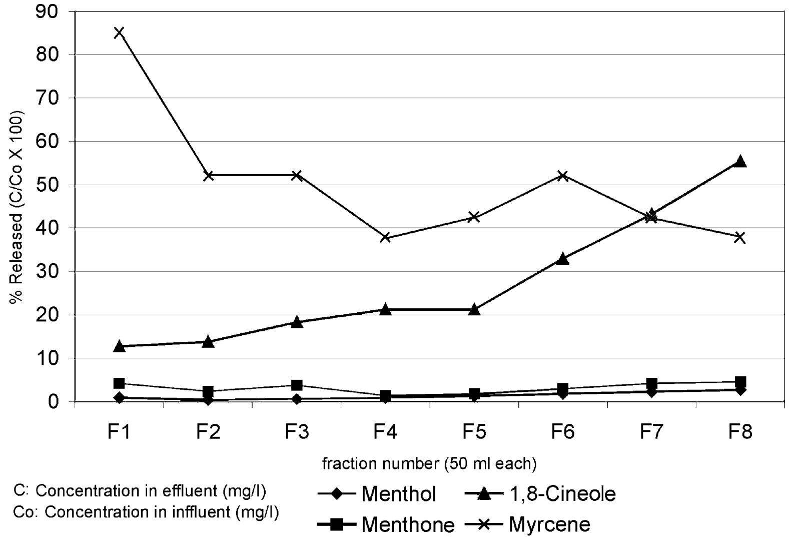

Fig. 2. Distribution of the non-adsorbed aroma components of peppermint waste water among different effluent fractions.

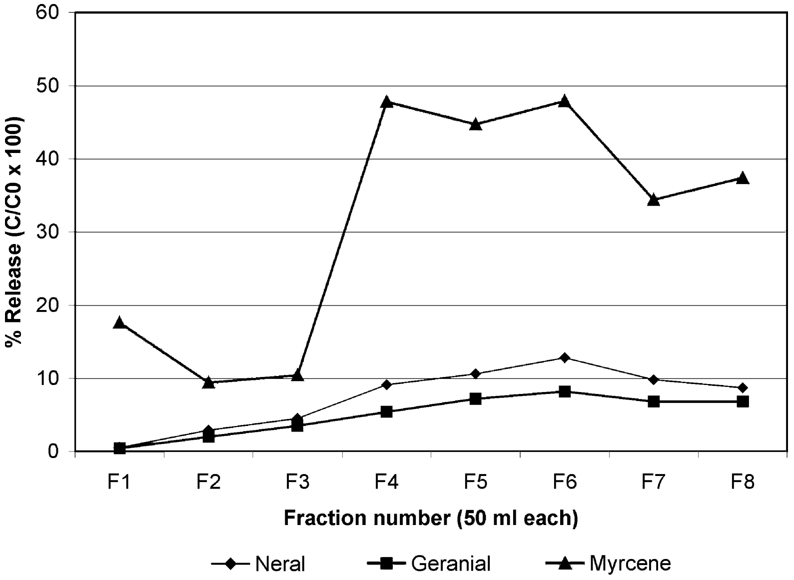

Fig. 3. Distribution of the non-adsorbed aroma components of lemongrass waste water among different effluent fractions.

adsorbent. In the early stages, the organic compounds are primarily adsorbed on the external surface of the adsorbent particles, then they slowly diffuse internally. The two isomers neral (*cis*) and geranial (*trans*) are more selectively adsorbed, whereas myrcene, as in the previous case of peppermint waste water, shows less affinity for the carbon. Based on the data in Table 2, this affinity is shown by the removal efficiency of the three components after the flow of the 400 ml batch (93.0, 95.0 and 69.0% for neral, geranial and myrcene, respectively). These components seem to possess certain structural properties that reduce their desorption, such that treatment with ether was not efficient for their complete recovery (only 65–69%). Their open chain nature probably promoted their slipping and diffusion inside the pore system where they are captured and retained to become unavailable for the ether extraction. In contrast (cf. Fig. 1), all other components under study (carvone, menthol, menthone and 1,8-cineole) are cyclic compounds which might limit their adsorption to the external surface area; this therefore results in their easy recovery. Moreover, it is also probable that the affinity of the open-chain aldehydic compounds (neral and geranial) for the carbon surface is higher than for the solvent (ether), which also reduces their liquid-solid partition.

3.4. Carbon removal of aroma components from spearmint waste water

This waste water contained a total of 275 mg l^{-1} of l-carvone, 1,8-cineole and limonene. l-Carvone represents 97.5% of this value while cineole represents only

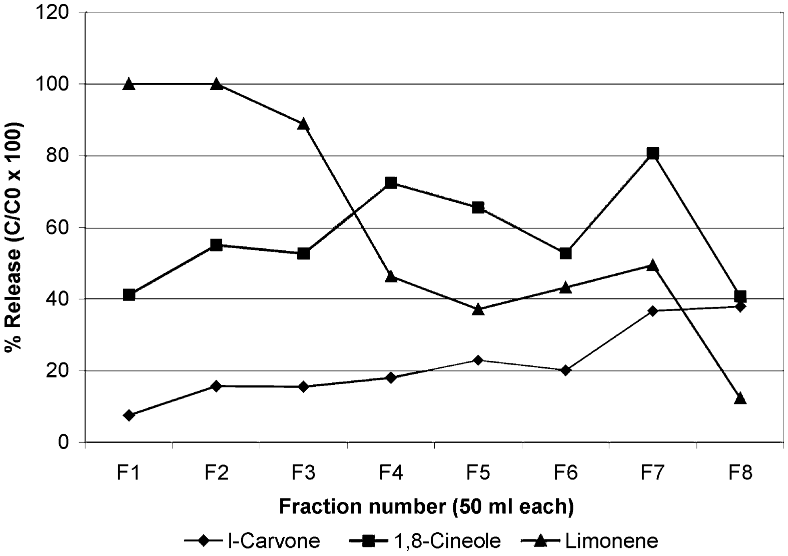

Fig. 4. Distribution of the non-absorbed aroma components from spearmint waste water among different effluent fractions.

2.5% and limonene only a very low amount. Fig. 4 and Table 2 show that l-carvone is adsorbed with a high efficiency although not complete (78%), and 1,8-cineole is captured to a moderate but lower extent (42%). Limonene is not retained in measurable amounts before the fourth fraction. Despite this observation, their affinity to the carbon surface is small, such that leaching with ether resulted in complete recovery of the adsorbed components. Adsorption of 1,8-cineole is here much lower than in case of the peppermint oil (42 and 72%, respectively). This may be due to a strong competitive action of l-carvone due to its high content, which shields most of the available adsorption sites. The remaining sites will thus accommodate a smaller amount of the 1,8-cineole molecule, as well as of limonene. Such behaviour may be explained by the trends of their breakthrough curves; that of l-carvone increases slowly and regularly with time and those of cineol and limonene seem irregular. The drop in effluent concentration at the eighth fraction may be ascribed to the same effect postulated earlier, as the consequence of slow pore diffusion.

4. Conclusions

Activated carbon, obtained from pistachio shell residues by chemical activation with H_3PO_4, is a high quality product as is evident from its high surface area and pore volume. Treatment of waste waters resulting from aromatic plant distillations was achieved in a fixed carbon bed in order to demonstrate the feasibility of capture and recovery of the valuable aroma components. Assessment was based on three waste waters of different sources: peppermint, lemongrass and spearmint. Removal of aroma components, through adsorption on the carbon bed, was considerable and ranged from excellent (⩾90%) to moderate (44–77%). Some components were more selectively adsorbed and almost disappeared in the effluents, whereas others were fairly adsorbed (40–70%). Selectivity of uptake was ascribed to the hydrophilicity of the *O*-containing substituent groups in the aroma compounds, and to the hydrophilic acidic carbon surface. The aroma-loaded carbon was leached with diethyl ether to recover adsorbed matter and showed excellent to complete recovery for certain components in peppermint and spearmint waters, but only moderate recovery (≈70%) in the case of lemongrass water. Separation of aroma matter by adsorption on an activated carbon column proved highly efficient for recovery and reuse of these compounds, and to reduce the volume of discharged waste water loaded with these compounds. The process involves no heat treatment that might affect the structure, and the adsorbent causes no alteration in the chemical nature of the aroma components. In seasons of continuous distillation in aromatic plants, large volumes of aromatic waste waters can be passed over a cartridge filled with activated carbon for capturing the soluble aroma components which then can be used for re-fortification of their natural parent oils, to accomplish a full and authentic aroma.

Acknowledgements

The authors are indebted to Dr. Said Gabr and Dr. Ebrahim Haridi, at the Horticultural Research Institute, Medicinal and Aromatic Plant Research Section, El Kanater El Khairya, Egypt, for donating the waste water necessary for this study.

References

Boehm, H. P. (1994). Some aspects of the surface chemistry of carbon blocks and other carbons. *Carbon*, *32*, 759–763.

Coulibaly, K., & Jeon, J. (1992). Solid-phase extraction of less volatile flavour compounds from ultrahigh-temperature processed milk. *Journal of Agricultural and Food Chemistry*, *40*, 612–616.

Deeth, C., Fitz-Gerald, C., & Snow, A. (1983). Gas chromatographic method for the quantitative determination of free fatty acids in milk products. *New Zealand Journal of Dairy Science & Technology*, 18–20.

Fleisher, A. (1990). The Poroplast extraction technique in the flavour and fragrance industry. *Perfumer & Flavorist*, *15*(5), 27–36.

Fleisher, A. (1991). Water-soluble fractions of the essential oils. *Perfumer & Flavorist*, *16*, 37–41.

Fleisher, A., & Fleisher, Z. (1988). The essential oil of annual *carum carvi* L. grown in Israel. In B. M. Lawrence, B. Mookherjee, & B. J. Willis (Eds.), *Flavor and fragrance: a world perspective* (pp. 33–40). Amsterdam: Elsevier Science Publishers BV.

Gokhale, N. N. (1959). The distillation waters. *Industrial Perfums III, Part II*, 95–97.

Hutta, M., Simunicova, E., Kaniansky, D., Tkacova, J., & Brtko, J. (1989). Isotachophoretic determination of short-chain fatty acids in drinking water after solid-phase extraction with a carbonaceous sorbent. *Journal of Chromatography*, *470*, 223–233.

Jyun, Y., Shinnyow, C., & Chou, S. (2000). Contaminated site remidal investigation and feasibility removal of chlorinated volatile compounds from ground water by activated caarbon fiber adsorption. *Chemosphere*, *41*(3), 371–378.

Kirk-Othmer, J. (1999). *Concise encyclopedia of chemical technology* ((4th ed.)). New York: John-Wiley.

Kozhin, S., Fleisher, A., & Smirnov, A. (1972). Recovery of essential oils under field conditions. *Rastitielnie Resursi*, *8*, 279–282.

Matsukura, M., Takaahashi, K., Ishiguro, S., & Matsuchita, H. (1984). Adsorption of volatiles from roasted tobacco on activated carbon and desorption recovery by ether extractionn. *Agricultural and Biological Chemistry*, *84*(4), 971–975.

Sadtler, R. (1986). *The Sadtler standard gas chromatography retention index library Volume 4*. Philadelphia Pennsylvania: Sadtler Research Laboratories, Inc.

Sakano, T., Yonamura, H., Toma, H., Miyakara, M., & Okajaki, M. (1996). Improvment of coffee aroma by removal of pungent volatiles using A-type zeolite. *Journal of Food Science*, *61*, 473–476.

Selles-Perez, M., & Martin-Martinez, J. (1991). Application of α- and n plots to N_2 adsorption isotherms of activated carbons. *Journal of the Chemical Society Faraday Trans.*, *87*, 1237–1245.

Stenhagen, E., Abrahamsson, S., & Mclafferty, F. (1972). *Registry of mass spectral data*. New York: Wiley.

Suryadi, I., & Suresh, B. (2000). Adsorption of flavour esters on granular activated carbon. *The Canadian Journal of Chemical Engineering*, *78*, 892–901.

Takacs, F. (1989). *Solid-phase extraction of stale flavour components from ultra-high-temperature processed milk*. MS thesis, Kansas State University, Manhattan, KS.

Wartelle, L., Marshall, W., Toles, C., & Johns, M. (2000). Comparison of nutshell franular activated carbons to comercial adsorbents for he purge-and-trap gas chromatographic analysis of volatile organic compounds. *Journal of Chromatography A*, *879*, 169–175.

Zukov, D., Andreevick, E., & Chipiga, A. (1979) Technology and equipment for essential oil industry. In *Food industry, Moscow*.