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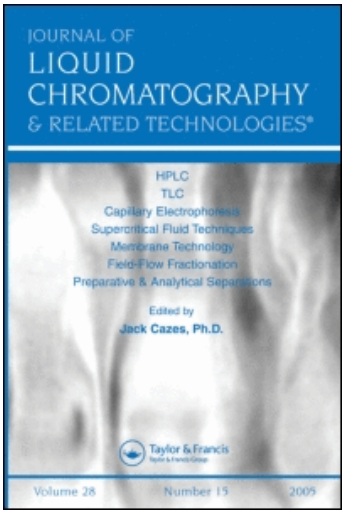

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DETERMINATION OF INTERACTION OF PACKAGING AND FOOD COMPONENTS WITH PACKAGING MATRIX BY HPLC

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ABSTRACT

Interaction of polymeric oligomers of different molecular weights and food components with the packaging matrix was studied by immobilizing polyvinyl alcohol on silica support and using food based solvent like water as a mobile phase. The enthalpy of sorption data as determined from the slopes of the plots of lnK' against 1/T indicated an exothermic adsorption process for polymeric oligomers and niacin. An endothermic adsorption process for caffeine was observed.

INTRODUCTION

Polymeric materials are being used abundantly as containers for packaging of foods and drugs. The presence and migration of oligomers or residues of low molecular weight can affect the quality of packaged products. Therefore, it is important to study interaction of such residual materials from packaging with

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food components or packaging itself. Inverse gas chromatography (1, 2) have been used to study interaction of volatile food components with the packaging products. Changes in thermodynamic Parameters such as enthalpy of adsorption was used to conduct such study. During the present investigation, HPLC has been used to study interaction of nonvolatile components by immobilizing polymeric material such as polyvinyl alcohol on the silica support and using food based solvent like water as a mobile phase.

EXPERIMENTAL

Materials

Polyvinyl alcohol (PVA) of molecular weight 125,000, 95,000, 25,000 and 14,000, caffeine, niacin, methanol, glycerol and acetonitrile were purchased from Sigma Chemical Co., Inc. (St. Louis, MO). Epoxypropylsilanized PartiSphere-5 was obtained from Whatman Inc. (Clifton, NJ).

Preparation of Packing Material

PVA of molecular weight 125,000 was immobilized on silica support by reacting 10 g of epoxypropylsilanized PartiSphere-5 with 300 mL of 3.5% solution of PVA (larger excess amount) (pH 7.4) at 60-70^{o}C for 2 hrs. Elemental analysis, 1. epoxypropyl-silanized PartiSphere-5: C = 1.7%, H = 0.24%; 2. PVA bonded epoxy phase: C = 3.7%, H = 0.48%.

The column was packed by slurrying the PVA immobilized phase in methanol and applying a pressure of 6,000 psi. The unreacted epoxy groups were deactivated by treating the columns with 2% aqueous solutions of glycerol.

Sample Preparation

The solution of PVA of different molecular weights were prepared by dissolving 100 mg of PVA (MW, 95,000) in 8 mL water, 100 mg of PVA (MW, 25,000) in 8 mL water and 200 mg of PVA (MW, 14,000) in 12 mL water. The solutions of caffeine and niacin were prepared by dissolving 20 mg caffeine in 20 mL water and 20 mg of niacin in 5 mL water.

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HPLC Analysis

HPLC was performed by using a variable wavelength UV detector, Spectroflow monitor SF-770 (Kratos Analytical, Ramsey, NJ); a programmable solvent delivery system, Series 3B (Perkin-Elmer Corp., Norwalk, Conn.); a manual injection valve, with 50 L loop (Valco Instruments Co., Houston, TX) and a chart recorder (Laboratory Data Control, Riviera Beach, FL). The columns were run by using water as a mobile phase at 0.7 mL/min. Acetonitrile was used as a reference to provide an unretained peak. The enthalpy changes were determined from the slope of the plots of lnK′ against 1/T by using the equation 1 (3-6).

$$\ln K' = \Delta H^0/RT - \Delta S^0/R + \ln \Phi \quad \text{(Equation 1)}$$

RESULTS AND DISCUSSION

The capacity factor, K′, decreases on increasing the absolute temperature of the column, T (3-6). The relationship between K′ and T is shown by equation 1 where ΔH^0 is the standard enthalpy changes on transferring a solute from a stationary phase to a mobile phase, ΔS^0 is the standard entropy change and Φ is the phase ratio of the column. The standard enthalpy change can be derived from the slope of the plots of lnK′ against 1/T (van't Hoff's plot). The value of ΔH^0 determines the effect of temperature on the retention or it can decide the migration of the residual materials from packaging to the container contents.

Figure 1 represents the plot of lnK′ or capacity factor against 1/T for polymeric oligomers. The values of ΔH^0 as determined from the slopes of the graphs are shown in the Table 1.

Enthalpy of sorption showed negative values for all oligomers. Negative values indicated an exothermic adsorption process. It showed that there is interaction of all these probes with the polymeric surface due to O···H bond formation etc. which is weaker than their affinity for the mobile phase. It explains

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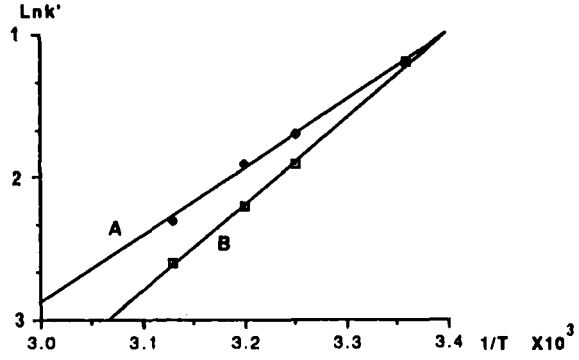

Fig. 1. Plot of lnK′ or capacity factor against 1/T, where T is the column absolute temperature.
Column: PVA (MW, 125,000) immobilized epoxypropyl-silanized PartiSphere-5 (20 cm x 4.6 mm, I.D.). Mobile phase: water; flow rate: 0.7 mL/min; λ_{max} : 203; sample size: 10 μL in each case.
A. The linear plot of PVA of molecular weight 95,000 and 25,000.
B. The linear plot of PVA of molecular weight 14,000.

Table 1. Enthalpy of sorption of polyvinyl alcohol of different molecular weights as determined from the Figure 1.

PVA	ΔH^0 [Kcal/mol]
95,000	-6.103
25,000	-6.103
14,000	-4.715

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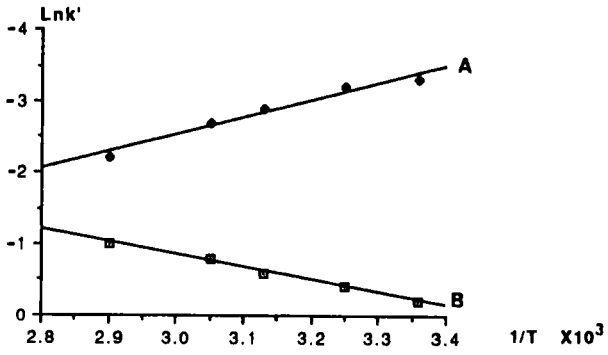

Fig. 2. Plot of lnK' or capacity factor against 1/T, where T is the column absolute temperature.
Column: PVA (MW, 125,000) immobilized epoxypropyl-silanized PartiSphere-5 (20 cm x 4.6 mm, I.D.). Mobile phase: water; flow rate: 0.7 mL/min; λ_{max} : 280; sample size: 10 μL in each case.
A. The linear plot of niacin.
B. The linear plot of caffeine.

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their fast elution from the column at various temperatures from 25°C to 47°C. It indicates that it will be possible for these types of residual oligomers to migrate from the packaging system of polymeric matrix such as polyvinyl alcohol into the water based food systems.

Figures 3 and 4 show the resolution of food components such as niacin and caffeine at 47°C and 70°C. Figure 2 represents the plots of lnK' against 1/T. Table 2 exhibits the values of ΔH^{o} determined from the slopes of the graphs in the Figure 2.

Enthalpy of sorption in these cases showed negative value in case of niacin and positive value in case of caffeine. These values indicated an exothermic adsorption process for niacin and exothermic value for caffeine. This indicates that the polar component like niacin exhibits interaction with surface due to N $\cdots$ H bond formation etc. It is evident from negative ΔH^{o} value that niacin shows more interaction with the surface than caffeine. It is possible for the polar component like niacin to

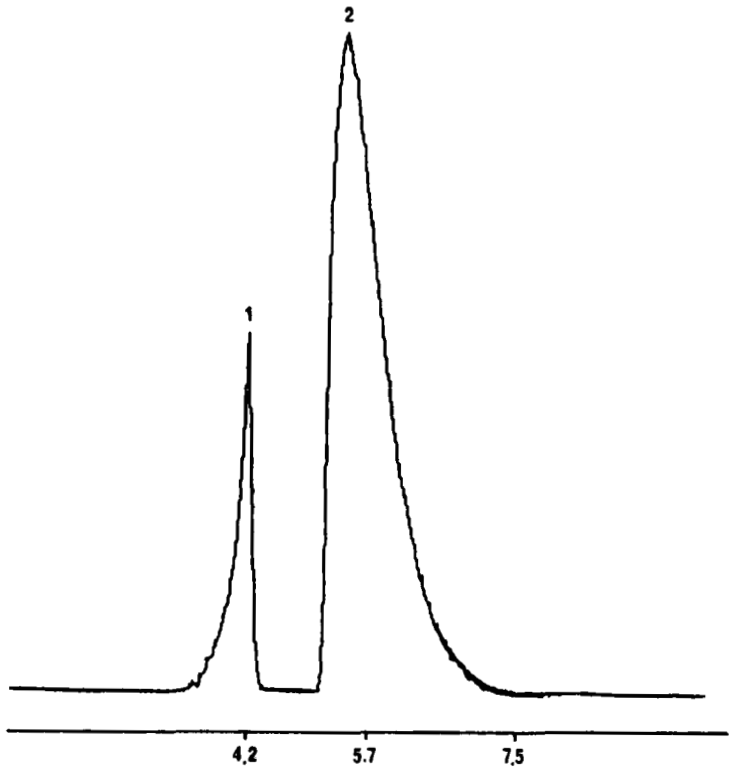

Fig. 3. Resolution of niacin from caffeine on PVA immobilized epoxypropylsilanized PartiSphere-5 (20 cm x 4.6 mm, I.D.). Mobile phase: water; flow rate: 0.7 mL/min; temperature: 47°C; λ_{max} : 280; sample: mixture containing 20 mg each of niacin and caffeine in 40 mL water; sample size: 10 μL.
1. Niacin 2. Caffeine.

Table 2. Enthalpy of sorption of niacin and caffeine as determined from the Figure 2

Components	ΔH^{o} [Kcal/mol]
Niacin	-2.428
Caffeine	+1.734

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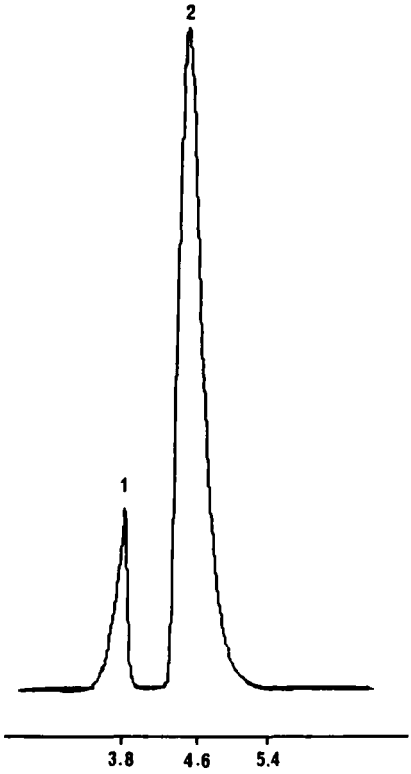

Fig. 4. Resolution of niacin from caffeine on PVA immobilized epoxypropylsilanized PartiSphere-5 (20 cm x 4.6 mm, I.D.). Mobile phase: water; flow rate: 0.7 mL/min; temperature: 72°C; λ_{max} : 280; sample: mixture containing 20 mg each of niacin and caffeine in 40 mL water; sample size: 10 μL.
1. Niacin 2. Caffeine.

migrate from the food based solvent such as water into the packaging matrix such as polyvinyl alcohol due to its stronger interaction for the surface.

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