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Sorption Behavior of 1-Methylcyclopropene on Adsorbing Agents for Use in Extending the Freshness of Postharvest Food Products

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Abstract The physiochemical interactions of 1-methylcyclopropene (1-MCP) and adsorbing agents can be described using a very powerful tool, inverse gas chromatography (IGC). Sorption behavior of 1-MCP on various adsorbing agents was assessed using the profile peaks of 1-MCP at an infinite dilution concentration using the IGC technique. Chromatogram peaks of 1-MCP adsorption were not observed for the adsorbing agent activated carbon. The forms of sorption isotherms followed Henry's law, and behaved according to the binding site theory. Specific retention volume and distribution coefficients for 1-MCP on the adsorbing agents were determined at 50, 60, 70, and 80°C, respectively. Silica gel had a much higher number of binding sites for 1-MCP compared to Tenax-TA and activated clay agents. Meanwhile, activated carbon proved to be a very strong binding agent for 1-MCP based on 1-MCP efficiency experiments on the selected adsorbing agents. However, as a proper means of delivering 1-MCP molecules to fresh food products, activated carbon is not fit for the binding and release of 1-MCP gas under dry or high humidity conditions because activated carbon has a strong affinity for 1-MCP, even when treated with distilled water.

Keywords: inverse gas chromatography, 1-methylcyclopropene, sorption behavior, adsorbing agent, fresh food product

Introduction

In the fresh food industry, postharvest treatments are the most important processes used to extend freshness during storage, transport, and handling. Biochemical and physiological changes in fresh food products depend mostly on environmental conditions. Postharvest environmental treatments include low temperature storage under air or controlled atmosphere (CA), and/or by treating them with chemicals which affect plant hormone action (1). Lower storage temperatures reduce respiration and fungal growth in fruits. However, very low temperature treatment causes chilling injury (2), or can stimulate autocatalytic ethylene synthesis (3). CA storage is used commonly to delay the ripening process of fruits (4). CA storage needs to be used with selected temperatures for certain fruits because CA storage does not control all diseases or disorders effecting fruits without temperature control. However, CA storage with selected temperatures requires costly environmental systems.

As a major ripening hormone for climacteric fruits, ethylene promotes undesirable reactions inducing abscission and physiological damage, and tends to shorten storage life (5). Chemical treatments with silver thiosulphate (STS), diazocyclopentadiene (DACP), and 2,5 norbornadine (NBD) inhibit ethylene action in fresh food products. These chemicals reduce the effects of ethylene by blocking the receptor site that signals ethylene action. STS, DACP, and NBD compete with ethylene for the binding receptor and are very effective inhibitors of the ethylene response. However, these inhibitors are limited in their commercial application in the horticultural industry because of their unstable and explosive potential (DACP), toxicity and odor (NBD), and environmental contamination (STS) (6). Thus, there is a need to find alternatives to these compounds.

Recently, 1-methylcyclopropene (1-MCP), a nontoxic gas, has begun to be used commercially as an agent to extend the storage life of fruits, vegetables, and flowers. 1-MCP acts by inhibiting the binding of the ethylene to its receptor sites and is effective at low concentrations (parts per billion levels) (7). Several studies have been conducted on the efficacy of 1-MCP as an inhibitor of ethylene action in ethylene-sensitive cut flowers, vegetables, and fruits (8-10). However, pure 1-MCP has problem with regard to stability and its potential explosive hazard as a compressed gas (11). As a convenient and safe means of delivering 1-MCP to fresh produce, 1-MCP gas may be delivered by the addition of an inert carrier system such as silica, talc, and dust. Therefore, by properly delivering 1-MCP to fresh produce in the package, it is possible to prevent or retard undesirable postharvest ethylene effects. A system that can deliver small quantities of 1-MCP vapor into the headspace of a package containing fresh produce is desirable. An adsorbing agent can absorb and/or desorb active molecules of 1-MCP. The physiochemical interactions of 1-MCP and the adsorbing agent can be described using a very powerful tool, inverse gas chromatography (IGC). IGC can be used to study the characteristics of the stationary phase of specific solid materials and their interaction with sorbate molecules (12).

The objective of this study was to investigate the interaction behavior of 1-MCP with selected adsorbing agents to obtain valuable information for delivering 1-MCP associated with adsorbing agents for application to fresh food products.

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Materials and Methods

Materials The selected materials used as adsorbing agents in these studies were silica gel, grade 634-60A, 100-200 mesh (Sigma-Aldrich Chemical Co., Milwaukee, WI, USA), Tenax-TA, 60-80 mesh (Alltech Associates, Inc., Deerfield, IL, USA), activated clay (a modified montmorillonite mineral, I-28E; Nanocor, Inc., Arlington Heights, IL, USA), activated carbon, 20-40 mesh (Sigma-Aldrich Chemical Co.), and activated carbon, 80-100 mesh (Alltech Associates, Inc.). 1-MCP was obtained from Floralife, Inc., (Walterboro, SC, USA) as EthylBloc® powder containing 0.14% active ingredient. 1-Butene (99.9% purity) was purchased from AGA Specialty Gas Inc., (Maumee, OH, USA) for quantitative analysis of 1-MCP.

Preparation of 1-MCP gas 1-MCP was released from the EthylBloc® as gas by adding aliquots of deionized water to pre-weighed EthylBloc® powder in a 250 mL glass serum vial sealed with aluminum top-hole caps and PTFE/Silicone 20 mm septa (Supelco Inc., Bellefonte, PA, USA). The concentration of 1-MCP was measured by sampling 200 mL of the headspace volume using a gas tight microsyringe (Hamilton Co., Reno, NV, USA) relative to certified pure 1-butene gas. The standard calibration solutions were prepared using a serial dilution procedure using 0.05, 0.10, 0.15, and 0.20 μg concentrations of a certified pure 1-butene.

Inverse gas chromatography (IGC) A Hewlett-Parkard gas chromatograph Model 5830A (Avondale, PA, USA) equipped with a flame ionization detector was used for inverse gas chromatography measurements. The adsorbing agents used were packed in 300×6.4 mm o.d. stainless steel chromatographic columns and secured with glass fiber stoppers placed at both ends of the column. The amount of adsorbing agent used was determined by weighing the difference in the column before and after packing. The column was installed in the gas chromatograph (GC). Helium was used as the carrier gas at a flow rate of 27.27 mL/min at 24°C. The void volume of the column was determined using non-interactive air. The packed columns were pre-conditioned at 150°C under a stream of helium gas for 24 hr before the chromatographic studies were conducted. One hundred μL of air was injected into the column using a gas-tight syringe. This low volume of gas approached an infinite dilution of concentration inside the column. The operation conditions of the gas chromatograph were set as follows: oven temperatures for column: 50, 60, 70, and 80°C; inlet port: 150°C; and flame ionization detector: 250°C.

Calculations IGC was carried out by injecting a quantity of sorbate into the inlet of a column containing the stationary phase. The adsorption measurements were made using an elution chromatography procedure from which a data point in the isotherm (concentration of 1-MCP in column and partial pressure) can be calculated from symmetrical peaks. Small quantities of sorbate of approximately 3×10^{-7} g in 0.5 mL were injected into the column inlet port containing the stationary phase. By analyzing the corresponding elution peak, sorption parameters could be obtained using a calculation for this elution method (13).

Figure 1 shows a typical gas elution chromatogram with an additional small peak (air peak) early in the chromatogram under the assumption that the chromatographic system can be equilibrated instantaneously.

The relationship between the two peaks is

$$V_{ret}=t_{ret}\cdot F$$

$$V_{air}=t_{air}\cdot F$$

where, F is the correct gas flow rate (mL/sec) within the column, V_{ret} is the retention volume that is retained on the column, V_{air} is the retention volume that is not retained on the column corresponding to air, t_{ret} is the time that the gas is retained on the column, and t_{air} is the retention time that is not retained on the column (air).

The net retention volume, V_{net}

$$V_{net}=J\cdot F\cdot(t_{ret}-t_{air})=K_p\cdot V_s$$

where, K_p is the distribution coefficient; V_s is the volume of the stationary phase, t_{ret} is the retention time of the 1-MCP, t_{air} is the retention time of a non-sorbed species (air), and J is the James and Martin compressibility factor.

$$J=\frac{3}{2}\left(\frac{(P_i/P_o)^2-1}{(P_i/P_o)^3-1}\right)$$

where, P_i is the column inlet pressure, P_o is the column outlet pressure, and F is the correct gas flow rate (mL/sec).

$$F=f_m\cdot\left(\frac{T_{column}}{T_{room}}\right)\cdot\left(\frac{P_o-P_{H2O}}{P_o}\right)$$

where, T_{column} is the column temperature (K), T_{room} is the room temperature (K), P_o is the column outlet pressure, P_{H2O} is the water vapor saturation pressure at the room

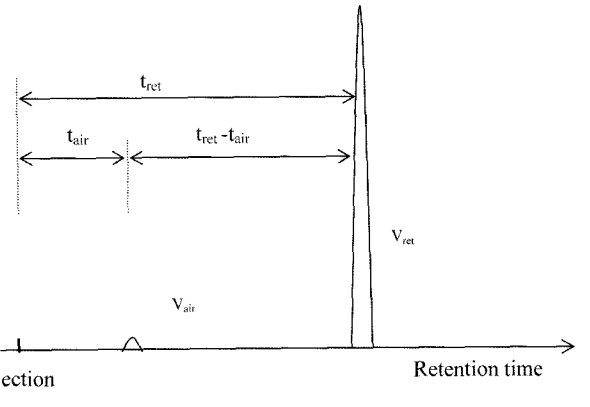


Fig. 1. The typical gas chromatogram of one component using IGC.

temperature because the gas becomes almost saturated after bubbling absorbed the aqueous solution of a soap-bubble meter, and f_m is the measured flow rate (mL/sec).

The corrected retention volume is then

$$V^o_{ret} = J \cdot t_{ret} \cdot F$$

$$V^o_{air} = J \cdot t_{air} \cdot F$$

Specific retention volume (V^o_g) is the amount of carrier gas required to elute a given amount of sorbate from a column containing a given weight of interacting stationary phase at 273K per gram of adsorbing agent (14). V^o_g is expressed in mL (STP) per gram of stationary phase.

The specific retention volume (V^o_g) is

$$V^o_g = \frac{V^o_{ret} - V^o_{air}}{W} \times \frac{273}{T_{column}} = \frac{JF(t_{ret} - t_{air})}{W} \times \frac{273}{T_{column}}$$

$$V^o_g = \frac{V_{net}}{W} \times \frac{273}{T_{column}}$$

$$V^o_g = \frac{K_p \cdot V_s}{W} \times \frac{273}{T_{column}}$$

where, V_{net} is the net retention volume, W is the adsorbing agent weight in the column (g), and T_{column} is the column temperature (K).

$$V^o_g = \frac{K_p}{\rho_s} \times \frac{273}{T_{column}}$$

where, K_p is the distribution coefficient defined as the ratio between the 1-MCP concentration in the stationary phase containing an adsorbing agent and in the mobile phase, respectively. ρ_s is the density of adsorbing agent on the stationary phase in the column.

Adsorption efficiency About 0.4 g of adsorbing agent was conditioned at 95°C for 24 hr to activate the binding sites on the surface. After cooling for two hours, samples of each test material were placed into empty 15 mL glass vials (Supelco Inc,). The vials were flushed with dry air and sealed immediately using screw caps and PTFE/silicon covered septa (Supelco Inc.). A known amount of 1-MCP was transferred through the septa and exposed to the surface of the adsorbing agents in dry air conditions.

After exposing 1-MCP gas to the surface of the adsorbing agents, 0.5 mL of deionized water was added to the glass vial to obtain an environmental condition of high humidity. After one hour, 40 μL of gas in the headspace was extracted from the sealed glass cell using a 100 μL gas tight syringe (Hamilton Co.).

The gas sample was injected into the gas chromatograph to quantitatively determine the amount of 1-MCP under dry air and high humidity conditions, respectively. Each sample analysis was performed in triplicate.

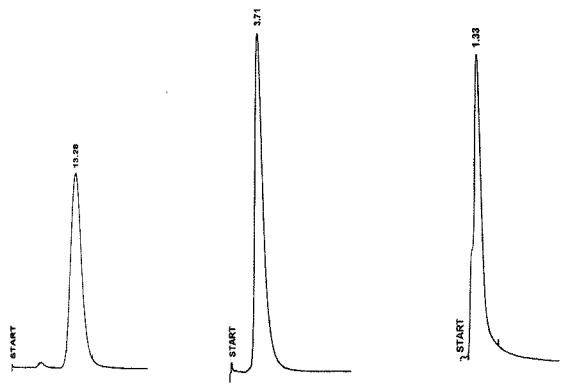
Results and Discussion

Chromatogram The peak profiles are represented as typical chromatograms using the inverse gas chromatography elution technique. Smaller amounts of 1-MCP resulted in longer retention times than with larger amounts. Peak shapes and retention times were dependent on the amount of 1-MCP injected. The adsorption of 1-MCP by silica gel, Tenax-TA, and activated clay at 80°C is shown in Fig. 2. The amount injected for silica gel, Tenax-TA, and activated clay was 9, 6, and 4 nmol, respectively. Chromatogram peaks of 1-MCP adsorption were not observed for the adsorbing agent activated carbon. This is probably because 1-MCP has too strong an affinity for these adsorbing agents. Activated carbon has a high affinity for organic liquids or vapors because of strong hydrophobic interactions with the carbon surface (15).

Sorption behavior and specific retention volume Sorption isotherms of 1-MCP adsorption versus 1-MCP partial vapor pressure were determined at 50, 60, 70, and 80°C. In order to determine the sorption behavior of 1-MCP gas at an infinite dilution concentration, results were obtained from the profile peaks of 1-MCP interaction with the adsorbing agents silica gel, Tenax-TA, and activated clay. Sorption behavior of 1-MCP molecules for silica gel, Tenax-TA, and activated clay are presented in Fig. 3.

Sorption isotherms of 1-MCP and their respective adsorbing agents were linear, that is, ruled by Henry's law. Silica gel had a much higher Henry's law constant (K_s) than Tenax-TA and activated clay. This implies that the amount of 1-MCP adsorbed on the silica gel, at the corresponding partial vapor pressure of 1-MCP, was greater than that for Tenax-TA and activated clay. An increase in temperature decreased the amount of 1-MCP absorbed on adsorbing agents.

Linear plots were derived from a least square fit of the log constant value (Ln K_s) versus the reciprocal of the absolute temperature (1,000/T, K^{-1}) for each adsorbing agent as shown in Fig. 4. The linear equations were as follows: Y = 5.5356X – 12.244 (silica gel); Y = 6.0074X



(a) Silica gel (b) Tenax – TA (c) Activated clay

Fig. 2. Adsorption of 1-MCP on (a) Silica gel, (b) Tenax-TA, and (c) Activated clay by inverse gas chromatography.

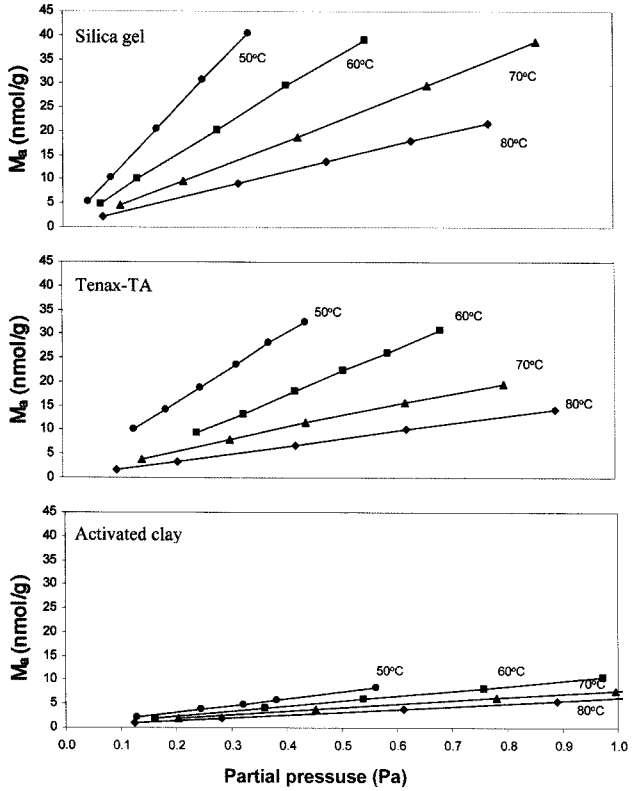


Fig. 3. Sorption isotherms of 1-MCP on silica gel, Tenax-TA, and activated clay at 50, 60, 70, and 80°C. M_a is the concentration of 1-MCP adsorbed in the column. Partial pressure is the partial pressure (Pa) of the 1-MCP vapor entering the gas chromatographic detector.

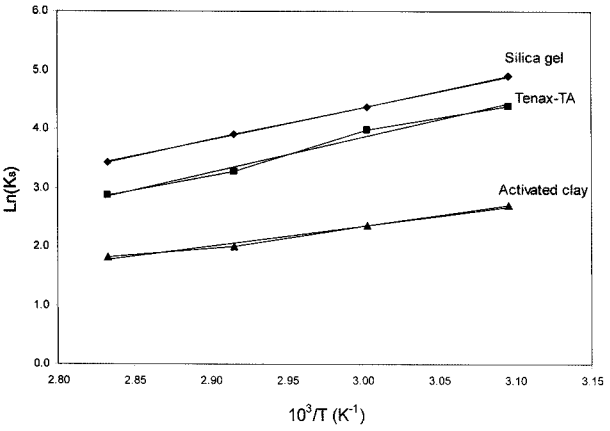


Fig. 4. Linear plot of the log constant value (Ln K_s) versus the reciprocal of the absolute temperature (10^3/T, K^{-1}).

– 14.159 (Tenax-TA); Y = 3.4438X – 7.9804 (activated clay), where X is 1000/T (K) and Y is the –Log (K_s). All correlation coefficients for the isotherms were higher than 0.98. They were used to predict the amount of 1-MCP gas adsorbed by the adsorbing agents (mol/g) at 23°C. K_s values at 23 °C were 640 nmol/g·Pa for silica gel, 460 nmol/g·Pa for Tenax-TA, and 40 nmol/g·Pa for activated clay. Gavara *et al.* (16) determined the sorption isotherms for four food aroma components on three polymeric

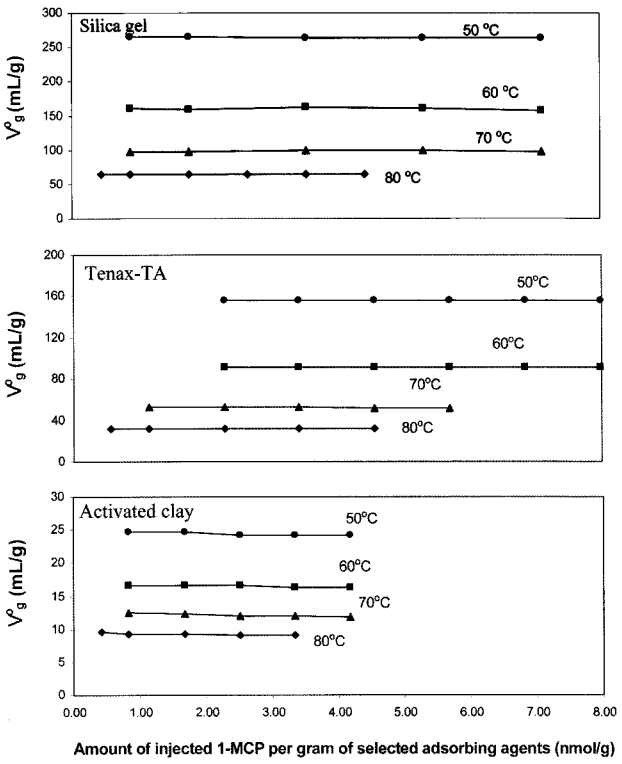


Fig. 5. Specific retention volume (V^o_g) for silica gel, Tenax-TA, and activated clay as a function of 1-MCP concentration at 50, 60, 70, and 80°C.

materials. The isotherms for both copolymers and three polymeric materials were linear in the low and high vapor pressure range.

Specific retention volumes (V^o_g) for each adsorbing agent at 50, 60, 70, and 80°C are presented in Fig. 5. Silica gel was found to strongly interact with 1-MCP compared to Tenax-TA and activated clay, based on the specific retention volume (V^o_g) for 1-MCP and their adsorbing agents based on binding site theory. The specific retention volume of 1-MCP gas with silica gel, Tenax-TA, and activated clay at 50°C was approximately 265, 156, and 25 mL/g, respectively.

These values indicate that 1-MCP gas has a stronger binding force with regard to the silica gel than the other adsorbing agents. The larger the V^o_g value is, the stronger the attraction between the sorbate and the stationary phase. V^o_g was not affected by the amount of 1-MCP injected. This assumes that 1-MCP levels are relatively low and are not saturating the available number of binding sites in the respective stationary phases. Figure 6 shows a straight line for Log V^o_g against 1/T between 50 and 80°C for 1-MCP and the respective adsorbing agents. At lower temperatures, lower kinetic energy was necessary to bind 1-MCP to the adsorbing agents' binding sites so that more binding sites could be occupied. Therefore, the specific retention volume for all adsorbing agents increased with decreasing temperature.

The V^o_g values of vinyl chloride monomer (VCM) for resin E and resin S of polyvinyl chloride polymers (PVC) at 55°C for studying the effect of polyvinyl chloride

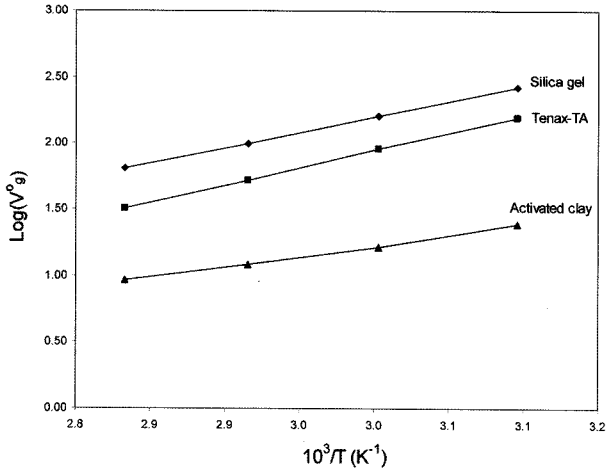


Fig. 6. Log specific retention volume (V^o_g) for 1-MCP as a function of temperature.

polymer structure, monomer level, and temperature on vinyl chloride monomer migration were approximately 12 and 0.13 mL/g, respectively (17). The VCM specific retention volume for resins was dependent on the concentration and temperature. These results show that the specific retention volume of 1-MCP gas for adsorbing agents represents a relatively higher value compared to those of water and VCM on PVC polymer. Delarue and Giampaoli (14) observed retention behavior using a linear plot of Log V^o_g vs. 1/T of aroma molecules on carbohydrate matrixes such as starches or maltodextrins, and evaluated the enthalpy of adsorption of these molecules using IGC. Gauthier *et al.* (18) studied the physicochemical modifications of cellulose using acyl chlorides having 5 and 16 carbon atoms. He found a linear relationship between Log V^o_g and 1/T between 50 and 140° C for *n*-octane on a C_{16}-CE IGC column. The slope of the linear line was used to determine the differential enthalpy adsorption value.

Distribution coefficient Distribution coefficient (K_p) values for each adsorbent at 50, 60, 70, and 80°C are given in Table 1. The K_p values for each adsorbent decreased with increasing the temperatures. An increase in temperature becomes the equilibrium of 1-MCP in the stationary phase containing an adsorbent and in the mobile phase, and thus K_p values decrease. The distribution coefficient (K_p) for 1-MCP on a stationary phase column containing silica gel adsorbent represents higher values than those for Tenax–TA and activated clay adsorbents. This is under the assumption that the stationary phase of silica gel adsorbent for 1-MCP has more active sites than those of other adsorbents.

Table 1. Distribution coefficient values of 1-MCP in selected adsorbent agents as a function of temperature

Temperature (°C)	Distribution coefficient (K_p)[1)]		
	Silica gel	Tenax-TA	Activated clay
50	187.83±0.54	46.12±0.07	12.17±0.14
60	117.52±1.22	27.87±0.04	8.44±0.09
70	74.61±0.47	16.39±0.05	6.41±0.16
80	49.94±0.02	10.36±0.10	5.04±0.10

[1)]Each value represents the average of triplicate tests at 5 different 1-MCP concentrations.

1-MCP adsorbing efficiency The efficiency of 1-MCP adsorption to the adsorbing agents was determined from the difference between the initial added 1-MCP, and that remaining in the headspace of the adsorbing material in a closed system at 23°C. Table 2 shows the adsorbing efficiencies for the different adsorbing agents. This result is in agreement with the distribution coefficient values and those obtained from IGC analyses comparing the 1-MCP adsorbing efficiencies of silica gel, Tenax-TA, and activated clay. However, in adsorbing efficiency experiments using a closed system, activated carbon (80/100 mesh) had an adsorbing efficiency of 98.86% in dried air, indicative of a strong affinity. This information strongly supports the result showing no chromatogram peaks of 1-MCP adsorption on activated carbon as determined by IGC. After treating with deionized water, silica gel showed a relatively lower adsorbing efficiency than that obtained with dry air. Activated carbon had a strong affinity for 1-MCP, even when treated with distilled water. Smísek and Cerny (19) found that activated carbon had a high affinity for organic molecules and other non-polar substances because most forms of activated carbon are non-polar. Zhou *et al.* (20) measured the adsorption equilibria of nitrogen on silica gel over a large range of temperatures. The transition of adsorption mechanisms from the sub- to the supercritical region is considerably different for the adsorption of gaseous nitrogen by activated carbon and silica gel.

Table 2. 1-MCP adsorbing efficiency of selected adsorbing agents in a closed system

Solid matrix	Average weight (g)	Adsorbing efficiency (%)[1)]	
		A dry air (0%RH)	After treating with distilled water
Silica gel	0.5023	88.57	70.56
Tenax-TA	0.4610	89.42	79.93
Activated carbon (20/40 mesh)	0.5072	98.01	98.17
Activated carbon (80/100 mesh)	0.4712	98.86	98.21

[1)]Adsorbing efficiency is the amount of 1-MCP in solid matrix×100/amount of 1-MCP applied in a closed system. The values are the results of duplicate analyses.

Activated carbon proved to be a very strong binding agent for 1-MCP due to its high adsorbing capacity. Activated carbon could not desorb 1-MCP molecules at conditions of higher humidity. However, 1-MCP gas held by the adsorbing agent must be released only under the conditions of high relative humidity found in packaged fresh produce, because most fresh produce continues to lose moisture into the storage or package atmospheres through transpiration. Thus, it is important to change the binding efficiency of 1-MCP on the adsorbing agent, depending on the relative humidity during storage. Silica gel is suitable for the binding and release of 1-MCP gas because it has high binding efficiency in dry air and a relatively lower binding efficiency of 1-MCP at conditions of high relative humidity.

In conclusion, this study shows that IGC can be a useful method to study the interaction between 1-MCP molecules and the surface of an adsorbing agent. The relationship between 1-MCP and adsorbing agents must be known in order to estimate the amount of 1-MCP needed under different environmental conditions with regard to other gases and moisture levels. The information presented in this work may be useful in the designing of methods to release 1-MCP molecules from the surface phase of the adsorbing agent into the headspace phase of the produce.

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