

# Effects of Sucrose, Guar Gum, and Carboxymethylcellulose on the Release of Volatile Flavor Compounds under Dynamic Conditions

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The effect of viscosity and thickener type (sucrose, guar gum, and carboxymethylcellulose) on dynamic flavor release was tested with model flavor solutions at two equiviscous levels. Dynamic flavor release was measured under simulated mouth conditions in an apparatus at 37 °C, with a shear rate of 100 s<sup>-1</sup>. The volatilized flavors were swept in a flow of helium gas into a mass spectrometer for selected ion monitoring chemical ionization. A plot of time versus ion abundance was recorded for each data set. The highly volatile compounds showed a large decrease in maximum ion abundance ( $I_{\max}$ ) as viscosity increased. Carboxymethylcellulose, guar gum, and sucrose solutions with a viscosity of 160 mPa s showed 36, 44, and 86% decreases compared to water, respectively, for the release of  $\alpha$ -pinene. Similarly, 1,8-cineole decreased 32, 40, and 70% and ethyl 2-methylbutyrate decreased 58, 63, and 94%, respectively. The less volatile compounds methyl anthranilate, vanillin, and maltol showed less of an effect. Thickened solutions of similar viscosity did not show the same flavor release, indicating that both viscosity and binding of flavors with the food matrix affect flavor release.

**Keywords:** *Viscosity; mass transfer; diffusion; volatility; flavor release; mouth; shear; Stokes–Einstein; thickeners; binding; hydrocolloid*

## INTRODUCTION

Consumption of a thick liquid often results in a perceived flavor different from that of a thin liquid. The mechanisms underlying viscosity-induced flavor changes have not been fully elucidated. Viscosity is defined as the internal friction of a fluid or its tendency to resist flow (Bourne, 1982) and is caused by the work necessary to overcome the frictional forces exerted by the dissolved molecules on the fluid and, in concentrated solutions, by entangling chains (Bohdanecky and Kovar, 1982). Several solute parameters affect the viscosity: molecular weight, molecular weight distribution, degree of hydration, extent of extra- and intermolecular interaction, radius of gyration, degree of substitution, and type of coil formation (Da Silva and Rao, 1992). Diffusion of flavor molecules is reduced as solution viscosity increases, as predicted by the Stokes–Einstein and Wilke–Chang equations (Wilke and Chang, 1955). The volatility of a flavor molecule may also be affected by the formation of barriers occurring in high-viscosity matrices and by specific binding interactions with the thickener. Binding interactions with carbohydrate-based thickeners are often due to adsorption, entrapment in microregions, complexation, encapsulation, and hydrogen bonding (Kinsella, 1989).

To develop a full understanding of food flavor, it is necessary to determine to what extent matrix alterations of food affect the binding, entrapment, diffusion, and release of flavor compounds and, in turn, how changing a food's composition influences flavor release

profiles. One important question is whether viscosity imparted by several different thickeners will give the same volatile flavor release. This information will help discern the importance of viscosity as opposed to specific thickener–flavor interactions. Published work shows support for both possibilities and will be discussed below.

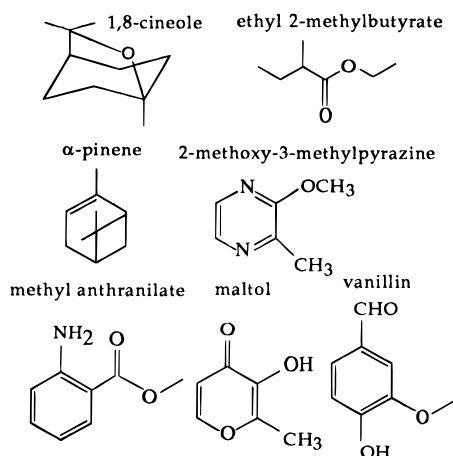
In the evaluation of viscosity effects, sweetness, rather than volatile flavor, has been mainly studied. Increases in solution viscosity have been shown to decrease sensory panel rated taste sensations (Vaisey et al., 1969; Moskowitz and Arabie, 1970; Kokini et al., 1982) with differences seen between thickeners (Pangborn et al., 1973; Paulus and Haas, 1980). However, the concept of flux is useful to taste and aroma. Fick's law states that flux is proportional to the diffusion coefficient, which itself is inversely proportional to viscosity. Hence, perceived sweetness intensity increases with increase in flux of the sweetener. Sweetness will decrease as viscosity increases, and solutions of equal viscosity should have equal flavor (Cussler et al., 1979). The coil overlap value,  $c^*$ , is a characteristic concentration for disordered or "random coil" polymer systems such as guar gum, in which individual polymer coils begin to overlap (Baines and Morris, 1987). This value is determined by noting the sharp break when concentration is plotted versus viscosity, as measured by rotational viscometers. This is associated with a marked increase in viscosity and a decrease in perceived flavor (Baines and Morris, 1988; Morris, 1987). Using flavored guar gum solutions, sensory panels found both flavor and taste to decrease with increasing viscosity above  $c^*$  (Baines and Morris, 1987). Consequently, Morris (1987) suggested that the decrease in flavor perception was due to decreased mixing as the polymer chains became obstacles to diffusion rather than to a direct binding of flavor molecules to the polymer.

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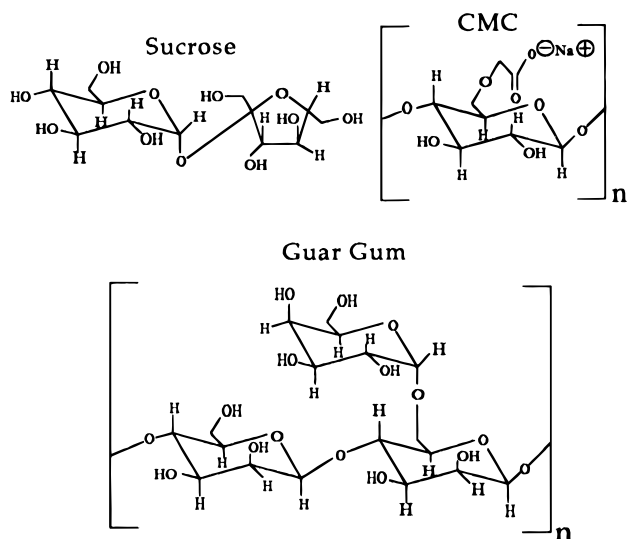
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**Figure 1.** Flavor compounds used in this study.



**Figure 2.** Thickeners used in this study: sucrose, CMC, and guar gum. For guar gum, D-galactosyl residues are irregularly distributed along the D-mannan backbone (Winter et al., 1984; McCleary et al., 1984).

Contrary to the view that no binding occurs, another study showed differences in the time-intensity flavor perception of  $\alpha$ -pinene, ethyl caproate, and cinnamic acid from equiviscous aqueous solutions containing different thickeners. The authors concluded that these differences were explained by a binding interaction (Malkki et al., 1993). Likewise, the reduced odor intensity of dimethyl sulfide caused by the addition of hydrocolloids was independent of hydrocolloid level and was explained by entanglement of disulfide with the hydrocolloid rather than viscosity (Pangborn and Szczesniak, 1974).

This experiment was designed to test the effect of viscosity on aroma release and to determine whether any decreases in aroma release were due to mass transfer alone or to odorant binding by the thickeners. Seven flavor compounds (Figure 1), comprising a range of chemical properties (Roberts and Acree, 1995), were tested for their volatility in carboxymethylcellulose (CMC), sucrose, and guar gum solutions (Figure 2) of the same measured viscosity. If the solutions thickened to a similar viscosity exhibited a similar flavor release, the limiting mechanism of volatile flavor release could be attributed to a reduction in mass transfer. A flavor binding interaction (occlusion, complexation, noncovalent forces, adsorption, etc.) would be the conclusion if flavor release was not solely viscosity dependent. While

the sucrose levels used in this study are much higher than would be found in most foods, it is interesting to compare different food components for their effect on volatile release.

Since human consumption of food involves placing food in the mouth and chewing, a dynamic flavor release apparatus (Elmore et al., 1996) was used. The device incorporated the elements of mastication and temperature regulation with instantaneous flavor release measurement.

## MATERIALS AND METHODS

**Experimental Samples.** Low- and high-viscosity solutions were made for each thickener. Both solutions of CMC sodium salt (low viscosity; BDH, Poole, Dorset) and the low-viscosity sucrose (BDH) solution were made by dissolving in 40 °C distilled water. The high-viscosity sucrose solution required additional heating to 60 °C. Solutions of high molecular weight guar gum (220 000 MW) (Sigma, St. Louis, MO) were prepared at room temperature. Table 1 shows the concentrations of the thickeners and the water activity of each. Water activity was determined as the average of three measurements at 37 °C, using a Hygroline Series HTC Thermoconstant test chamber (Rosemount, Irvine, CA).

The viscosities of CMC and guar gum were matched to the viscosity of sucrose at 37 °C using a Brookfield (Stoughton, Mass.) synchroelectric cone and plate viscometer with a 1.5° cone at shear rates from 1.15 to 230 s<sup>-1</sup>. The concentrations and viscosities are shown in Table 1. For sucrose and CMC solutions, the viscosities were constant throughout the measured shear rates. For shear thinning guar gum at 100 s<sup>-1</sup> shear rate, the low viscosity was set to be 12.5 mPa s and the high viscosity was extrapolated to be 160 mPa s.

The flavor compounds ethyl 2-methylbutyrate,  $\alpha$ -pinene, 1,8-cineole, 2-methoxy-3-methylpyrazine, vanillin, maltol, and methyl anthranilate (Aldrich, Milwaukee, WI) were dissolved in ethanol at 1250 mg/L. This stock solution was added to the thickener samples at 23 °C to give a final flavor concentration of 1.25 mg/L. The ethanol level present in the samples was at 0.1%, which would have little effect on the flavor volatility (Williams and Rosser, 1981). The samples were then incubated overnight at 37 °C in sealed bottles to avoid volatile loss, shaken, and analyzed by the dynamic flavor release apparatus.

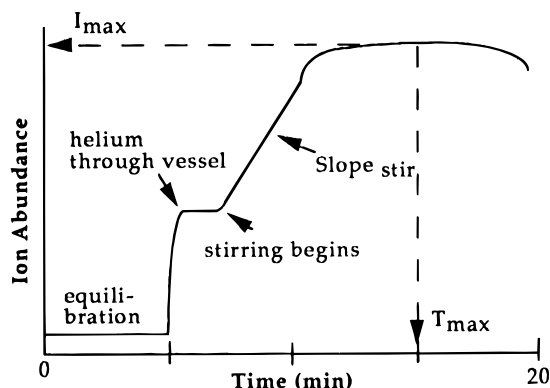
**Dynamic Flavor Release Measurement.** The dynamic flavor release curves of each of the flavor compounds were measured in triplicate using a constructed flavor release apparatus (Elmore et al., 1996). The samples (20 mL), incubated (37 °C) in a glass vessel, were stirred at a shear rate of 100 s<sup>-1</sup>, as calculated by the concentric cylinder equation (Bourne, 1982). Helium carrier gas at 30 mL/min flowed over the samples, sweeping volatiles into a Hewlett-Packard 5988A quadrupole mass spectrometer, via a jet separator (SGE, Ringwood, Australia). The protonated molecular ( $M + 1$ ) ions for each volatile compound were monitored in real time, using single ion monitoring, under chemical ionization conditions, allowing simultaneous measurement of the release of all of the volatile compounds in the thickeners. From the ion trace obtained for each volatile, the maximum intensity of the flavor release curve ( $I_{\max}$ ) was measured. A schematic of a typical release curve is shown in Figure 3. There is an increase in ion abundance as the helium begins to enter the flask (at 5 min) and also when stirring is initiated (at 7 min). A standard (flavors in water at 1.25 mg/L) was run at least once each day, and the values from the samples were related to the standard, to correct for variations in mass spectrometric detection. Approximately seven samples were run on each of four days within two weeks. Hence, the values for  $I_{\max}$  were relative to the standard (standard = 1.0).

**Statistical Analysis.** Analysis of variance was performed using SPSS (Chicago, IL) on values from the release curve to determine if flavor volatility was affected by thickener type

**Table 1. Viscosity, Concentration, and Water Activity Values for the Thickeners at 37 °C**

	CMC		sucrose		guar gum	
	low	high	low	high	low	high
shear rate (s <sup>-1</sup> )						
115	12.4 (0.1) <sup>a</sup>		11.9 (0.2)		12.7 (0.1)	
46	12.5 (0.1)		12.5 (0)		15.3 (0.2)	
23	12.1 (0.4)		12.8 (0.8)		16.8 (0.5)	
11.5	11.2 (0.3)	154 (8.0)	13.7 (2.1)	155.3 (4.0)	19.0 (1.0)	
5.75		149 (1.0)		157.7 (5.1)	24.3 (2.0)	837 (26)
2.3		149 (1.0)		155.0 (8.7)		1130 (25)
av viscosity	12.3 (0.5)	151 (3.0)	12.7 (0.8)	156 (1.5)	13 <sup>b</sup>	163 <sup>b</sup>
concn (% w/w)	0.5	1.84	51.9	68.9	0.25	0.75
water activity	1.0	1.0	0.94	0.83	1.0	1.0

<sup>a</sup> Values in parentheses are standard deviations. <sup>b</sup> Viscosity at 100 s<sup>-1</sup> extrapolated using ln shear rate vs ln viscosity graph.



**Figure 3.** Diagrammatic representation of flavor release curves obtained from the flavor release vessel.

and viscosity. The statistical significance of the difference between mean values was performed using Fisher's lsd test at  $\alpha = 0.05$ .

## RESULTS AND DISCUSSION

**Viscosity and Thickener Effects.** For the volatile flavor compounds to be detected by the mass spectrometer, they must be transported to the surface of the solution and then change phase by volatilization. As opposed to high-boiling compounds, low-boiling compounds will more easily volatilize on reaching the surface. It is thus the highly volatile compounds that are most affected by a change in viscosity. Increasing the viscosity of the solution can slow the flavor compound migration process by the formation of barriers.

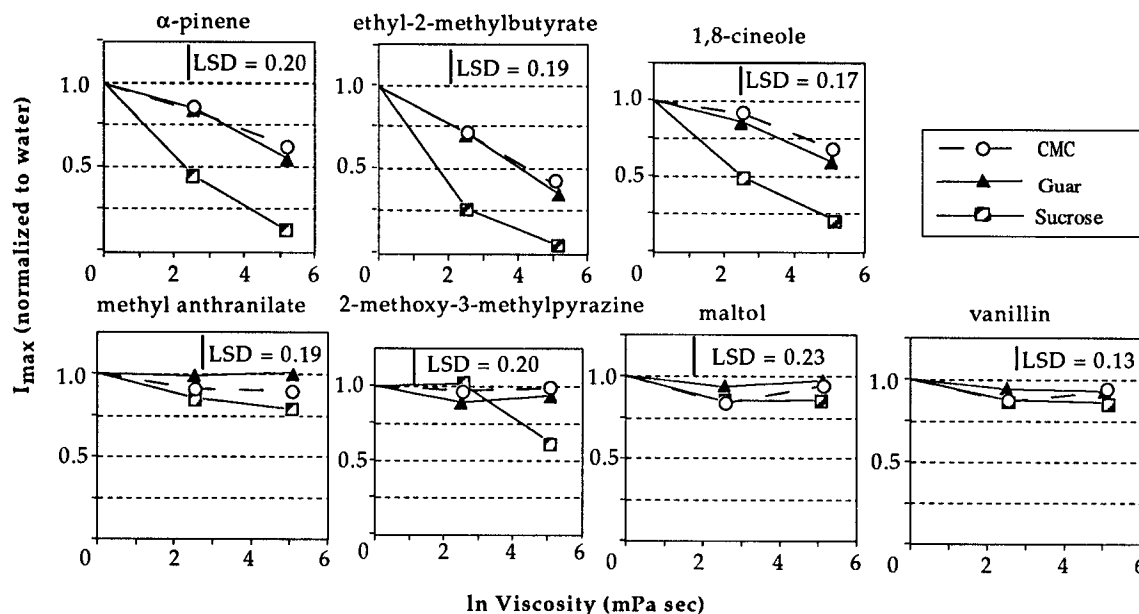
Figure 4 shows the effects of thickener type and viscosity on  $I_{\max}$ . The absolute values were not equated to concentration. However, the relative values are important because they give comparative information on thickeners. Thickener type, thickener level, and the particular flavor compound all affected the decrease in relative  $I_{\max}$ . Primarily the highly volatile flavor compounds showed an effect of gum thickener and level. The volatility of the flavor compounds in water (volatility rate constants) were  $\alpha$ -pinene (33 000), ethyl 2-methylbutyrate (1400), 1,8-cineole (230), 2-methoxy-3-methylpyrazine (14), methyl anthranilate (1), maltol (<0.1), and vanillin (<0.1) (Roberts and Acree, 1995).

Sucrose solutions showed a statistically significant ( $\alpha = 0.05$ ) lower flavor release than CMC and guar solutions for the most volatile flavors,  $\alpha$ -pinene, ethyl 2-methylbutyrate, and 1,8-cineole. These three most volatile compounds, all nonpolar, clearly showed a decreasing  $I_{\max}$  with increasing viscosity for all thickeners. However, the less volatile compounds, maltol, vanillin, and methyl anthranilate, did not show the large effect of viscosity or thickener type and were only

slightly released in all of the matrices tested. Although 2-methoxy-3-methylpyrazine had a moderate release in water, it did not show as large a decrease in volatility with thickeners as did the three most volatile compounds. Because 2-methoxy-3-methylpyrazine is a base, this could be an indication of a type of flavor binding interaction that occurs with the nonpolar volatile compounds. It may be destabilized by basic or nucleophilic action. The three thickened systems did not have the same flavor release, indicating that a binding interaction is probably present.

Morris (1987) predicted that there would not be an effect of CMC and guar gum addition on flavor volatility at levels below the coil overlap value ( $c^*$ ) but a large decrease above  $c^*$ . The concentrations of CMC and guar gum used in this study were above the reported  $c^*$  value for high molecular weight guar gum (0.2% w/w) (Baines and Morris, 1987). Morris also found that at levels above  $c^*$ , a difference existed in perceived sweetness and flavor intensity for the same intrinsic viscosity of these three gums: xanthan gum > CMC > guar gum. Similarly, time intensity curves of volatile flavors in equiviscous CMC and guar gum solutions showed that CMC had a higher total intensity and longer length of perception than guar gum (Malkki et al., 1993). Although not statistically significant, in the present study CMC did show a higher release of flavor compounds than guar gum.

**Volatility Dependence on Flavor Polarity.** Several earlier studies on the volatility of flavors in sucrose solutions appear to be in conflict, showing both depression and enhancement of volatility. However, when these studies are analyzed in terms of the polarity of the flavor molecule, a trend appears: the more polar flavors show an enhancement, while the nonpolar flavors show a depression of volatility. Indeed, a series of acetates (Kieckbusch and King, 1979) and ketones (Nawar, 1971) decreased in volatility in a sucrose solution as the carbon number increased. The volatility of the polar compounds, acetone (Nawar, 1971; Voilley et al., 1977), ethyl acetate, 2-propanol, diacetyl (Voilley and Bosset, 1986), isopentyl acetate (Bredie et al., 1994), ethanol (Chandrasekaran and King, 1972), and polar acetates (Kieckbusch and King, 1979), increased as the sucrose or glucose concentration was increased to about 60% w/v. This effect has been postulated to be due to a salting-out phenomenon as sugar binds the free water (Wientjes, 1968). In contrast, the headspace of nonpolar compounds, 2-heptanone, 2-heptanal (Nawar, 1971), butylbenzene (Massaldi and King, 1973),  $\alpha$ -ionone, and naphthalene (de Roos and Wolswinkel, 1994), decreased with added sucrose. Similarly, nonpolar limonene (Ahmed et al., 1978) showed an increase in threshold (decrease in volatility) with added sucrose and a de-



**Figure 4.** Effect of viscosity on the release of flavor compounds from model solutions of guar, sucrose, and CMC. There are significant effects of viscosity and thickener type ( $P < 0.0001$ ) for ethyl 2-methylbutyrate,  $\alpha$ -pinene, and 1,8-cineole and of thickener type ( $P < 0.05$ ) for methyl anthranilate.

crease in volatility with added glucose (Bredie et al., 1994) and sucrose (Massaldi and King, 1973).

In the present study, guar and CMC depressed the volatility of  $\alpha$ -pinene, ethyl 2-methylbutyrate, and 1,8-cineole yet did not statistically significantly affect the other compounds. The gums also showed a flavor release similar to each other as previously reported for sweetness intensity (Vaisey et al., 1969). The following reports indicate that in guar and CMC solutions, release of highly volatile nonpolar odorants was reduced, yet release of polar odorants was increased, as compared to water. The nonpolar odorants dimethyl sulfide (Pangborn and Szczesniak, 1974), ethylbenzene, styrene, limonene, ethyl sulfide (Schirle-Keller et al., 1992), hexanal, and hexanone (Franzen and Kinsella, 1974) did indeed show a decrease in volatility from water with added CMC or guar gum. Conversely, polar odorants acetaldehyde and diacetyl (Pangborn and Szczesniak, 1974; Schirle-Keller et al., 1992) increased in volatility with added gums. However, exceptions exist: polar butyric acid decreased with added CMC (Pangborn and Szczesniak, 1974) and nonpolar octanone and ethyl heptanoate increased with an added CMC/MCC mixture (Schirle-Keller et al., 1992). These exceptions are lower in volatility; thus, it is probably only the highly volatile odorants that show the large effect of gums.

**Theory of Flavor Volatility Reduction.** From mass transfer theory (Crank, 1975), an increase in matrix viscosity would decrease the amount of flavor released. From the Stokes-Einstein relationship and assuming a constant diffusion rate, the amount of flavor released will be inversely proportional to the matrix viscosity and independent of matrix composition. Although the diffusion rate will be dependent on structure, changes in the rate should be less than the changes observed in the present experiments. In calculations from data for chromate diffusion through agarose (Belton and Wilson, 1982), increasing agarose concentration from 1 to 3% w/w gave a decrease of 10–12% in the rate of diffusion. This discrepancy is less than the 25–75% decreases observed in this experiment. This experiment indicates that although, in general terms, mass transfer as predicted by the Stokes-Einstein theory does occur,

other mechanisms are also important. The reduction in release in the presence of sucrose could be an example of steric hindrance, whereas the effect of flavor compound polarity shows flavor-matrix interaction.

Sucrose was present at a 40–200 times greater percentage (w/w) to give the same viscosity as the guar and CMC solutions. As seen in Table 1, the water activities of the sucrose solutions were reduced from 1.0 by the addition of sucrose. CMC and guar gum are both random coil long-chain polysaccharides which impart high viscosity at low concentration (Morris et al., 1981) and had water activities of 1.0. For  $\alpha$ -pinene, ethyl 2-methylbutyrate, and 1,8-cineole, sucrose solutions had the lowest water activity and also had the lowest release. However, for these compounds, the high-viscosity CMC and guar gum showed decreases in release from water yet no change in water activity. Water activity may play a part in determining flavor release; future research is needed to fully understand its importance.

The higher sucrose solution is above the saturation point of 67% solids (Richardson et al., 1987). As sucrose solutions are concentrated, they become less amorphous and more glassy or crystalline in state (Slade and Levine, 1991). A solid-liquid state diagram showed that the most viscous sucrose solution used in this study was amorphous but was almost at the border between amorphous and crystalline (Slade and Levine, 1991). Trapping of the volatiles could occur due to partial crystalline formation. A theory that volatiles remain entrapped in amorphous microregions of hydrogen-bonded sucrose molecules was based on studies of volatile retention in freeze-dried carbohydrates (Flink and Karel, 1970). The amorphous free volume is much less in the sucrose solutions than in the other thickeners. For small molecules in a fully amorphous system, the permeability of the molecules has been related to the amorphous free volume (Karel et al., 1975). The decrease in amorphous free volume could be the limiting factor, causing the decrease of flavor volatility in the sucrose solutions due to a decrease in permeability. Indeed, a decrease in the water mobility as sucrose concentration increased in solution has been published

containing reports of causative sucrose–sucrose interactions (Richardson et al., 1987).

Inclusion complexes, such as hydrophobic microregions, may be entrapping the nonpolar aroma compounds. Hydrogen bonding of water with the equatorial hydroxy groups has been well established and could result in hydrophobic regions (Franks, 1983). Pair interactions of sucrose have been computed to be favorable and could form a hydrophobic region (Kojak et al., 1968). Sucrose also bears an  $\alpha$ -glycosidic linkage and can move to form a hydrophobic region where the nonpolar flavor molecules aggregate. The presence of intramolecular hydrogen bonding of sucrose molecules in solution (McCain and Markley, 1986; Stevens and Duda, 1991; Christofides et al., 1986; Davies and Christofides, 1987) as well as conformational flexibility of the molecule (Duker and Serianni, 1993; Tran and Brady, 1990) has been well documented.

Similarly, for CMC and guar gum, nonpolar regions could form as the polymer folds to form junctions (Suggett, 1975). CMC was found to be coil-like in its behavior, with significant molecular entanglements (Clark, 1992). These could be similar to the hydrophobic inclusion complexes that form with flavors for the carbohydrates amylose (Rutschmann and Solms, 1990) and cyclodextrin (Reineccius, 1989). The presence of sucrose at such a higher percentage (w/w) in solutions as compared to the gums in this study implies that more of these hydrophobic complexes would be present and could account for the greater depression of volatility by sucrose.

**Conclusions.** Flavor release is complex and several mechanisms can occur: mass transfer, matrix structural hindrance, and flavor–matrix interactions. As the viscosity increased, the flavors with high volatility ( $\alpha$ -pinene, ethyl 2-methylbutyrate, and 1,8-cineole) showed a reduction in flavor release. The less volatile flavors, methyl anthranilate, vanillin, and maltol, did not show a significant decrease in volatility with increasing viscosity. Adding thickeners to a system will affect the release of flavor compounds to different extents and therefore upset the balance of the released flavor profile. Also, the effect will differ depending on the thickener used. The equiviscous solutions of CMC, guar gum, and sucrose did not show the same flavor release: the addition of sucrose to water depressed flavor volatility to a much greater degree than did the addition of CMC and guar solutions of the same viscosity. CMC had a slightly higher flavor release than guar gum. Inclusion complexes may be present in the sucrose solutions for hydrophobic molecules. Consideration of these factors may aid in understanding flavor release in the formulation of foods.

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