

Food Chemistry 72 (2001) 363-368

Food Chemistry

www.elsevier.com/locate/foodchem

The effect of sugars and pectin on flavour release from a soft drink-related model system

A. Hansson^{a,*}, J. Andersson^b, A. Leufvén^a

^aThe Swedish Institute for Food and Biotechnology (SIK), PO Box 5401, S-402 29 Göteborg, Sweden b Danisco Sugar AB, PO Box 32, S-232 21 Arlöv, Sweden

Received 18 April 2000; received in revised form 25 July 2000; accepted 18 August 2000

Abstract

Three types of sugar and high-methoxyl pectin at different concentrations were added to a soft drink-related model system consisting of water and six flavour compounds. The addition of these ingredients contributes to changes in viscosity and water activity, which in turn affects the release of the flavour compounds to the gas phase above the soft drink. In the study, a higher concentration of sucrose and invert sugar increased the release of five flavour compounds, probably owing to a so-called "salting-out" effect. Starch syrup at a concentration of 60% increased the amount of three of the flavour compounds released and would probably increase the release of more compounds at a higher concentration. When pectin was added to the system, it was seen that viscosity does not influence release of the flavour molecules, but that the kind of stabiliser used is more important. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Flavour release; Viscosity; Pectin; Sugars; Water activity; Soft drink

1. Introduction

Sweeteners are among the most important ingredients in sugar confectionery and soft drinks. They contribute to a wide range of functionalities influencing, for example, sweetness, texture, microbiological stability, and colour. Some of the most commonly used sugars and sugar-containing ingredients in foods are sucrose, invert sugar, and glucose syrups. Sucrose occurs naturally as a product of polymerisation of glucose and fructose by glycosidic linkage. Hydrolysis of sucrose results in invert sugar, which is a mixture of equal amounts of glucose and fructose (Chinachoti, 1995). Glucose syrup is formed by a more or less complete hydrolysis of starch, leading to mono- and oligosaccharides. Recently, some investigations of sweetness-flavour interactions in soft drink model systems were carried out (Nahon, Navarro y Koren, Roozen & Posthumus, 1998; Nahon, Roozen & de Graaf, 1998). Nahon, Navarro y Koren et al. (1998) showed that an increase (from 0 to 60% w/v) in the sucrose concentration in a

solution leads to an increased release of the flavour compounds with short gas chromatography/flame ionisation detection (GC/FID) retention times, and a decreased release of the compounds with longer retention times.

Polysaccharides, or hydrocolloids, are used as thickeners, stabilisers and gelling agents. In soft drinks, hydrocolloids are sometimes used for thickening, as well as to improve "mouth feel", mask flavours, and aid in carbonation retention (Dziezak, 1989). In soft drinks with a cloudy nature, they can also be used as densityadjusting agents and prevent precipitation of the cloud. Hydrocolloids may influence the rate and intensity of flavour release through a physical entrapment of flavour molecules within the food matrix, or through a specific or non-specific binding of flavour molecules (Carr, Baloga, Guinard, Lawter, Marty & Squire, 1996).

Pectin is one of the most important gelling polysaccharides in food, and is mainly extracted from citrus peel, apple pomace (King, 1992) or lime peel (according to Hercules Copenhagen A/S, Lille Skensved, Denmark). Some investigations on the effect of pectin on flavour release have been carried out by sensory and chemical analysis; Lundgren et al. (1986) found that the average perceived intensities of aroma, flavour, sweetness

^{*} Corresponding author. Tel.: +46-31-335-13-27; fax: +46-31-83- 37-82.

E-mail address: ahn@sik.se (A. Hansson).

and sourness from an orange-flavoured pectin gel decreased with raised pectin levels, while firmness increased. Guichard, Issanchou, Descourvieres and Etievant (1991) showed that the addition of pectin to jam not only modifies the oral consistency, but may also cause a decrease in both taste and flavour intensities. The observed decrease in flavour intensity in their study was probably related to a slower diffusion resulting from the small molecules being trapped between the pectin chains in the gel. Roberts, Elmore, Langely and Bakker (1996) investigated the effect of sucrose, guar gum and carboxymethyl cellulose on the release of six different flavour compounds. They found that as viscosity increased, flavours with high volatility showed reductions in flavour release while the less volatile compounds were not affected. They also found that both the viscosity and the binding of flavours with the food matrix affect flavour release.

The aim of our study was to examine how the addition of three kinds of sugars and high-methoxyl pectin, all at different concentrations, affects the release of six different flavour compounds, viz. isopenthyl acetate, ethyl hexanoate, cis-3-hexenyl acetate, linalool, L-menthone and limonene, from a soft drink-related model system. Different sugars (sucrose, invert sugar, and glucose syrup) interact with water to a greater or lesser degree, giving rise to different water activity values, and may therefore affect the release of flavour compounds in various ways (Godshall, 1997). Pectin was added to the model system, at levels corresponding to the viscosity of the samples, with different concentrations of sucrose. The objective was to find out whether viscosity has any effect on flavour release. The methods used were static head space and GC/FID. The viscosity was measured with a Bohlin rheometer.

2. Experimental design

The soft drink model system basically consisted of water and a non-commercial tutti-frutti flavour (donated by Danisco Cultor, Aarhus, Denmark). The flavour contained several compounds (24 molecules) with different properties, all dissolved in polypropylene glycol. Six of these (Table 1) were investigated in this study since they were the best separated on the GC column and therefore most suitable for quantification. The tuttifrutti flavour was mixed with de-ionised water at 0.1% (w/w) . Thereafter, 10 ml of the mixture was placed in a gas-tight vial (21 ml). The sample was equilibrated for 1 h at 30° C and the amount of flavour compounds in the gas phase was determined. At equilibrium, 500 μ l of the headspace was drawn from the vial with a gas-tight syringe and injected into a gas chromatographic column. The flavour compounds were quantified and identified by GC/FID and gas chromatography combined with mass spectrometry GC/MS, using an external standard (dodecan in ethanol). The analytical instrumentation consisted of a Hewlett Packard gas chromatograph HP 5890 equipped with a nitroterephtalic acid modified polyethylene glycol phase (DB-FFAP) 30 m \times 0.32 mm ID 0.25 µm column (J&W Scientific, Cologne, Germany), a Finnigan Incos 500 mass spectrometer and a flame ionisation detector. The temperature programme used was $40(5)-220(10)$ 4/min.

2.1. Water activity

The effect of water activity on flavour release was investigated by adding different concentrations of sucrose (5, 10, 20, 40 and 60% w/w), invert sugar (10, 20, 40 and 60% w/w) and glucose syrup (DE 40, glucose 15% w/w, maltose 12.3% w/w, maltotriose 9.2% w/w, higher sugars 48% w/w) (5, 10, 20, 40 and 60% w/w) to the soft drink model system. The tutti-frutti flavour was added in a concentration of 0.1% (w/w) to the sugarwater solution. All sugars and syrups were supplied by Danisco Sugar AB, Arlöv, Sweden. Water activity was measured with a water activity instrument (AquaLab, Decagon Devices, Inc., Washington, DC, USA).

2.2. Viscosity

An extra slow-set, high-ester pectin standardised with sugars, GRINDSTEDTM Pectin CF 120 (Danisco Cultor, Aarhus, Denmark), was added to the model system to study the effect of viscosity changes on flavour release. The tutti-frutti flavour was added in a concentration of 0.1% (w/w) to the pectin-water solution. The pectin was added to pre-heated water containing 0.4% Na-citrate and mixed thoroughly with a food processor. Since for gelling, the pectin used requires a pH value of ≤ 3.7 and a sugar content of 60%, it only increased the viscosity and did not form a gel. The levels used (0.05, 0.1, 0.25, 0.7 and 2.5%) corresponded to the viscosities given by the different levels of sucrose $(5, 10, 10)$ 20, 40 and 60%). The viscosity of the different mixtures was measured with a Bohlin VOR rheometer (Bohlin Reologi AB, Lund, Sweden). Measuring parameters used were measuring system C25 (cup and bob) with torsion bar 4.25 g/cm. The shear rate used was $92.1 s^{-1}$, based on the fact that chewing/swallowing corresponds to a shear rate of $10-10^2$ s⁻¹. Initial equilibrium time was 60 s, measurement delay time 15 s, and integration time, 5 s.

3. Statistical analyses

Student's *t*-test and the twosampletest with equal variance (Excel 97) were used to analyse individual differences between samples. The significance level was expressed as P-values.

Table 1

Concentrations of the measured flavour compounds in the "tutti-frutti" mixture, and their hydrophobic fragmental constants (log P), calculated by a method described by Rekker (1977)

Flavour compound	Concentration $(\%)$ w/w ^a	log P	$log P$ (literature values)
Isopentyl acetate	0.40	2.12	
cis-3-Hexenyl acetate	0.10	2.63	
Ethyl hexanoate	0.12	2.76	2.76 (Nahon, Navarro y Koren et al., 1998)
L-Menthone	0.20	2.81	2.83 (Gunning, 2000)
Linalool	1.20	3.06	
Limonene	0.10	4.28	4.35 (Gunning, 2000)

^a Concentration of the flavour compounds in the polypropylene glycol mixture (%) w/w.

Fig. 1. The effect of three different types of sugars (\bullet = invert sugar, \blacksquare = sucrose, \blacktriangle = glucose syrup) on release of six flavour compounds.

4. Results and discussion

An increase in the concentration of sucrose (from 20 to 60% w/w) was shown to significantly $(P<0.05)$ increase the release of isopenthyl acetate, ethyl hexanoate, cis-3-hexenyl acetate, linalool and L-menthone to the gas phase above the soft drink (Fig. 1). This was probably due to a "salting-out" effect of sucrose (Voilley, Simatos & Loncin, 1977) whereby sucrose interacts with water, increasing the concentration of flavour compounds in the remaining "free" water. There was, however, no significant change in the release of limonene in response to the increased sucrose concentration. The release of limonene was high, irrespective of the sucrose concentration, because of the strongly nonpolar nature of this compound. To define the hydrophobicity of a flavour compound, $log P$ -values can be used, with negative values indicating hydrophilic compounds. The log P -values for the different flavour compounds were calculated following a method described by Rekker (1977) and can be seen in Table 1.

An even more pronounced increase in the release of the above five flavour compounds was shown when invert sugar was added to the soft drink model system at different concentrations (20-60% w/w; Fig. 1). Invert sugar is a mixture of equal amounts of glucose and fructose formed by hydrolysis of sucrose. This means that when invert sugar is added at the same concentrations as sucrose, more water molecules are structured, giving rise to less free water. The flavour compounds are therefore even more concentrated in the available free water and are more easily released to the gas phase. As with sucrose, there was no increase in the release of limonene, probably owing to its non-polar nature.

Glucose syrup at a concentration of 60% w/w was shown to significantly increase the release of ethyl hexanoate ($P < 0.05$), L-menthone ($P < 0.05$) and cis-3-hexenylacetate ($P < 0.01$). Likewise, linalool had a tendency to be released at raised levels if 60% glucose syrup was added; however, neither isopenthyl acetate nor limonene were affected by the addition of glucose syrup (Fig. 1). Glucose syrup is formed by hydrolysis of starch and contains larger molecules than does sucrose. Therefore, it has fewer binding sites for water when used at the same concentration as sucrose. More free water is thus available, decreasing the concentration of the flavour molecules in the water, reducing the release to the gas phase. The different concentrations of the sugars can be directly related to water activity values. Low concentrations of each of the sugars give a high water activity value, and vice versa (Fig. 2). A concentration of 60% glucose syrup has a water activity value corresponding to that of a concentration of about 55% of sucrose and 45% of invert sugar. The increased release of the named flavour compounds in response to the addition of sugar was brought about by a decrease in water activity, as exemplified by *cis*-3-hexenyl acetate in Fig. 3. Although the regression coefficient ($R^2 = 0.7811$) for *cis*-3-hexenyl acetate) is not significant in a strict sense there is, however, a trend showing that water activity correlates with the flavour concentration in the gas phase. If glucose syrup is added at a higher concentration, thus leading to a lower water activity value, there will probably be an increase in the release of isopenthyl acetate and linalool as well.

The addition of pectin at a level of 2.5% caused a decrease in the release of limonene, as compared with the samples containing smaller amounts of pectin (0.7, 0.25, 0.10 and 0.05% ; Fig. 4). The pectin used in this study was HMP, with which, hydrophobic interactions between methyl groups are important for the gelling process. More than half of the hydrophilic carboxylic groups are changed into hydrophobic ester groups, which have a tendency to turn against each other to reduce the contact with the polar water molecules (Parbo, 1997). Even though no gel is formed, pectin molecules stretch out and align with sections of other molecules, forming micelles that are more hydrophobic

Fig. 2. Water activity in relation to concentrations of different types of sugars.

Fig. 3. Effect of water activity on release of cis-3-hexenyl acetate.

Fig. 4. Effect of viscosity on release of six flavour compounds.

since with them, bound water is replaced by intermolecular hydrogen bonds (Chinachoti, 1995). Since limonene is the most non-polar compound, it may be captured in the hydrophobic parts of the pectin solution. None of the other compounds were affected when pectin was added to the soft drink.

An increase in pectin concentration, however, changes the pH value in the solution. In the sample containing pectin at 2.5%, the pH value was 4.9, and pH increased successively to 7.3 as the amount of pectin was decreased. This could perhaps also explain why there was a decrease in the release of limonene when the largest amount of pectin was added to the solution. However, this phenomenon will need to be investigated further.

The soft drink model system containing 60% sugar showed an increasing effect on the release of all flavour compounds except limonene. In the model system containing 2.5% pectin limonene was the only compound affected, despite the fact that this system had the same viscosity as the solution containing 60% sucrose. However, the water activity was not affected when different concentrations of pectin were added to the model system, while the opposite was true when different concentrations of sugar were added. This indicates that not viscosity but water activity affects flavour release, as well as the type of polysaccharide used.

5. Conclusions

In these experiments, the addition of sucrose, invert sugar (20 -60%) and glucose syrup (60%) significantly $(P<0.05)$ increased the release of the most polar flavour

compounds, probably owing to a "salting-out" effect. When pectin was added at concentrations corresponding to the viscosity of the samples containing sucrose, the effect on flavour release was not the same. Only at the highest pectin concentration (2.5%) a decrease in release of limonene was seen. No effect on the release of the other compounds was seen even at the highest concentration. This shows that viscosity has no effect on flavour release, and that the water activity and the type of stabiliser used are what is actually important.

References

- Carr, J., Baloga, D., Guinard, J.-X., Lawter, L., Marty, C., & Squire, C. (1996). The effect of gelling agent type and concentration on flavour release in model systems. In R. J. McGorrin, & J. V. Leland, Flavour-Food Interactions (pp. 98-108). Washington, DC: American Chemical Society.
- Chinachoti, P. (1995). Carbohydrates: functionality in foods. American Journal of Clinical Nutrition, 61, 922-929.
- Dziezak, J. D. (1989). Ingredients for sweet success. Food Technology, $93 - 116$
- Godshall, M. A. (1997). How carbohydrates influence food flavor. Food Technology, 51, 63-67.
- Guichard, E., Issanchou, S., Descourvieres, A., & Etievant, P. (1991). Pectin concentration, molecular weight and degree of esterification: influence on volatile composition and sensory characteristics of strawberry jam. Journal of Food Science, 56, 1621-1627.
- Gunning, Y. M., Parker, R., Ring, S. G., Rigby, N. M., Wegg, B., & Blake, A. (2000). Phase behavior and component partitioning in low water content amorphous carbohydrates and their potential impact on encapsulation on flavours. Journal of Agricultural Food Chemistry, 48, 395-399.
- King, K. (1992). Pectin: an untapped natural resource. Food Science & Technology, 7, 147-152.
- Lundgren, B., Pangborn, R.-M., Daget, N., Yoshida, M., Laing, D. G., McBride, R. L., Griffiths, N., Hyvönen, L., Sauvageot, F., Paulus, K., & Barylko-Pikielna, N. (1986). An interlaboratory study of firmness, aroma, and taste of pectin gels. Lebensmittel-Wissenschaft & Technologie, 19(87-88), 66-75.
- Nahon, D. F., Navarro y Koren, P. A., Roozen, J. P., & Posthumus, M. A. (1998). Flavour release from mixtures of sodium cyclamate, sucrose and an orange aroma. Journal of Agricultural Food Chemistrys, 46, 4963-4968.
- Nahon, D. F., Roozen, J. P., & De Graaf, C. (1998). Sensory evaluation of mixtures of sodium cyclamate, sucrose and an orange aroma. Journal of Agricultural Food Chemistry, 46, 3426-3430.
- Parbo, H. (1997). Carbohydrater sukker og stivelse. In: H. Parbo, Hydrokolloider holder maden i form (pp. 10-19). Copenhagen: Kemi Forlaget og Henrik Parbo.
- Rekker, R. F. (1977). The hydrophobic fragmental constant. Its derivation and application; a means of characterizing membrane systems. In: Nauta, W. T., & Rekker, R. F., Pharmacochemistry, Library (vol. 1, pp. 39-70). Amsterdam: Elsevier Scientific Publishing.
- Roberts, D. D., Elmore, S., Langely, K. R., & Bakker, J. (1996). Effects of sucrose, guar gum and carboxymethylcellulose on the release of volatile flavour compounds under dynamic conditions. Journal of Agricultural Food Chemistry, 44, 1321-1326.
- Voilley, A., Simatos, D., & Loncin, M. (1977). Gas phase concentrations of volatiles in equilibrium with a liquid aqueous phase. Lebensmittel-Wissenschaft & Technologie, 10, 45-49.