Volatile Changes during Dehydration of d'Agen Prunes

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Solid phase microextraction (SPME) was used in conjunction with GC-MS to monitor the changes in some major volatile flavors during drying of plums. The aroma profile was significantly modified during the process and substantial loss of the original volatile flavors was observed. The generation of some compounds was shown to be due to the thermal decomposition of carbohydrates. This paper discusses the importance of aroma profiling in detecting the progress of the chemical (degradation) reactions and identifying marker volatiles in quality control of the product.

Keywords: *Prunes; volatiles; dehydration*

INTRODUCTION

During drying of plums (*Prunus domestica* L.) there are a number of chemical degradation reactions that may influence the aroma of the final product. In prunes certain chemical changes are desirable to give the unique "prune" flavor. However, there is a potential for too much chemical change leading to loss in flavor, and nutritive and commercial value. Better understanding of these reactions would provide a means of optimizing the drying process in relation to product quality.

Carbohydrates are very important to the quality of dried prunes. In plums, there are four major simple carbohydrates: glucose, fructose, sucrose, and sorbitol (Forni et al., 1992; Wilford et al., 1997). The most important chemical reactions affecting carbohydrates during drying are acid hydrolysis, Maillard reactions, and caramelization. These reactions have a decisive influence on the quality of the dried product and have been shown to occur during drying of plums depending upon the drying temperature and pH of the fruit (Price et al., 1997; Wilford et al., 1997). Other drying conditions (i.e., drying-air velocity and humidity) have also been found to affect these reactions (Sabarez, 1998; Price et al., 1997**)**. In previous work (Wilford et al., 1997), the changes in amounts of simple carbohydrates (fructose, glucose, sucrose, and sorbitol) in d'Agen plums during drying were studied. It was found that the changes in sugar content could be correlated with the possible onset of these particular sugar degradation reactions. With a better knowledge of these reactions, the operation of drying should therefore be expedited as much as possible without causing an intolerable degree of degradation. It is of considerable interest to monitor the changes of volatile flavors during drying to further understand the underlying mechanisms and onset of these degradation reactions of the sugars. This was the major motivation of the current paper.

Thermal degradation often leads to a wide spectrum of flavor compounds being produced depending upon the type of reactions (Hurrell, 1982). Several studies of variation in volatile profile of plums and prunes as influenced by thermal processing have been published. Ismail et al. (1977) studied the aroma of canned plums and found benzaldehyde, nonanal, and 2-furancarboxaldehyde as the major constituents. It was suggested that these three compounds probably arose from thermal treatment of the fruits, either by activation of enzymatic degradation of precursors at the first stage of heating or by chemical degradation of sugars and amino acids. Crouzet et al. (1990) showed that sugar degradation reactions were implicated as observed by the presence of furfural, methylfurfural, acetylfuran, and furfuryl alcohols in dried prunes. However, there has been little work on monitoring the changes of the volatile constituents of foodstuffs during drying. The recent work of Nijhuis et al. (1998) on aroma profiling during hot air-drying of mushrooms provides one relevant example. In particular, no information has been found on aroma profiling during drying of d'Agen prunes.

The aim of this paper was to measure any major changes in the volatile profile of d'Agens plums during drying. Changes in the volatile constituents were followed to establish avenues of detecting the progress of chemical degradations occurring during drying. The investigation used established SPME headspace techniques (Arthur et al., 1992; Potter and Pawliszyn, 1992) in conjunction with gas chromatography−mass spectrometry (GC-MS). The results are used to highlight the presence and onset of particular degradation reactions during drying.

EXPERIMENTAL PROCEDURES

Sample Preparation for Analysis. Flavor component analyses were carried out on fresh and dried d'Agen plums. Dried samples were prepared by drying plums using a computer-controlled experimental drying setup described elsewhere (Sabarez, 1998; Price et al., 1997). Each drying run was carried out past the normal moisture content of commercial dried prunes (i.e., 18−20% dry basis) in order to study the entire drying process. Simulated commercial drying conditions were used: 80 °C air temperature with a relative humidity of 35% and air velocity of 5 m/s.

A comparison of several methods of sample preparation was undertaken to optimize the extraction process of volatile components. Volatile headspace analysis of whole and mashed plum samples was examined. A glass tube fitted with a rubber septum which could accommodate three whole plums was used, while for the mashed samples 10 plums for each test

10.1021/jf990553x CCC: $19.00
Published on Web 04/22/2000

Table 1. Identified Volatile Components in Fresh Whole and Blended d'Agen Plums and Qualitative Changes in the Volatile Components of Plums during Drying at 80 °C (Rh = 3%; V = 5 m/s)

compound	retention time (min)	fresh plums		drying time (h)								
		whole	blended	1	3	5	7	9	11	13	15	18
hexanal	5.50	not found[a]	1.0[b]	nd[c]	nd	nd	nd	nd	nd	nd	nd	nd
2-hexenal	7.10	not found	1.0	nd	nd	nd	nd	nd	nd	nd	nd	nd
1-hexanol	7.72	found	1.0	nd	nd	nd	nd	nd	nd	nd	nd	nd
phenylacetaldehyde	14.18	not found	1.0	0.16	0.13	nd	0.15	nd	0.14	nd	nd	0.09
nonanal	16.63	found	1.0	0.36	0.48	1.52	0.68	0.83	0.59	0.89	1.23	0.74
unidentified ketone	26.32	found	1.0	0.65	3.1	2.5	2.55	1.82	2.1	1.65	1.4	1.84
2-furancarboxaldehyde	6.36	not found		nd	nd	nd	nd	0.14	0.23	0.36	0.71	1.0
benzaldehyde	10.97	not found		nd	nd	nd	0.34	0.46	0.81	1.21	1.32	1.0
ethyl cinnamate	29.22	not found		nd	nd	nd	nd	0.23	0.38	0.54	0.89	1.0

[a] Amounts in whole plum samples very small. [b] Amounts during drying relative to that in fresh blended sample. [c] Not detected.

was utilized. These latter fruit were destoned and the flesh homogenized with Milli-Q water in a mechanical blender (Panasonic). Dilution with water (1 g/g sample) ensured a homogeneous sample and avoided difficulties of blending the dried samples. This was also necessary to enhance the effect of salting. About 25 g of mashed plum sample was then taken and immediately transferred to a glass vial (50 mL). Salt was added to the vial and the sample was thoroughly stirred and quickly capped. Preliminary experiments showed that one gram was sufficient for the mass of sample to enhance the amount of volatiles on the fiber. The addition of salt is widely used to increase the sensitivity of an analytical method by changing the properties of the phase boundary and decreasing the solubility of hydrophobic compounds in the aqueous phase (Yang and Peppard, 1994). The dimensions of the headspace were such to ensure that the entire length of the fiber was fully exposed without touching the sample. The sample was allowed to stand at room temperature for 30 min.

Solid-Phase Microextraction (SPME) Analysis. A commercially available manual SPME fiber holder containing a SPME fiber (Supelco, USA) was used. Preliminary experiments found that a 100 μm poly(dimethylsiloxane)-coated fiber gave the best extraction of volatiles from the headspace. The sample-containing vial was held at 60 °C in an oven for about 1 h to establish equilibrium between the headspace and the sample prior to SPME sampling. Using this fiber, the optimum extraction temperature and exposure time were found to be about 60 °C and 15 min, respectively.

Blank runs were performed regularly prior to sample analysis to ensure the removal of impurities. Each new fiber was conditioned before use by a procedure recommended by the manufacturer of desorbing any material on the fiber in a GC injector port at 250 °C for about an hour. Further conditioning of the fiber was carried out before each test in the injection port of the GC at 200 °C for about 2 min.

Gas Chromatography−Mass Spectrometry (GC-MS) Analysis. Volatile components absorbed into the SPME polymeric coated fiber were thermally desorbed onto a gas chromatography−mass spectrometry (GC-MS) system for separation and identification. The desorption of the analytes was carried out by directly exposing the fiber in the injector port of the GC for 1 min at 200 °C. GC-MS analysis was carried out using a GC-17A gas chromatography directly interfaced to a QP-500 quadrupole mass spectrometer (Shimadzu Corp., Japan). A splitless injection mode was used. Separation of components was carried out on a BP5 column from SGE (Australia) (length 25 m; i.d. 0.22 mm; film: 0.22 mm). The GC column temperature was programmed as follows: initial temperature held at 32 °C for 2 min, then increased to 250 °C at a rate of 4 °C/min, and finally held at 250 °C for 2 min. Helium was the carrier gas with a flow rate of 1.5 mL/min. The pressure was held at 4 kPa for 2 min, then increased to 40 kPa at a rate of 0.6 kPa/min and finally held at 40 kPa for 2 min. The total time of a single run was about 57 min.

Compounds were identified by matching their mass spectra with the data found in the NBS library of standard compounds. In addition, where standards were available, the retention times and mass spectra of the compounds were confirmed to yield a positive identification. The standard samples of hexanal, 2-hexenal, 1-hexanol, nonanal, 2-furancarboxaldehyde, benzaldehyde, phenylacetaldehyde, and ethyl cinnamate were obtained from Sigma.

The reproducibility of the SPME technique was examined using blended fresh plum samples. Three replicate measurements were performed in three separate vials. The precision was evaluated by calculating the mean and the relative standard deviation (% RSD) of the observed values.

RESULTS AND DISCUSSION

Volatile Constituents of Fresh Plums. Results for the volatile flavors identified in both blended and whole fresh plums are shown in Table 1. A total of six major volatile components from the blended fresh plums were positively identified by GC-MS analysis. All of these compounds found had been identified before in fresh plums. It should be noted that d'Agen plums are widely used for prune production because of their very high solids content (greater than 30% by wet weight basis). However, they do not possess a very strong aroma compared with eating varieties of plum. The results for the headspace analysis of whole plums (also shown in Table 1) are consistent with this: only two significant peaks were found. To our knowledge the volatile profile of d'Agen plums has not been investigated before.

There was also an unidentified minor peak present at 25 min whose mass spectra did not match anything likely in the NBS database. However, its mass spectrum indicates that it is probably a ketone. In addition, there was a peak tentatively identified through mass spectral comparison as octamethylcyclotetrasiloxane (OMCTS). This is thought to have originated from the fiber. Interestingly though, results from blank fiber runs did not find this compound in the trace. The presence of other constituents during headspace sampling may possibly trigger the loss of this compound from the fiber when used in plum samples.

The unresolved peak was found to comprise two major components: air as a consequence of injection onto the GC-MS system, and ethanol. Mass spectral comparison confirmed the identification of the alcohol. The presence of ethanol in plum cultivars other than d'Agen has been well documented by several authors (Forrey and Flath, 1974; Ismail et al., 1981a,b).

The major components identified in blended fresh plums were three C_6 compounds: hexanal, 2-hexenal and 1-hexanol, and nonanal. In addition, a minor peak, phenylacetaldehyde, was also found. By contrast, only nonanal and the unidentified ketone at around 25 min, in addition to the initial unresolved peak, were extracted from the headspace of whole d'Agen plums.

The C_6 compounds identified have previously been shown (Williams and Ismail, 1981; Etievant et al., 1986)

Table 2. Reproducibility of Headspace SPME Technique from Blended Fresh d'Agen Plums

compound	retention time (min) mean	% RSD	peak area mean	% RSD
hexanal	5.46	0.77	1 520 000	31.6
2-hexenal	7.09	0.28	2 640 000	16.9
1-hexanol	7.70	0.30	1 630 000	74.8
octamethylcyclotetrasiloxane	12.13	0.15	592 000	26.5
benzeneacetaldehyde	14.16	0.12	230 000	29.9
nonanal	16.62	0.089	476 000	70.4
unidentified ketone	26.36	0.33	250 000	61.0

to be significant in the aroma of fresh plums. Enzymatic activity as a result of tissue disruption (e.g., blending) during sample preparation is thought to trigger the release of these constituents from their bound state. According to Frankel (1982), the presence of these compounds could be due to lipoxygenase activity, which is initialized by the disruption of the fruit tissues when blended. These flavor compounds may be also produced through the same mechanisms during the normal preparation (i.e., cutting, peeling, crushing, etc.) of fruits. Therefore, they are essential to the flavor of most fresh fruits which are usually prepared in this manner before consumption. The presence of these compounds in cut fresh plums has also reported in the literature (Forrey and Flath, 1974; Williams and Ismail, 1981; Krammer et al., 1991; Gomez et al., 1993).

Several studies have been carried out to characterize the volatile constituents of fresh plums (Crouzet et al., 1990). Over 280 different volatile constituents have been identified from plum cultivars using various techniques. It has been found that the composition of fruit aroma varies qualitatively and quantitatively depending on the cultivar, maturity stage, climatic conditions, and the production area of the cultivar (Chassagne et al., 1996).

Table 2 shows the results of the reproducibility study of the major volatile constituents of fresh blended plums with their corresponding retention time and peak area. The reproducibility in terms of the retention time for all the compounds extracted was excellent with RSD of less than 1%. On the other hand, the reproducibility in terms of the intensity of the extracts varied with different components. Of the seven major compounds extracted, four had a RSD greater than 30%. Several researchers have reported a relative standard deviation of the SPME technique in the range of about 3−30% depending upon the type of application (Arthur et al., 1992; Potter and Pawliszyn, 1992). The differences in the reproducibility of the SPME technique obtained from this study may be attributed to high natural variation in the composition between fruit samples.

There were much fewer compounds detected in intact whole plums compared with blended plums at much lower concentrations and, in particular, no C_6 compounds were found from the headspace of whole intact plums. This is consistent with the blending being responsible for the generation of these products. Qualitative and quantitative differences in the extracts between blended and whole intact plums could also be due to the additional effect of salting. In this study, the blended fruit sample was homogenized in water with the addition of sodium chloride. Several researchers have shown that the efficiency of extraction could be improved by the addition of salt to the sample matrix (Zhang and Pawliszyn, 1993). The wax coating present on the skin surface of a plum is hydrophobic and could trap the flavors within the fruit matrix.

The detection of nonanal both in blended and intact plum samples indicates that this compound is present in the fruit and is not subject to enzymatic cleavage to release them. The greater amount found in the blended sample may indicate that physical entrapment of components within the whole fruit may be important.

Change in Volatile Constituents of Plums during Drying. There were some major changes in the composition of volatile compounds in d'Agen plums during drying (see Table 1). The first notable difference in volatile constituents of plum detected before and after drying was the complete disappearance of C_6 compounds after 1 h of drying. By contrast, phenylacetaldehyde, nonanal, and the unidentified peak (25 min) were retained. It was found that three major new compounds were generated at different stages of drying: benzaldehyde (7 h), 2-furancarboxaldehyde (9 h), and ethyl cinnamate (9 h).

The disappearance of C_6 compounds could be due to the loss of enzyme activity during drying. In this study, plums were dried as whole fruit and the sample for flavor analysis was prepared by mashing the dried plums in water immediately after drying. A number of studies (Davidek et al., 1990; deMann, 1990) have shown that lipoxygenase enzymes are rapidly denatured through heating. These volatile compounds could also have been lost as a consequence of heating associated with high ventilation during drying or indeed chemically changed.

The retention of some original compounds after drying may be due to their lower volatility. In addition, since the flavor volatiles are generally larger than water molecules they may not readily diffuse (Rulkens, 1973) or are trapped (Flink and Karel, 1970) within the fruit matrix during drying. According to Saravacos (1986), carbohydrates are known to lock-in volatile flavors. This shows that some original flavor components could be retained within the dried solid which are responsible for the natural aroma of the product.

For components that were originally present in fresh plums, phenylacetaldehyde, nonanal, and the unidentifed ketone (25 min), it was found that their amounts varied with drying time. The variation in the amount of nonanal showed no clear pattern with drying time. This observation might reflect the natural variation in the chemical composition of the fruit samples. Aldehydes and ketones are known to be the precursors of many heterocyclic compounds such as furans and pyrazines (Cantalejo, 1997).

One important aspect of the results was the increasing amount of benzaldehyde, after being first detected at about 7 h of drying (Table 1). This may be due to the degradation of its glucoside precursor, amygdalin, during heating (Williams and Ismail, 1981). Amygdalin has been identified in the kernels of peach, apricot, cherry, plums, and prune (Davidek et al., 1990). The hydrolysis of amygdalin has been shown to produce a free sugar moiety and mandelonitrile which in turn dissociates nonenzymatically to form benzaldehyde and hydrogen cyanide (Davidek et al., 1990).

The most significant feature of the results in Table 1 was the formation of 2-furancarboxaldehyde. This furan derivative compound was generated with a rapidly increasing profile after about 9 h of drying. This substance was probably derived from the degradation of sugar alone (caramelization) or in combination with amino acids (Maillard reaction) by thermal heating of

the fruits. High temperatures are known to activate these reactions. This is consistent with Ismail and coworkers (1977) who found 2-furancarboxaldehyde as a major aroma component in cooked canned plums.

It is therefore possible that the 2-furancarboxaldehyde detected during drying of plums could be either from Maillard or from caramelization reactions. Since caramelization (Kroh, 1994) normally requires high temperature (>150 °C), it may be more likely that this volatile flavor was initially formed through Maillard reactions. Its rapid increase in the later part of drying may, however, be evidence of caramelization. This may be likely given the fact that the fruit had, by this stage, been heated for a long period (>10 h) and that the moisture content was very low (<15% in dry basis).

In previous work (Sabarez, 1998; Wilford et al., 1997; Price et al., 1997) we have shown that the onset of Maillard reactions occurs after 5−6 h of drying under these same drying conditions. This was manifested in the observed significant reduction in the amounts of both glucose and fructose monitored by HPLC techniques. The two reducing sugars are known to be the main reactants of Maillard reactions. The sorbitol content of the plums was found to decrease at about 8−9 h under the same drying conditions. The decrease in the amount of sorbitol has been implicated to be only due to caramelization reactions as it is a nonreactant molecule in Maillard reactions due to its lack of a carbonyl group. The rise in the amount of 2-furancarboxaldehyde in the present work at the same stage of drying would seem to indicate that the 2-furancarboxaldehyde is formed at least partly by caramelization.

This is supported by work of Pons et al. (1990) who identified 2-furancarboxaldehyde as one of the major degradation products found in the aroma of caramel obtained by heating a mixture of 10 kg of sucrose, 2.5 g of citric acid, and 1.7 kg of water at 195 °C. In addition, to further clarify the situation, d'Agen plums were dried at a lower temperature, 70 °C, for 18 h (Rh = 3%; V = 5m/s) and the headspace was examined. The major peaks found were the unresolved peak, phenylacetaldehyde, nonanal, the unidentified ketone, and benzaldehyde. In particular, 2-furancarboxaldehyde was not detected during drying at 70 °C. Results from our previous carbohydrate studies found that under these lower temperature conditions the sorbitol content of the plums was relatively unchanged throughout (Wilford et al. 1997). This was thought to imply the unlikely occurrence of significant caramelization. The lack of 2-furancarboxaldehyde at this temperature is consistent with formation of 2-furancarboxaldehyde being as a result of caramelization degradation reactions. It is interesting to note that in these experiments the onset of caramelization occurs at relatively low temperatures, the onset being around 80 °C. This is lower than often quoted, but this is probably due to the low water content in dehydrating prunes.

The major carbohydrate degradation reactions could therefore be followed via the formation of these compounds and could probably be used for routine monitoring of product quality. However, further work should be undertaken given the fact that these compounds could be precursors for the formation of various derivatives; its yield would depend on the rates of the subsequent reactions. Davidek et al. (1990) pointed out that 2-furancarboxaldehyde could be used as a measure of reactions, which are usually considered to cause deterioration of the sensory quality during sterilization and storage of canned fruit products. The generation of the above compounds is therefore relevant and could be used as an indicator of the progress of carbohydrate degradations.

CONCLUSIONS

The results have shown that SPME in conjunction with GC/MS is able to identify a number of important degradation products formed during drying of d'Agen prunes which could be associated with either Maillard or caramelization reactions. The appearance of these compounds could be correlated with previous work investigating changes in the carbohydrate concentrations during processing. Thus, it is possible to use specific marker compounds to monitor chemical changes during the processing of plums. In particular, the presence of 2-furancarboxyaldehyde is shown to be connected with the onset of caramelization in the drying fruit. This has implications for quality control of the product and also may act as an example for other industries.

ACKNOWLEDGMENT

This work was funded through a grant (UW01) from the Dried Fruits Research and Development Council (DFRDC), an agency of the Department of Primary Industries, Federal Government of Australia.

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Received for review May 24, 1999. Revised manuscript received February 14, 2000. Accepted February 23, 2000.

JF990553X