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Deodorisation of off-odour during sweet potato juice production by employing physical and chemical deodorants

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Abstract

The mechanism and efficiency of three types of deodorants, namely activated carbon (AC), maltosyl cyclodextrin (MCD) and apple polyphenol (AP), in reducing the "boiled heavy odour" of saccharified sweet potato juice was investigated. The highest deodorising efficiency of AC, followed by MCD and AP, was confirmed by using the electronic nose and sensory analysis. Furthermore, flavour compounds in the sweet potato juice were identified by GC–MS analysis. While AC decreased the peak intensities of all the compounds to below the minimum detection limit, MCD, which eliminated the odour components by the formation of enclosure compounds, did not reduce the peak intensities to a similar extent. The mechanisms of adsorption with AC and envelopment with MCD for the identified odour components of sweet potato juice was also clarified.

Keywords: Sweet potato juice; Deodorisation; Sensory analysis; Electronic sensor analysis; Activated carbon; Maltosyl cyclodextrin

1. Introduction

"Benihayato" is a sweet potato cultivar, that is being developed as a functional juice. It resembles a carrot in colour, odour and higher content of β-carotene (Sakamoto, Marumine, Ide, & Yamakawa, 1987). β-Carotene, is anticancerous, as evidenced by its effectiveness in different animal species, at different cancer sites, and in several different cancer model systems (Birt, 1986; Krinsky, 1991).

It is known that baked sweet potato possesses a pleasant aroma and the aroma compounds which may contribute to this aroma have been reported (Horvat, Arrendale, Dull, Chapman, & Kays, 1991; Purcell, Later, & Lee, 1980; Sun, Severson, Seversn, & Kays, 1993; Tiu, Purcell, & Collins, 1985). Sun, Severson, Schlotzhauer, and Kays (1995) found that maltol was a critical component of the characteristic aroma of baked sweet potato.

Although sweet potato has a great potential as an ingredient in functional foods, it exhibits a potential off-flavour in the boiling process.

In this report we evaluated the deodorisation efficacy of three different deodorants, which reduced the off-odour generated during the boiling process of sweet potato juice. The first deodorant used was an activated carbon (AC) with a porous structure creating a broad inner surface on which the odour components are adsorbed by Van der Waal's force. The second deodorant was maltosyl cyclodextrin (MCD), which is a mixture of maltosyl α-cyclodextrin and maltosyl β-cyclodextrin, which was enzymatically produced by combining maltose with cyclodextrin to enhance its water solubility. MCD encapsulates odour components into its cavities, and subsequently deodorises the unpleasant odour. The third deodorant was apple polyphenol, which is oxidised into a quinone type structure. Its phenolic groups react with compounds containing sulfur or nitrogen and eliminate the foul smell. This type of reaction is called chemical deodorisation.

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To show quantitatively the degree of reduction in the off-odour, samples of the sweet potato juice treated with three different deodorants were evaluated, using an electronic nose, sensory analysis and GC–MS. Since the mechanism of deodorisation for each deodorant differs depending on the odour components, the deodorisation scheme for each deodorant is also explained.

2. Materials and methods

2.1. Preparation of sweet potato juice

After steaming 10 kg of sweet potato by stepwise heating (at 60–65 °C for 2.5 h and at 100 °C for 1 h), 18 l of water and 50 g α-amylase (uniase BM-8; with a specific activity of 80,000 units per g) were added and the mixture was incubated for 3 h at 80 °C. Then 50 g β-amylase (uniase L, with a specific activity of 80,000 units per g) was added to the partially hydrolysed starch of the saccharified sweet potato and incubated at 55 °C for 1 h, and then 50 g of glucoamylase was added and the mixture was incubated at 55 °C for a further period of 1 h.

2.2. Deodorants

Two kinds of physical deodorants (AC from Japan Enviro Chemicals, Ltd., and MCD from Bio Research Corporation of Yokohama, Japan) and a chemical-based deodorant (AP from Asahi Breweries Ltd., Japan) were assessed. The AC had a surface area of 1077 m^2 per g, specific pore volume of 0.64 ml per g and an average pore diameter of 2.37 nm. Each deodorant was added at 2% to the juice samples.

Table 1
Material and properties of six sensors in the electronic nose

Sensor	Material	Selectivity
CH-1	SnO_2, sintered-metal oxide	Nitrogen
CH-2	SnO_2, thin-film metal oxide	Nitrogen
CH-3	SnO_2, thin-film metal oxide	Sulfur
CH-4	SnO_2, thin-film metal oxide	Hydrocarbon
CH-5	SnO_2, sintered-metal oxide	Alcohol, aromatic
CH-6	ZnO, thin-film metal oxide	Sulfur

2.3. Evaluation of deodorisation efficiency

2.3.1. Electronic nose analysis

A newly developed sensor was used for the assessment of the odour strength. Two different types of sensor elements, a (sintered metal oxide), which is highly sensitive to low molecular weight odour compounds and the other a thin-film oxide, which is sensitive to relatively large molecular weight odour compounds, were chosen for the electronic nose analysis. The production and properties of these sensors have been reported previously (Ehara, 1993; Tamaki et al., in press). This electronic nose consisted of six semiconductor sensors, combined to cover a broad range of chemical properties. The material and characteristics of each sensor are described in Table 1.

As shown in the schematic diagram of the electronic nose design in Fig. 1, the sensor was mounted in a 30 ml capacity gas chamber, in which a heat resistant cup containing 3 ml of the sample was placed. The chamber contained an open window, which could be sealed tightly after placing the sample on the plate. Output from the bridge circuit was connected to the data collector for the measurement of change in resistance. The data collector

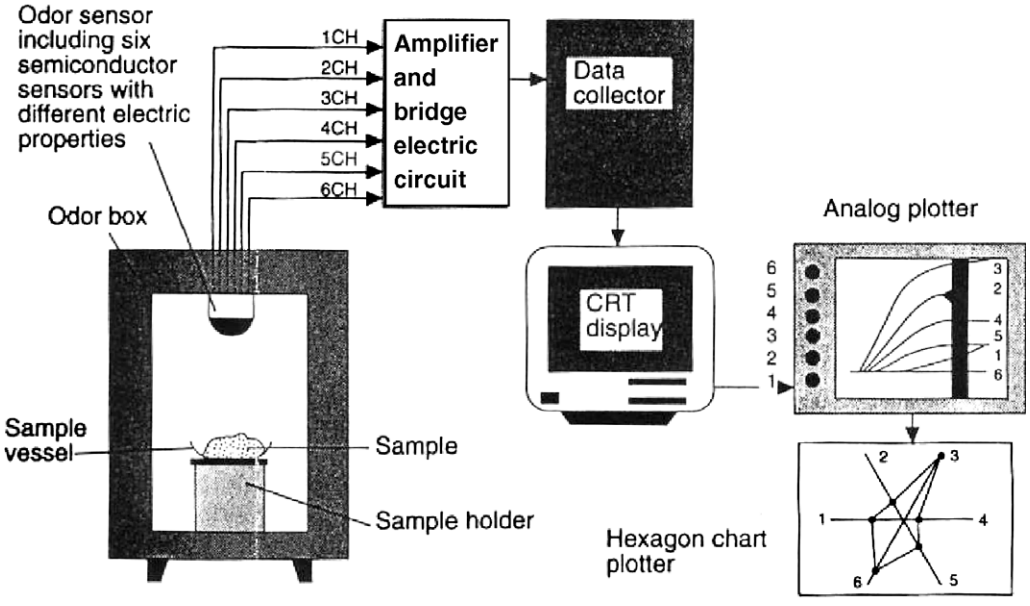

Fig. 1. Schematic diagram of the electronic nose system.

was linked to an external computer equipped with the software to handle data processing. Voltage changes transmitted through the data collector were displayed on a CRT display at specific time intervals and processed. A radar chart was also generated to show the sensor response at any given time point.

For data collection a platinum coil wire, upon which sintered-oxide and thin-film oxide metals were mounted, was heated to 300 °C. Soon after the odour compounds were released inside the chamber, the detector started collecting odour signals, and the output values were plotted against time. The instant response pattern of the six sensors was evident on a radar chart generated immediately after the output value reached a saturation level after 1 min of measurement. The data collection process lasted for about 5 min. The strength of the odour was calculated and defined as an *S*-value, which was the area under the radar chart.

2.3.2. GC–MS analysis

Volatiles were concentrated on porous polymer (Tenax TA) precolumns by bubbling with nitrogen at a rate of 80 ml per min for 60 min at 50 °C. The Tenax tube was placed in the heating block of a TCT (Thermal desorption Cold Trap) injector and heated at 200 °C to desorb the volatiles. Trapped volatiles were injected onto the GC–MS, using the TCT injector, and total ion chromatograms were obtained. GC–MS was carried out using a Hewlett-Packard Model 6890 gas chromatograph attached to a 5973 mass spectrometer. Column temperature was held at 40 °C for 10 min, programmed to rise from 40 °C to 200 °C at 5 °C per min. A fused-silica capillary column (GL Science, TC-WAX; 0.25 mm × 60 m, 0.25 μm film thickness), with helium carrier gas at 100 kPa. The mass spectrometer operated in El mode, at 70 eV with an ion source temperature of 230 °C and an interface temperature of 230 °C. library search was carried out using the Wiley GC–MS library.

The reaction between the characteristic compounds of sweet potato and AP was studied using benzaldehyde and linalool standard reagents (Wako Pure Chemical Industries, Osaka, Japan). Standard gas was prepared as follows: 0.1 g of benzaldehyde and 0.1 g of linalool were placed in a tightly sealed 20 ml glass vial and after incubation for 1 h at 25 °C; 2 ml of the standard gas was mixed with 0.2 g of AP in the same vial and incubated for 15 min at 25 °C. The headspace gas was then injected into the GC–MS using a headspace sampler.

Again a 60 m × 0.25 mm × 0.25 mm TC-Wax column was used. The column temperature, rose from 100 °C to 200 °C at 5 °C per min; other conditions were the same as for the sweet potato analysis. The ions: m/z 106 and 77 were monitored for benzaldehyde; m/z 93 and 71 were monitored for linalool.

The head-space sampler was a 7694 (Agilent Technologies, Inc.), with an oven temperature of 60 °C a vial heating time of 0.1 min; a loop temperature of 190 °C; a transfer line temperature of 220 °C and a vial pressurisation time of 0.3 min.

2.3.3. Sensory evaluation

Assessors, consisting of 20 students of Tokyo Bunka college, were trained in evaluating the degree of off-flavour in boiled sweet potatoes. Raw sweet potato juice was diluted to different concentrations in water and served in small containers. The containers were kept sealed until the judge was asked to sniff them. A mark was assigned for the intensity of off-flavour on a scale ranging from 0 to 5.

Once the panelists were trained, the effect of off-odour reduction by AC, MCD and AP treatments was evaluated. Thirty millilitres of the sweet potato juice sample were served in 100 ml glasses as an original sample. Sweet potato juice was mixed with AC, MCD and AP to give a concentration of deodorant of 2% in each sample preparation. The four individual samples were randomised over judges so that all possible serving combinations occurred and were presented to judges an equal number of times. Four samples were presented on a tray and evaluated for the intensity of off-odour from left to right. Sessions were repeated for three days until all combinations were evaluated. Two-way analysis of variance using the deodorants, the judges and their interaction as variable factors was conducted to calculate the significance of the results obtained from the six-point scale.

3. Results and discussion

3.1. Results of deodorisation by three methods of evaluation

3.1.1. Electronic nose measurement

The deodorisation efficiency was measured by electronic nose, which is represented by the *S*-value. The smaller the *S*-value is, the weaker the odour strength is. AC treated sweet potato juice showed the smallest *S*-value of 10630 and AP treated sweet potato juice had the largest *S*-value of 17390 (Fig. 2). These results confirmed that among the three deodorants used AC had the highest deodorisation efficiency.

3.1.2. Sensory evaluation

There was a significant difference between the off-odour strengths for the three deodorant treated sweet potato juice (Table 2). The scores from the lowest to the highest were in the order of the sweet potato juice sample treated with AC, with MCD, with AP and no treatment. There was no significant interaction between judges and treatments, which means that judges showed a similar behaviour. The AC treated and the MCD treated sample showed a significant difference but no significance was found between the AP treated sample and the untreated sample. It was clear that while the AC treatment had the strongest deodorisation effect, the AP treatment showed almost no effect in reducing the off-flavour of the sweet potato juice.

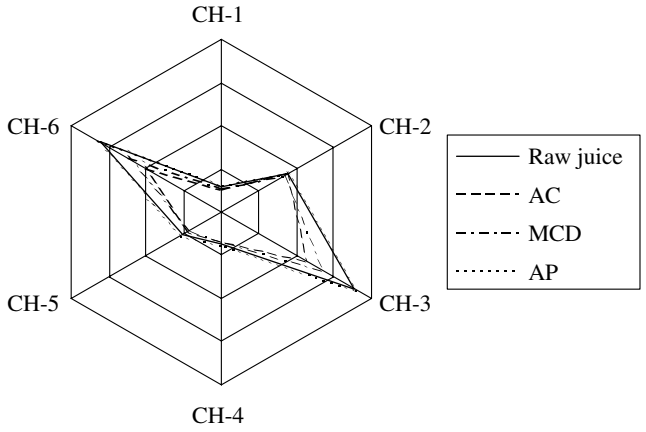

Fig. 2. Deodorisation of sweet potato odor by three kinds of deodorants. Radar chart from the six sensors' outputs (CH-1 to CH-6) of the electronic nose measurement of 6 ml sweet potato juice samples. Thick solid, broken, alternate long and short dash and dotted line indicate raw juice, juice with 2% AC and juice with 2% MCD and juice with 2% AP, respectively, with the sensor output (mV) of each sensor, *S*-value. Untreated juice: $S = 17222$; AC: $S = 10630$; MCD: $S = 11833$; AP: $S = 17390$.

Table 2
Mean values for sensory evaluation scores of the off-flavour intensity for each deodorant treatment[A]

Treatment	AC	MCD	AC	No treatment
Average	1.75[a]	2.95[b]	3.60[c]	3.75[c]
SD	0.44	0.22	0.50	0.44

[A] AC = activated carbon treated sweet potato juice. MCD = maltosyl cyclodextrin treated sweet potato juice. AP = apple polyphenol treated sweet potato juice.
[a,b,c] mean without a common letter differ ($P < 0.01$) according to Tukey HSD.

3.1.3. GC–MS analysis

The volatiles in the headspace were measured by GC–MS and twenty main components were identified (Table 3). Total ion chromatograms of headspace volatiles in the three treatments are shown in Fig. 3a–c.

The intensity of all the peaks was reduced by AC, and the extent of reduction was the largest in comparison to the other deodorants. The peak intensities of 13 of the 20 main odour components were lowered to below the minimum limit of detection. In sharp contrast to AC, only one of the 20 main odour components was reduced to below its detection limit.

The intensities of almost all the odour components were the highest in the AP treatment, indicating a very low deodorisation efficiency. However, since AP has been shown to rapidly deodorise thiol compounds (Negishi, Negishi, Yamaguchi, & Sugahara, 2004; Shimoda, Kanda, Yanagida, & Tanabe, 1996), it is thought that the odour components of sweet potato did not react with the AP due to low nucleophilicity. The poor deodorisation efficiency of AP for sweet potato odour was also confirmed by electronic nose measurement and sensory analysis.

While a comparison between the AC and the AP treated samples revealed large differences in the odour intensities of benzaldehyde, β-cyclocitral and β-ionone, only a small difference in odour intensities was noted for acetone, ethyl acetate and diacetyl.

AC is unable to adsorb odor components selectively, and it adsorbs even functional components. On the other hand, MCD exhibits some advantages, such as in making

Table 3
Size and HLB value of the main compounds in sweet potato odor and the quantities of each compound after deodorant treatments

PK no.	Compound	Axis (nm)		HLB	Deodorant		
		Major	Minor		AC	MCD	AP
1	Acetone	0.67	0.42	10.8	141	156	262
2	Ethyl acetate	0.89	0.42	7.50	112	929	921
3	2,3-Butanedione (Diacetyl)	0.80	0.42	16.3	77.8	215	296
4	Unknown	–	–	–	<5	<5	515
5	Toluene	0.83	0.42	1.07	<5	273	271
6	Unknown	–	–	–	<5	189	179
7	2-Methyl-2-butenal	0.82	0.42	7.44	<5	11.3	158
8	4-Methyl-3-penten-2-one	0.89	0.42	6.70	<5	35.1	598
9	Unknown	–	–	–	<5	173	202
10	Unknown	–	–	–	<5	63.1	177
11	2,2,6-Trimethylcyclohexanone	0.93	0.67	4.17	<5	227	370
12	3,5,5-Trimethyl-2-cyclohexene-1-one	0.93	0.67	4.28	<5	83.9	159
13	2-Ethyl-4,5-dimethylphenol	1.07	0.55	5.75	<5	235	362
14	Cycloisosativene	1.06	0.83	1.39	<5	78.7	353
15	Benzaldehyde	0.88	0.38	5.71	29.9	100	2.46×10^3
16	Unknown	–	–	–	43.5	166	402
17	β-Cyclocitral	0.90	0.67	3.75	<5	1.62×10^3	2.72×10^3
18	Geranyl acetone	1.45	0.63	2.88	26.1	84.8	228
19	2,6-Di-tert-butyl-p-cresol (BHT)	1.18	0.67	4.42	<5	46.1	227
20	β-Ionone	1.21	0.67	3.29	78.4	583	2.25×10^3

These volatile compounds were characterised and quantified by GC–MS. The analytical conditions described in the text. Values are expressed in peak height.

a complex with and stabilising the β-carotene, which is usually unstable in sweet potato juice. Furthermore, MCD also in decreases the value of cholesterol (Shizuka, Hara, & Hashimoto, 1996).

3.2. Adsorption of odor components

3.2.1. Adsorption with AC

The differential pore diameter distribution curve of the AC used in this study is shown in Fig. 4. The average pore

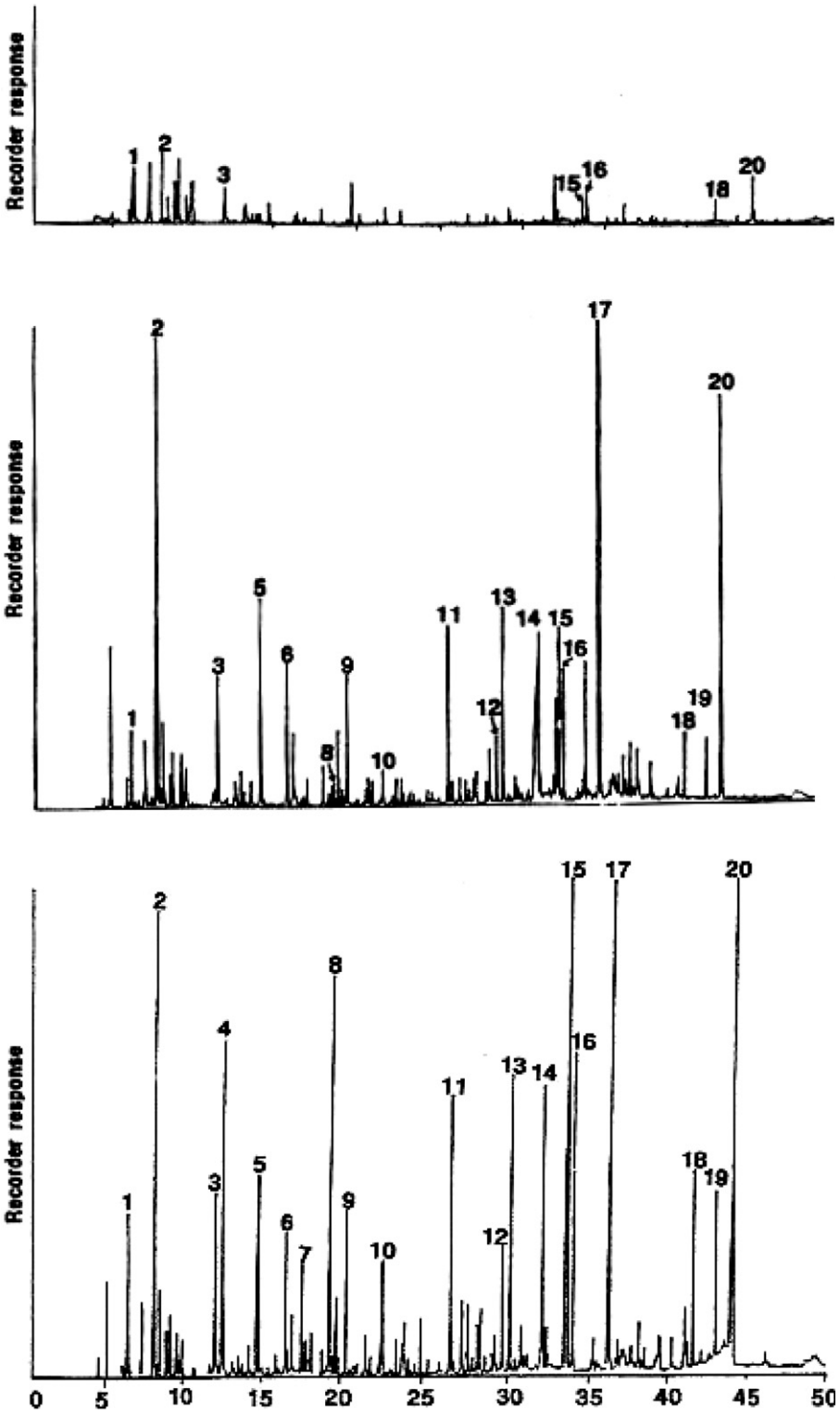

Fig. 3. Total ion chromatogram of sweet potato juice samples deodorised by AC (a), MCD (b) and AP (c). Peak identification numbers correspond to compounds listed in Table 3. These volatile compounds were characterised by GC–MS under the analytical conditions described in the text. Values were expressed in μV s ×10.

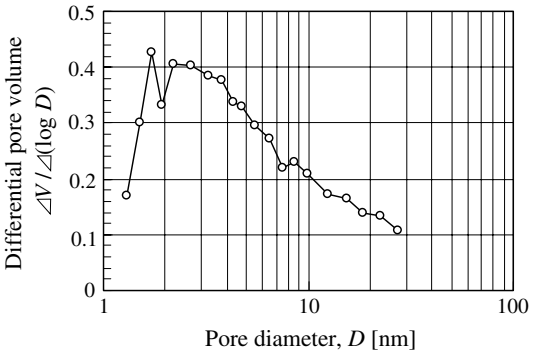

Fig. 4. Distribution of pore diameter for AC.

diameter was about 2.4 nm, and the diameter of most pores ranged between 1.5 and 6 nm. Since the largest molecule of the odour components in the sweet potato is geranyl acetone, with a diameter of 1.45 nm on the major axis, it is expected that all the odour component molecules are small enough to enter the micropores of the AC. However, AC differed in its adsorption removal ability for individual odour components. It is thought that the odour removal ability depends on the difference in physical and/or chemical interactions between the AC surface and the odour components.

Since, several kinds of coexisting odour components in water were removed by the AC adsorption, it is difficult to evaluate the potential physical and/or chemical interactions between the AC surface and the respective odour components.

Table 3 shows the hydrophile–lipophile balance (HLB) values for the 15 main odour components identified in the sweet potato juice samples (Imai & Yamori, 1997; Shinoda & Kunieda, 1983). The peak intensities of each compound after deodorant treatments are also shown. The peak intensity for acetone was 141 and acetone was not removed completely even after treatment with AC. In this regard we speculate that the relatively high hydrophilicity of acetone, whose HLB value was 10.8, prevented much adsorption on AC.

MCD removed only a small amount of acetone. Although acetone being small in size could be enveloped easily in its pores, the inner hydrophobic environment prevented hydrophilic acetone from being adsorbed. Ethyl acetate, with a relatively high hydrophobicity, showed a higher HLB value and the peak intensity remained high after AC treatment. When treated with AP deodorant, the peak intensity for ethyl acetate was approximately four times larger than that for acetone. On the contrary, the peak intensity for acetone was slightly larger than that for ethyl acetate during AC treatment. In comparison to the AP treatment a conspicuous reduction in peak intensity was noted for the AC treatment. The most probable reason for such observations was the hydrophilic and hydrophobic nature of acetone and ethyl acetate, respectively. In tune with the above results diacetyl, which was the most hydrophilic among all the other odour components, showed a HLB value of 16.3. Although the strong hydrophilic nature of diacetyl made it difficult to be adsorbed, its peak intensity was small after the AP treatment. This indicated that the concentration of diacetyl originally present in the sweet potato was low. Similarly, in comparison to the acetone and ethyl acetate, which showed lower hydrophilicity than diacetyl, a greater reduction in the peak intensity of diacetyl was noted after treatment with AC. The other odour components, such as toluene, 2-methyl-2-butena1, 4-methy1-3-penten-2-one, 2,2,6-trimethylcyclohexanone, 3,5,5-trimethy1-2-cyclohexen-1-one, 2-ethy1-4,5-dimethylphenol, cycloisosativene, β-cyclocitra1 and 2,6-di-tert-butyl-p-cresol (BHT) were adsorbed by the AC to below their detection limits. The HLB value of these compounds ranged between 1.07 and 7.44, indicating a relatively high hydrophobicity. AC could adsorb these compounds more efficiently, due to its strong hydrophobic surface. However, benzaldehyde, geranyl acetone and β-ionone, which were highly hydrophobic, were not removed completely by the AC treatment. Judging from the residual amounts of these components detected after MCD and AP treatments, it is thought that the initial concentrations of these odour components were very high.

3.2.2. Envelopment of the odor components with MCD

As mentioned earlier, since, cyclodextrin has the capability of enveloping various kinds of compounds into its cavities by forming complexes with them, it was observed that MCD showed lower GC–MS peak intensities in comparison to AP, indicating its relatively higher deodorisation efficiency (Table 3).

Both acetone and diacetyl possess the ketone group and comparatively high hydrophilicity. A significant difference was not observed for these components when the peak intensity of MCD and that of AP were compared, which indicated that these deodorants did not efficiently adsorb ethyl acetate.

While the size of toluene is just adequate to enter into the cavities of α-cyclodextrin, it also can be enveloped by β-cyclodextrin in a loose manner with in the γ-cyclodextrin cylinder (Szejtli, 1982). As shown in Table 3, while the intensity values for toluene upon treatment with MCD and AP were quite similar, a much higher reduction was observed with the AC treatment. Although the exact mechanism or reason for the above result remains unclear, we speculate that this may be due to the influence of the coexisting odor components during the the formation of the CD complex.

3.2.3. Peak nos. 7 to 20

The chemical structure of 2-methyl-2-butenal and that of 4-methyl-3-penten-2-one are similar, and their peak intensities were reduced almost equally by MCD, without any steric hindrance.

The chemical structure of 2,2,6-trimethylcyclohexanone and 3,5,5-trimethyl-2-cyclohexene-1-one are also

similar chemical structure, and MCD reduced them by a similar amount relative to AP treatment peak intensities. However, the reduction was much lower than for 2-methyl-2-butenal and 4-methyl-3-penten-2-one, probably because the cyclohexanone and the cyclohexene compounds possess several methyl groups around the benzene ring.

The peak intensities of 2-ethyl-4,5-dimethylphenol, β-cyclocitral and cycloisosativene after MCD treatment were only slightly lower than those for AP. The reason for the observed absence of any significant difference between the two compounds is attributed to their high molecular weight and the occurrence of several side-chains. In particular, 2-ethyl-4,5-dimethylphenol possesses one ethyl and two methyl groups and its peak intensity was reduced less than that of cycloisosativene. It was expected that benzaldehyde would be strongly enveloped by MCD, based on the crystal structure study of the complex between benzaldehyde and cyclodextrin by Harata, Uekama, Otagiri, Hirayama, and Ogino (1981). Its peak intensity of MCD was much lower than that in AP.

The peak intensity of geranyl acetone and beta-ionone was considerably reduced by MCD. Although MCD can envelop comparatively small and simple structure molecular compounds and make them soluble, it is more difficult to form CD complexes with the molecules having ringed structure or branched-chains. Therefore, the adsorbing efficiency of MCD was less effective with larger complex compounds, and was not as efficient as AC.

3.2.4. Reaction of odor components with AP

Unripe apples contain condensed tannins that are mainly composed of various polymerised catechins. AP is prepared from unripe apples and its main components are oligomeric procyanidins, chrologenic acid, catechin, epicatechin and phloretin glycosides (Kanda et al., 1998). Proanthocyanidins (catechin olygomers) possess varying degrees of polymerisation and cause bitterness, astringency and browning of apple products.

The possible mechanism of deodorisation by AP could be as follows. First, it is well understood that phenols and polyphenols are oxidised into the corresponding quinones by air or oxidising enzymes. Methanethiol and allylthiol, known as good nucleophiles for 1,4-addition reactions (Perlmutter, 1992), are likely to rapidly attack the generated quinones, leading to the production of the corresponding Michael adducts. This type of reaction results in an effective and quick decrease in the amount of thiols.

Benzaldehyde, linalool, phenylacetaldehyde, β-damascenone and β-ionone are considered as the important constituents of sweet potato odour. However, they do not react with AP by the Michael reaction. Our experimental results indicated that AP did not greatly reduce the amounts of either benzaldehyde or linalool (Fig. 5), which supported this theory. Thus, it may be inferred that the characteristic odour of sweet potato was not deodorised by AP.

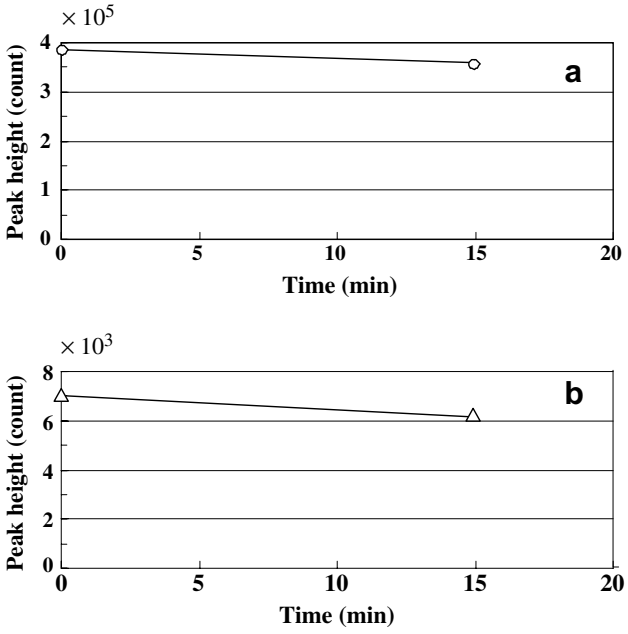

Fig. 5. The reaction between AP and benzaldehyde (a) or linalool (b).

4. Conclusion

Among the three deodorants studied, AC was the most efficient at removing the off-odour of saccharified sweet potato juice. MCD is effective at stabilising and solubilising the functional compounds such as β-carotene, although it did not deodorise the odour as much as AC. Based on the results obtained in the present study it is suggested that MCD may serve as an effective deodorant against off-odours of intermediate substances formed during the production of functional sweet potato juice.

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Appendix. HLB value

The Hydrophile-Lipophile Balance, HLB value is derived from the molecular structure of an organic compound by evaluating the strength of polarity and non-polarity around the intermolecular force between molecules with no chemical bonds. The extent of non-polarity and polarity for a molecule is shown as an organic value (OV) and an inorganic value (IV), respectively.

The OV of one-carbon atom (actually, $–CH_2–$) is defined as 20 and the total OV of an organic compound is calculated by multiplying the number of carbons in the compound.

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