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ODOUR AND TASTE ORIGINATING FROM FOOD PACKAGING BOARD

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ABSTRACT

Four commercial grades of food packaging board were analysed by chromatographic techniques and sensory evaluation. Attention was paid to providing as similar conditions as possible for the sample preparation and sample pre-treatment in the two types of analysis. A mathematical function for the dependence of sensory data on instrumental data with a prediction error of 12.3 % was found. Hexanal and pentanal were to a significant degree responsible for the odour impact perceived from the board. The developed method has been used to estimate the odour level in boards with fibres from various sources.

1 INTRODUCTION

Taste and odour properties rose greatly in importance in the late 1970's and early of 1980's. Food packaging was presumed to contribute to taint and off-flavour observed in the packed product itself. A need to understand he role of the different raw materials and the effect of different production processes on sensory properties arose very quickly. Since then the sensory properties have played a major role in the development of food-packaging boards.

To be able to evaluate sensory properties of samples a sensory panel is of primary interest. However, the panel work is very time consuming and expensive and in certain cases also not as objective as instrumental methods. This has created the need to evaluate and to quantify the samples by both sensory and instrumental methods.

Head-space GC is frequently used in the analysis of volatile compounds from packaging material. In very few reports the results are correlated to the sensory evaluation. In cases when both techniques have been used, the samples have not necessarily been analysed under the same conditions with the different methods. For instance analysis of gas phase composition above board at elevated temperature by chromatography compared to taste in water of components penetrating polyethylene (1), instrumental analysis of the gas phase above the sample as previously compared to off-taste in chocolate originating from board after a 2 day incubation period of board and chocolate at room temperature in a closed vial near to each other (2 - 9) and comparison of instrumental results as above with total odour impact at room temperature (10,11).

One aim in the present study is to describe the correlation between head-space FID-chromatograms and odour evaluated by a sensory panel. The sensory evaluation is in this case tailored to fit the head-space chromatography as well as possible, rather than the other way round. This means that the samples presented to the panelists are prepared in the same way as they are for the chromatography i.e. the same vials as for the chromatography and pretreated under the same conditions (time and temperature).

The other aim of the study is to correlate taste in water originating

from packaging material to chromatographic results.

Of those works in which both sensory and instrumental analyses have been carried out, the majority are concerned with pulp samples. This is one reason why the present study was directed to concern board samples. Another reason is the fact that in the board machine the pulp passes a washing and a drying process and thus the board represents more the final end product than the pulp. The possibility of choosing coated samples, either extrusion coated or clay coated, was also considered. The choice of polymer coating would have raised the question should one side coated materials be studied (cup board) or should the focus lay on two side coated materials (liquid packaging board). Further questions would have been should the polymers be restricted to PE or should barrier materials also be considered. In the case of clay coating the questions related to different latex types were of most interest. The variety of product grades increases very fast with the coating stage making the subject difficult to cover in one study. Thus, the boards were considered to be a common denominator for all fibre based packaging material end products, and an area on which the idea of correlating the results from both instrumental and sensory analyses was worth trying.

It was also decided to carry out the analysis on commercially available samples.

To be reliable a sensory test should not contain too many samples, 4-5 is recommended. Board samples from four different board mills, representing the main types of food packaging board grades were chosen. The types were:

A board of bleached chemical pulp
B board of bleached chemical and unbleached chemimechanical pulp
C board of unbleached and bleached chemical pulp
D board of bleached chemical and bleached mechanical pulp

Of these samples the sample D was a typical folding carton which had not been trough the clay coating process.

2 EXPERIMENTAL

The experimental part of the present work is described in three main sections. In the first section the chromatographic analyses and sensory evaluation of odour compounds originating from food packaging board are considered. The second section is concerned with chromatographic analyses and sensory evaluation of compounds that may cause off-taste in water originating from board. In both sections the chromatographic analyses are treated first, the sensory evaluation second and the combination of the instrumental and sensory data third. In the second section the use of hyphenated techniques is further included as a fourth topic. In the third section some applications related to different fibre sources are demonstrated. In the outlay there is not a separate section for the discussion. The discussions will instead be carried out within the text for each subsection.

2.1 Analyses of odour originating from food packaging board

The general principle in the odour analyses was to carry out both instrumental and sensory analyses in parallel during the same day for a set of samples. The replicates were analysed during the next day.

2.1.1 Chromatographic analyses of the potential odour compounds

Head-space GC analysis is an established method in the analysis of potential odour compounds (12,13). The theory of quantitative analyses for different kinds of sample matrices using static head-space analysis has been described by Kolb (14). A method to overcome difficulties in quantitative analyses of solid samples, based on a dynamic head-space technique, has been described by Kolb and Pospisil (15). In the present study the actual composition of the gas phase above the sample is of most interest and both static and dynamic techniques are used to describe this composition.

The samples were divided into two parts, one part which was freeze stored at - 18 °C for later use and another part which was stored under ordinary laboratory conditions at 23 °C 40 % relative humidity (unconditioned laboratory) 1-2 months before initial analyses. In both cases the samples were stored separately from each other wrapped in aluminiumfoil. The dry matter content,the dichloromethane (DCM)

extract content and the contents of the metals Fe, Cu and Mn which often are associated with lipid oxidation (16,17) were determined as background information of the samples. The head-space analysis were carried out partly by the static head-space technique (18) and partly by dynamic head-space analysis using the Jatec CIU-1C concentration injection system (19). Two similar HP 5880 capillary GC's have been used for the separations. The GC no 1 equipped with FI-detector was used mainly for the quantitative evaluation of the results. The GC no 2 equipped with the HP 5790 mass selective detector as the main detector was used for identification purposes.

Experimental conditions

Determination of background information

Dry matter:(20)
DCM extract content:(21)
Determination of Fe, Cu and Mn: (22), end determination was carried out using a Perkin Elmer ICP 40 emission spectrophotometer

GC-analyses

Static head-space injection

Samples: 5.0 g (dry matter) board samples, cut into pieces about 1 cm^2
Sample treatment: heating in a 120 ml vial 15 min, 75, 90 or 105 °C
Injector temperature: 150 °C
Pressure stabilisation period: 1 min
Pressure difference between column inlet and outlet: 6 psi
Injection time: 3 min, injected amount 1.2 ml
Oven temperature: during a 5 min period from the beginning of the injection sequence (pressure stabilisation) 0 °C

Dynamic CIU-1C injection

Sample amount: 25.0 g (dry matter) board samples, cut into pieces about 1 cm^2
Sample treatment: incubation in a 600 ml vial for 24 h at 25 °C
Stripping of volatiles: onto Chromosorb 101 adsorbent with He flow 20 ml/min, during a 20 min period
Drying of the adsorbent cartridge: with He flow 20 ml/min,during a 10 min period
Desorption of the volatiles: with an He flow of 10 ml/min during a 30 min period, desorption oven temperature 150 °C
Valve oven temperature: 110 °C
Cryo-focusing: - 196 °C
Desorption of cold trap: heating rate of the trap 12 °C/s, temperature held at 200 °C for a 5 min period.

Separation

Columns: GC1, fused silica 60 m Ø 0.32 mm SE-30 J & W Scientific
GC2, fused silica 60 m Ø 0.32 mm SE-30 Oriola
Carrier gas: He purity 99.9995 %
Oven temperature program: after injection sequence 4 °C/min from 0 to 200 °C, held at 200 °C for a 20 min isothermal period
Oven temperature post value: 15 min at 250 °C after each run

Detection

GC1: FID, 250 °C Attn 2↑0
GC2: MSD, 70 eV scanned from 20-350 amu.

Results and discussion

The background information on the samples is presented in Table 2.1

Table 2.1 The dry matter content, dichloromethane extract content and the Fe, Cu and Mn contents of the board samples.

Sample	Dry matter %	DCM-extract %	Fe ppm	Cu ppm	Mn ppm
A	96.0	0.14	52	1	1
B	95.4	0.17	43	<1	2
C	95.5	0.20	75	9	47
D	95.5	0.72	104	1	1

The dry matter contents of the boards were close to each other making the practical performance of the analyses easy. The boards were analysed without any pretreatment. Big differences in dry matter contents that often exist in pulp may make drying or conditioning of the samples before analysis necessary to get adequate results. Such a pretreatment of samples might not reflect reality. Analysed as it is the board represents the end product of a board mill and the quality that the converter is evaluating as his raw material.

As well as the content of extracts originating from wood the DCM extract content reflects the amount of sizing agent used in the board and production process. It should thus be considered as a sum parameter.

Of the metals iron is likely to originate from the process hardware. The copper is likely to originate from brass, which is, however, avoided in the process hardware as far as it is possible. The origin of manganese is probably the wood raw material itself. Manganese is removed partly in the bleaching process. In certain cases a low manganese level indicates the use of complexing agents such as DTPA. The head-space FID-chromatograms of the boards A, B, C and D at 105 C are presented in Fig. 2.1.

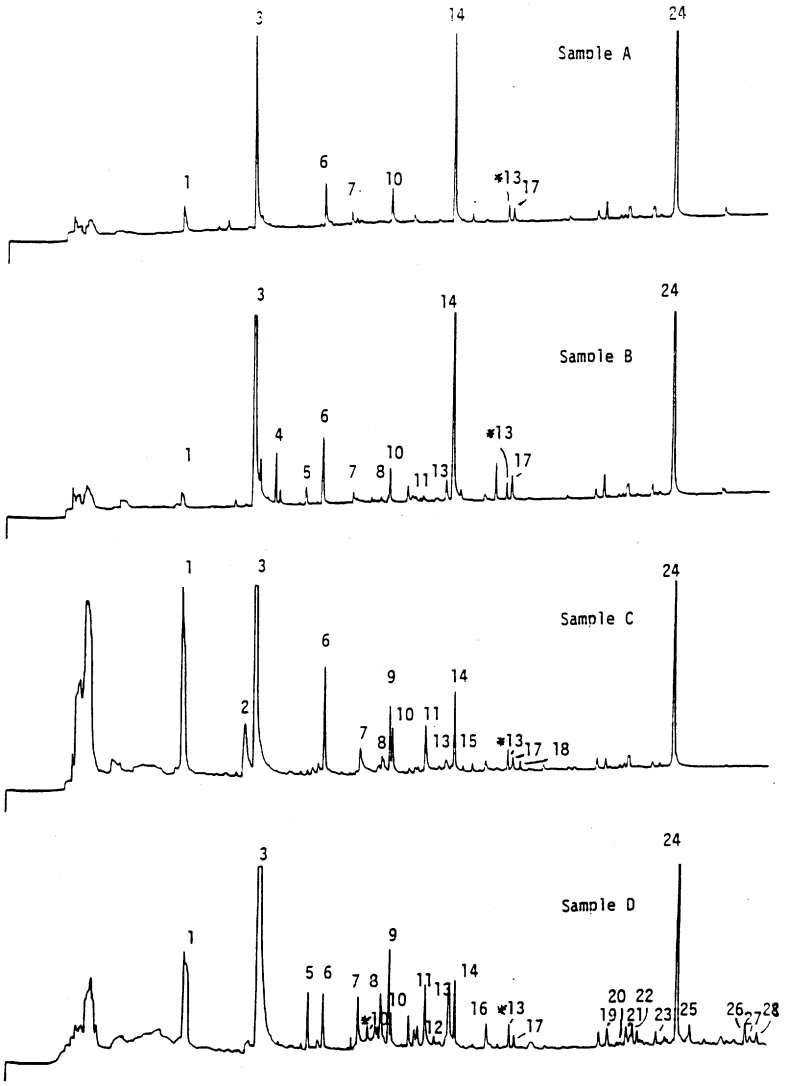

Fig. 2.1 The head-space FID chromatograms of the boards A, B, C and D run on a non-polar stationary phase SE-30. Peak identification referring to Table 2.2.

The identified compounds are listed in Table 2.2. The main identification of the compounds was done using a mass selective detector. The verification of the results was carried out using retention indices calculated on the basis of n-aldehydes (18). These indices are also listed in Table 2.2. The Kovats' indices of the compounds were also included both from a temperature programmed run 0-200 °C, 4 °C/min and an isothermal run at 120 °C.

The peaks corresponding to the compounds BHT (butylated hydroxytoluene) and TBHQ (tetra-butylhydroquinone) are background peaks originating from the infusion bottle rubber cap.

With reference to the literature (9,16,17,23,24) it can be concluded that considering the number of identified volatile compounds in the head-space above board the major part originates from the unsaturated fatty acids in wood.

The quantitative results are listed in Tables 2.3 - 2.6 from the analyses on the SE-30 stationary phase. External reference compounds have been used whenever possible for the quantification of the results. When an external reference has not been available the quantification has been done using compounds with the same FID response (same molecular formula) as the compound of interest.

From these results it can be concluded that considering the quantitative amounts of volatile compounds in the head-space above board the major part originates from the unsaturated fatty acids in wood.

It can also be seen that the volatiles as total amounts increases with temperature for all samples.

Table 2.2 The identified compounds from the boards A, B, C and D using a non-polar SE-30 column.

Compound no and name	Retention indices on SE-30 Head-space 0-200 °C	Temp. prog. 0-200 °C	Iso-therm. 120 °C	Literature reference
1 pentanal	680	680	679	675-696(25,26, 27)
2 n-pentanol	766	760	744	754-789(28,25)
3 hexanal	777	777	781	790-798(25,27)
4 2-octene	810	805	821	
5 4-methyl-hexanal	856			
6 heptanal	880	880	884	895-899(25, 27)
7 alpha-pinene	932	928	949	941-951 (29)
8 beta-pinene	966	967	990	981-993 (29)
9 2-pentyl-furan	976			
10 octanal	981	981	984	993 (25)
11 tr-2-octenal	1037	1033	1038	
12 gamma-terpinene	1052	1051	1059	1053-1061 (29)
13 6-nonenal	1075	1079	1082	
14 nonanal	1086	1086	1085	1091 (25, 28)
15 undecane	1101	1100	1100	1100 def.
16 tr-2-nonenal	1140	1135	1139	
17 decanal	1186	1186	1185	1193 (28)
18 dodecane	1197	1200	1200	1200 def.
19 C15H24 terp.	1343			
20 alpha-longi-pinene	1359	1353	1352	1360 (30)
21 C15H24 terp.	1383			
22 C15H24 terp.	1393			
23 TBHQ	1421			
24 BHT	1455			
25 C15H24 terp.	1473			
26 C15H24 terp.	1572			
27 C15H24 terp.	1580			
28 C15H24 terp.	1594			

Table 2.3 The amounts of different compounds (ppb v/v) observed in the head-space gas above the board A in two runs at different temperatures.

Comp. no	Name	Temperature							
		105 C		90 C		75 C		25 C	
		I	II	I	II	I	II	I	II
1	pentanal	527.0	604.3	0.0	0.0	0.0	0.0	0.3	0.6
2	n-pentanol	0.0	61.5	0.0	0.0	0.0	0.0	0.0	0.0
3	hexanal	6041.6	5480.5	1409.6	1120.9	190.6	399.2	0.7	0.5
4	2-octene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5	4-methyl-hexanal	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6	heptanal	587.9	529.0	0.0	0.0	0.0	0.0	0.0	0.0
7	alpha-pinene	56.7	46.2	0.0	0.0	0.0	0.0	0.0	0.0
8	beta-pinene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
9	2-pentyl-furan	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
10	octanal	710.3	673.2	0.0	0.0	0.0	0.0	0.0	0.0
11	tr-2-octenal	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
12	gamma-terpinene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
13	6-nonenal	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
14	nonanal	2672.6	2267.0	0.0	171.3	0.0	0.0	0.0	0.0
15	undecane	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
16	tr-2-nonenal	70.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0
17	decanal	183.5	124.7	0.0	0.0	0.0	0.0	0.0	0.0
18	dodecane	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
19	C15H24 terpene	55.8	69.9	0.0	0.0	0.0	0.0	0.0	0.0
20	alpha-longi-pinene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
21	C15H24 terpene	51.8	84.7	0.0	0.0	0.0	29.7	0.0	0.0
22	C15H24 terpene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
23	TBHQ								
24	BHT								
25	C15H24 terpene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
26	C15H24 terpene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
27	C15H24 terpene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
28	C15H24 terpene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL	AMOUNT	10957.4	9941.0	1409.6	1292.2	190.6	429.0	1.0	1.1

Table 2.4 The amounts of different compounds (ppb v/v) observed in the head-space gas above the board B in two runs at different temperatures.

Comp. no	Name	Temperature							
		105 C		90 C		75 C		25 C	
		I	II	I	II	I	II	I	II
1	pentanal	561.0	160.5	0.0	0.0	0.0	0.0	0.3	0.3
2	n-pentanol	83.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
3	hexanal	20915.6	34693.2	2129.6	514.9	1048.5	1366.5	0.2	0.5
4	2-octene	308.9	252.0	17.3	0.0	0.0	0.0	0.0	0.0
5	4-methyl-hexanal	0.0	0.0	222.6	311.6	0.0	0.0	0.0	0.0
6	heptanal	845.3	1085.5	0.0	0.0	0.0	0.0	0.0	0.0
7	alpha-pinene	89.1	77.0	0.0	0.0	0.0	0.0	0.0	0.0
8	beta-pinene	0.0	99.0	0.0	0.0	0.0	0.0	0.0	0.0
9	2-pentyl-furan	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
10	octanal	621.4	1030.2	0.0	0.0	0.0	0.0	0.0	0.0
11	tr-2-octenal	0.0	141.6	0.0	0.0	11.8	0.0	0.0	0.0
12	gamma-terpinene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
13	6-nonenal	268.4	362.8	0.0	0.0	0.0	0.0	0.0	0.0
14	nonanal	5087.6	6246.9	125.5	0.0	0.0	0.0	0.0	0.0
15	undecane	55.4	43.2	0.0	0.0	6.1	0.0	0.0	0.0
16	tr-2-nonenal	78.8	118.6	0.0	0.0	0.0	0.0	0.0	0.0
17	decanal	223.0	334.2	0.0	0.0	0.0	0.0	0.0	0.0
18	dodecane	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
19	C15H24 terpene	91.3	94.3	0.0	0.0	0.0	0.0	0.0	0.0
20	alpha-longi-pinene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
21	C15H24 terpene	94.0	373.8	0.0	0.0	0.0	0.0	0.0	0.0
22	C15H24 terpene	0.0	52.6	0.0	0.0	0.0	0.0	0.0	0.0
23	TBHQ								
24	BHT								
25	C15H24 terpene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
26	C15H24 terpene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
27	C15H24 terpene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
28	C15H24 terpene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL AMOUNT		29322.9	45165.3	2495.0	826.5	1066.4	1366.5	0.5	0.8

Table 2.5 The amounts of different compounds (ppb v/v) observed in the head-space gas above the board C in two runs at different temperatures.

Comp. no	Name	Temperature							
		105 C		90 C		75 C		25 C	
		I	II	I	II	I	II	I	II
1	pentanal	6530.7	6759.1	994.1	973.3	378.7	386.3	3.1	6.0
2	n-pentanol	4294.6	3262.1	0.0	0.0	0.0	0.0	0.0	0.0
3	hexanal	41088.9	40640.0	6264.6	7545.2	3238.4	3393.8	14.0	27.2
4	2-octene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5	4-methyl-hexanal	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6	heptanal	1150.1	1312.1	145.6	144.5	28.4	45.7	0.0	0.0
7	alpha-pinene	336.9	178.5	0.0	0.0	0.0	0.0	0.0	0.0
8	beta-pinene	181.3	154.3	0.0	0.0	0.0	0.0	0.0	0.0
9	2-pentyl-furan	527.5	517.0	45.0	46.5	0.0	0.0	0.0	0.0
10	octanal	870.1	853.6	86.8	79.5	0.0	0.0	0.0	0.0
11	tr-2-octenal	707.8	593.3	0.0	83.9	0.0	0.0	0.0	0.0
12	gamma-terpinene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
13	6-nonenal	248.9	196.7	0.0	34.8	0.0	0.0	0.0	0.0
14	nonanal	1036.1	1177.5	119.8	114.8	0.0	0.0	0.0	0.0
15	undecane	0.0	22.5	0.0	0.0	0.0	0.0	0.0	0.0
16	tr-2-nonenal	119.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
17	decanal	138.3	168.4	0.0	9.6	0.0	0.0	0.0	0.0
18	dodecane	32.9	35.7	0.0	5.3	0.0	0.0	0.0	0.0
19	C15H24 terpene	43.6	31.4	0.0	0.0	0.0	0.0	0.0	0.0
20	alpha-longi-pinene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
21	C15H24 terpene	87.9	54.9	0.0	0.0	0.0	0.0	0.0	0.0
22	C15H24 terpene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
23	TBHQ								
24	BHT								
25	C15H24 terpene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
26	C15H24 terpene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
27	C15H24 terpene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
28	C15H24 terpene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL AMOUNT		57394.6	55957.0	7655.8	9037.3	3645.4	3825.8	17.1	33.2

Table 2.6 The amounts of different compounds (ppb v/v) observed in the head-space gas above the board D in two runs at different temperatures.

Comp. no	Name	Temperature							
		105 C		90 C		75 C		25 C	
		I	II	I	II	I	II	I	II
1	pentanal	3292.4	4813.1	417.9	395.4	120.8	151.1	0.3	0.6
2	n-pentanol	816.0	1107.4	0.0	0.0	0.0	0.0	0.0	0.0
3	hexanal	68550.3	89407.1	10925.5	10544.6	3674.2	4288.3	3.9	6.9
4	2-octene	0.0	30.8	0.0	0.0	0.0	0.0	0.0	0.0
5	4-methyl-hexanal	441.7	548.1	44.5	0.0	0.0	0.0	0.0	0.0
6	heptanal	380.9	561.4	27.8	0.0	0.0	0.0	0.0	0.0
7	alpha-pinene	296.7	382.6	0.0	0.0	0.0	0.0	0.0	0.0
8	beta-pinene	310.8	407.3	0.0	0.0	0.0	0.0	0.0	0.0
9	2-pentyl-furan	667.9	750.8	41.5	0.0	0.0	0.0	0.0	0.0
10	octanal	305.5	451.2	0.0	73.9	0.0	0.0	0.0	0.0
11	tr-2-octenal	572.6	986.8	0.0	0.0	0.0	0.0	0.0	0.0
12	gamma-terpinene	40.9	75.9	0.0	0.0	0.0	0.0	0.0	0.0
13	6-nonenal	947.6	1422.6	0.0	0.0	0.0	0.0	0.0	0.0
14	nonanal	507.4	843.4	20.0	0.0	0.0	0.0	0.0	0.0
15	undecane	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
16	tr-2-nonenal	312.0	270.0	0.0	0.0	0.0	0.0	0.0	0.0
17	decanal	81.3	98.0	0.0	0.0	0.0	0.0	0.0	0.0
18	dodecane	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
19	C15H24 terpene	62.6	101.9	0.0	0.0	0.0	0.0	0.0	0.0
20	alpha-longi-pinene	24.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0
21	C15H24 terpene	147.5	156.9	9.8	9.1	5.1	0.0	0.0	0.0
22	C15H24 terpene	82.9	73.3	0.0	0.0	0.0	0.0	0.0	0.0
23	TBHQ								
24	BHT								
25	C15H24 terpene	139.6	146.4	12.9	6.6	0.0	0.0	0.0	0.0
26	C15H24 terpene	92.6	105.6	0.0	0.0	0.0	0.0	0.0	0.0
27	C15H24 terpene	0.0	37.0	0.0	0.0	0.0	0.0	0.0	0.0
28	C15H24 terpene	47.1	62.6	0.0	0.0	0.0	0.0	0.0	0.0
TOTAL	AMOUNT	78120.8	102840.4	11499.9	11029.5	3800.1	4439.4	4.2	7.4

The samples can further be ordered on the basis of total amount of volatiles as follows:

105 °C	A < B < C < D
90 °C	A < B < C < D
75 °C	A < B < C < D
25 °C	B < A < D < C.

The order of the samples are the same at all temperatures with the exception of the 25 °C temperature. This might reflect differences between the static and dynamic head-space techniques. The order of the samples for the temperatures 105 °C, 90 °C and 75 °C are the same as can be seen for the DCM extract content in Table 2.1. The connection between these two observations might be that in case of the elevated temperatures a thermal decomposition of unsaturated fatty acids has been clearly initiated while the decomposition at 25 °C is just about to start giving thus another order of the samples. Comparing the metal contents of the samples in Table 2.1 it might be a question of metal catalyzed autoxidation in the case of sample °C owing to the higher Cu and Mn amounts than for the other samples.

It is an open question whether the history of the samples should be accounted for or not. The storage of samples affects both the qualitative and quantitative gas phase composition above board. Attention to this has been paid in several references (2 - 5,7 - 9,10,31) in which both sensory and instrumental analyses have been carried out as a function of time in the aging process of the samples. The general observation is that the gas phase composition above board and pulp varies over time and affects thus the sensory properties of the samples. In cases of laboratory scale production of samples it is fairly easy to monitor the history of the samples as when the sample is produced can be fixed as a zero point. The same thing is true also when monitoring samples from full scale production when the history of only one sample is considered. In a study such as the current one, where the samples were collected from different board machines in different mills the monitoring of the aging process was not possible as a common zero point (production time) and a common history was lacking. The 1-2 month storage period is a realistic time delay from the production of the board at the supplier to the use of the board at the converter. Further in a study like this where the primary interest is to find a mathematical model between instrumental

and sensory data the storage time of the samples is not critical. However, the different history of the samples should be remembered in the interpretation of the sensory data.

2.1.2 Sensory evaluation of odour

An analysis of variance of the odour test data

The answers to the questions, does the raw material influence the odour level of the board, does the test temperature influence the odour level of the board and is the odour level dependent on the panelists can be answered with an analysis of variance.

The test design for the odour test in the present study was a three-dimensional analysis of variance. The samples from four different mills (M) were evaluated at four different temperatures (T) by eleven different panelists (P). The tests were carried out with two replications.

The tests were carried out serving each panelist a set of 4 samples (from the different mills A, B, C and D) in a randomized order at a fixed temperature (105, 90, 75 or 25 °C). The vials in which the samples were served were of the same type as used in the static head-space chromatographic analysis of the material. To eliminate visual bias effects in the odour classification, the vials were wrapped in aluminium foil. The vials containining 5.0 g sample were preheated in the same way as in the headspace chromatographic study, during a 15 min period in a thermostated aluminium block so that the actual temperatures were 105, 90 or 75 °C in the vials before evaluation. In the case of the 25 °C temperature level the samples were stored for a 24 h period in the vial before evaluation.

The samples were classified according to the total odour intensity, that could be perceived by sniffing the content of the vials, after the described pretreatment.

The scale that was used was the following:

0 = no odour
1 = weak odour
2 = clear odour
3 = strong odour

The use of +, - and 1/2 scores was allowed, so the scale represented in fact a 13 point interval scale. The sample A at 90 °C was in a preliminary study agreed to have the score 1. This sample was served with each sample set at a temperature of 90 °C as an external reference for the judgement.

In the present study panelists (P) were chosen as a random variable because the participants represented an aliquot of our pool of panel members. Further, this panel can be considered as an aliquot of people dealing in general with sensory evaluation of packaging material according to the results of the Technical Research Centre (32). The temperature (T) and the mills (M) were considered as fixed variables.

The distribution of the raw data from the 352 points, after elimination of the influence of the fixed variables T and M, are plotted in Fig. 2.2. The distribution is approximately normal suggesting that the methods of variance analysis can be used.

The results from the analysis of variance of the material are presented in Table 2.7.

Table 2.7 Analysis of variance of the odour test.

Source	df	SS	MS	F-ratio	
Total	351	156.94	0.45		
T(temp)	3	49.15	16.38	31.206	***
M(mill)	3	27.00	9.00	19.081	***
P(panel-ist)	10	15.47	1.55	12.664	***
TM	9	2.06	0.23	1.735	
TP	30	15.75	0.52	4.297	***
MP	30	14.15	0.47	3.861	***
TMP	90	11.87	0.13	1.080	
Sampl. error	176	21.50	0.12		

All the main effects have a significant effect on the variance. The main effects are plotted in Fig. 2.3, 2.4 and 2.5 with 95 % confidence intervals.

In Fig. 2.3 the influence of temperature on the odour is shown. The plotted values are arithmetical means of 4 samples judged by 11 panelists in two replicates.

The temperatures were selected to represent the temperature range at which the pretreatment of head-space GC analyses were usually made. These results are then often compared to odour tests performed at ambient temperatures or another elevated temperature. One of the main objects of this study is the question whether it is correct or not to correlate head-space GC analyses with a pretreatment at elevated temperatures with sensory analyses done at another temperature.

From the results of the experiment it can be concluded that the odour level in an odour test is clearly influenced by the temperature at which the test is conducted.

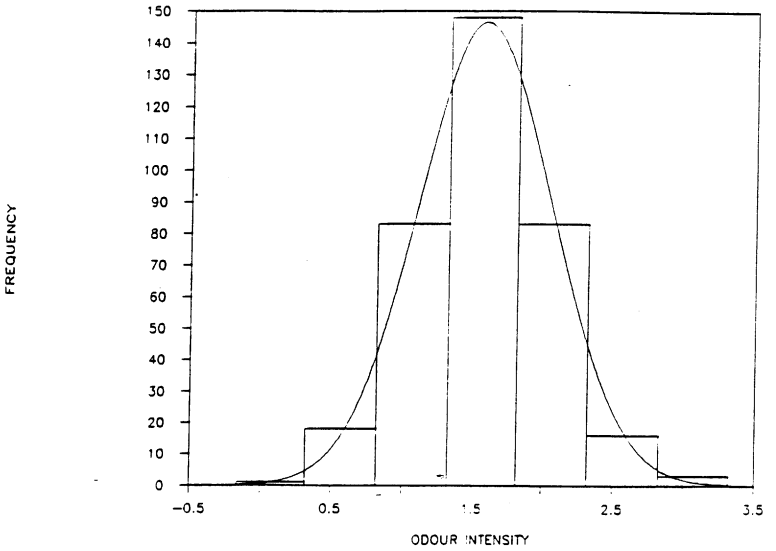

Fig. 2.2 The distribution of the odour test raw data after the influence of the fixed variables T and M has been eliminated.

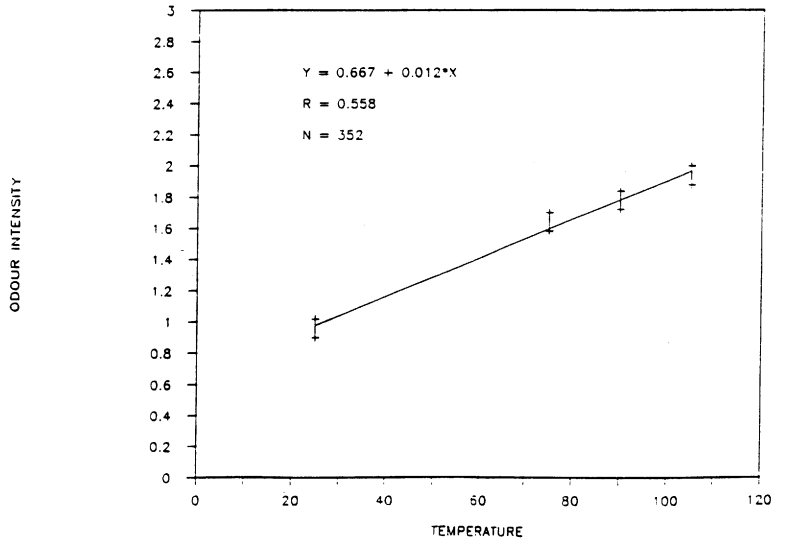

Fig. 2.3 The influence of temperature on odour intensity.

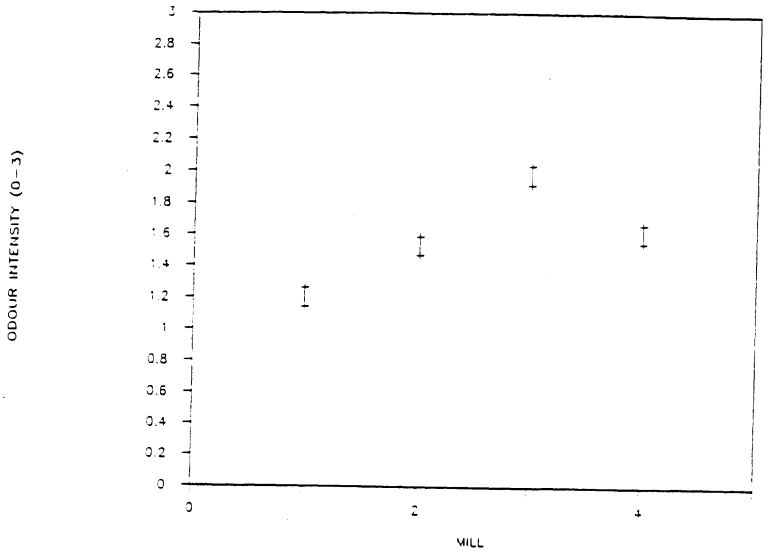

Fig. 2.4 The influence of sample type on odour intensity.

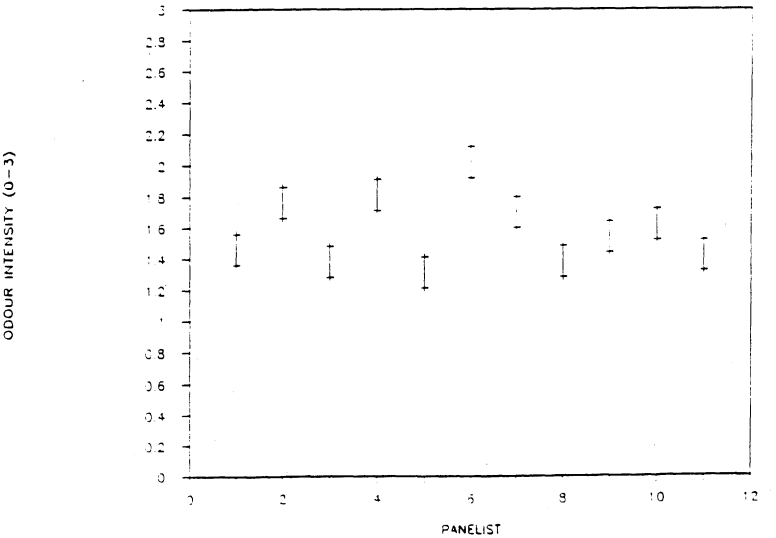

Fig. 2.5 The individual odour intensity levels for the panelists.

Compared to the chromatographic results in Tables 2.3 -2.6 it can be seen that within each sample type the total amount of volatiles is increased with an increasing preheating temperature.

The preheating influences the number of detected compounds in the gas phase as seen in Tables 2.3 -2.6. Further the preheating may also influence the relative composition of the gas phase (18). Both these matters may affect the nature of the odour.

These results indicate that static head-space GC analyses conducted at elevated temperatures, where the gas composition both quantitatively and qualitatively may vary with the heat treatment of the sample, do not necessarily measure the same thing as odour tests carried out at ambient temperature.

Moreover, it was found that there is a linear relationship between the odour intensity and the preheating temperature of the sample.

Consider now the relationship between the total chromatogram area and the preheating temperature which has been found to be of the form

$$A = u * 10^{(v*T)} \qquad (I)$$

for the board of grade C (18) where, A = total chromatogram area, T = sample preheating temperature and u and v are constants > 0. Assuming now that the total chromatogram area is a measure for the stimulus concentration C in an odour test the expression can be written after log transformation as

$$\log C = \log u + (v*T) \qquad (II).$$

Fechner's law states the following

$$I = a + b * \log C \qquad (III)$$

where I is the perceived odour intensity, C is the concentration of the volatile stimulus and a and b are constants.

By substituting (II) in (III) we hence get

$$I = a + b(\log u + (v*T))$$

which can be written as

$$I = r + q*T$$

where r and q are constants.

Under the assumption that the total chromatogram area is a measure for the total odour intensity we have thus derived the relationship which states that the total odour intensity measured on a linear interval scale is linearily dependent on the evaluation temperature.

The exponential growth of the total chromatogram area has later been verified to apply for all studied board grades.

The assumption of total chromatogram peak area to be a measure for the the total odour intensity is too general but for special cases where the increase of the stimulus concentration is related to the temperature by an exponential increase and no odour interaction effects are occurring it is theoretically possible to derive this relationship.

In Fig. 2.4 the influence of the sample type on the odour level is shown. The x-axis classification is in this case the following; 1 = mill A, 2 = mill B, 3 = mill C and 4 = mill D. The plotted values are the arithmetical means of one sample judged by 11 panelists at four different temperatures in two replicates.

The samples were selected from different mills using different raw materials and processes, to demonstrate the odour levels that can be found in the products on the market. The samples were further stored 1-2 months before analyses under the conditions given in 2.1.1. The storage simulates in a way the storage in machine rolls (no light, no control of humidity, storage at 23 °C which was supposed to be an average temperature at the converting plant and a temperature below which the roll is held on average during the transport, no excess of air), and storage time the time lag that exists before the board is converted to packages.

From this result it can be concluded that the raw material and the production process have a great influence on the odour level.

From the results it can be concluded that bleaching has a favorable effect in decreasing the odour level in boards of chemical pulp.

The other conclusion that can be drawn is that the use of mechanical and chemimechanical pulp in bleached boards of chemical pulp tends to raise the odour level.

Compared in relation to the total amount of volatiles for the samples presented in Tables 2.3 - 2.6 it can be seen that the total odour intensity is not necessarily related to the total chromatogram area. The nature of the compounds, the number of the compounds the distribution of the compounds and also the interaction between compounds may affect the total odour intensity.

In Fig. 2.5 the individual odour levels for the panelists are shown. The data values are arithmetic means of 4 samples judged at 4 temperature levels in two replicates. It is obvious from the plot that there are differences between individuals in the response to odorants.

These differences between the panelists influence the accuracy of the panel. The appearance of this effect shows the importance of using a panel instead of single person judgement.

The size of the panel depends on the type of analysis that is performed, on the differences between the samples, on the levels of significance that are required, etc.

Stone et al. (33) have discussed of using a panel instead of an expert on descriptive sensory evaluation. The size of the panel can be determined from the level of significance required for a test as discussed by Sidel and Stone (34). An alternative way is to do more replicate analyses with a smaller panel to obtain a certain level of significance. From a practical point of view when time is a limiting factor an appropriate panel size is the choice. Larmond (35) discusses the role of a semitrained panel and a highly trained panel. The individual variances will more likely balance out the larger the number of panelists are. For a highly trained panel the minimum of panel members is regarded to be five.

In general the panel is considered to be the tool for sensory analyses instead of experts.

The significant interaction effects are plotted in Fig. 2.6a, 2.6b and 2.7.

The mill/panelist interaction is presented in Fig. 2.6a and 2.6b and shows that there are individual differences both in the level and profile between the panelists.

The mill/panelist interaction should be interpreted in the way that different panelists experience the different samples in different ways. This effect has been recognized also in an earlier study (11). The solution to this problem is to use a panel instead of a single person judgement.

The panelist/temperature interaction is presented in Fig. 2.7 showing the general trend that the odour level decreases with temperature for the panelists. The other effect that can be seen is that there are differences in ranking order of the samples between the panelists originating from the temperature.

Considering the gas phase in the odour test vial as the actual sample the temperature/panel interaction can be explained as for the mill/panel interaction i.e. different panelists experience different gas compositions in different ways. The solution is the same as in the previous case.

2.1.3 Correlation studies

In this section the results from the instrumental data analyses and the sensory tests are tied together. Two mathematical models will be derived to fit the sensory data as well as possible to the GC data. The approach of deriving different mathematical models for the fit is a way to avoid big errors in the interpretation. The mathematical models that are derived will be judged with prediction ability of the model as described by Martens and Naes (36).

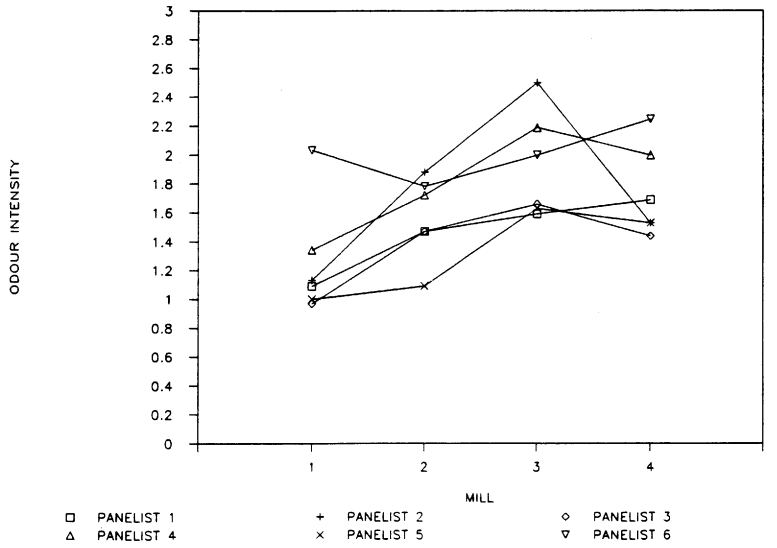

Fig. 2.6a The influeence of sample type on individual odour intensity perception for the panelists 1 - 6.

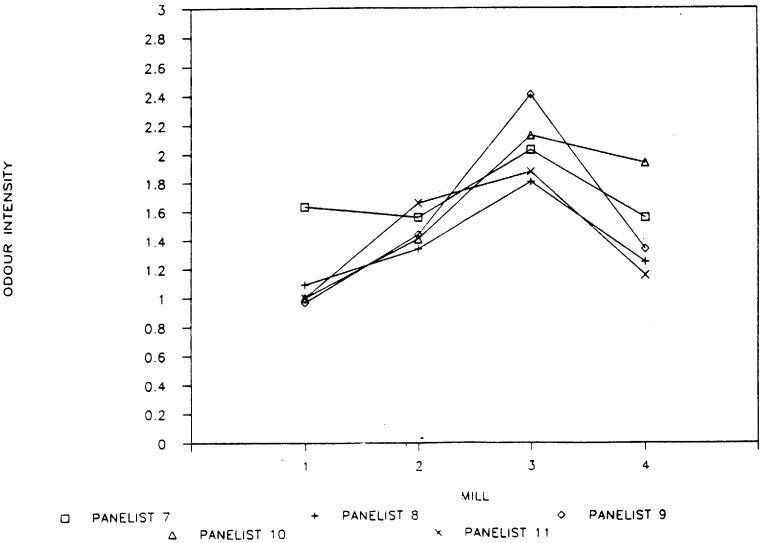

Fig. 2.6b The influence of sample type on individual odour intensity perception for the panelists 7 - 11.

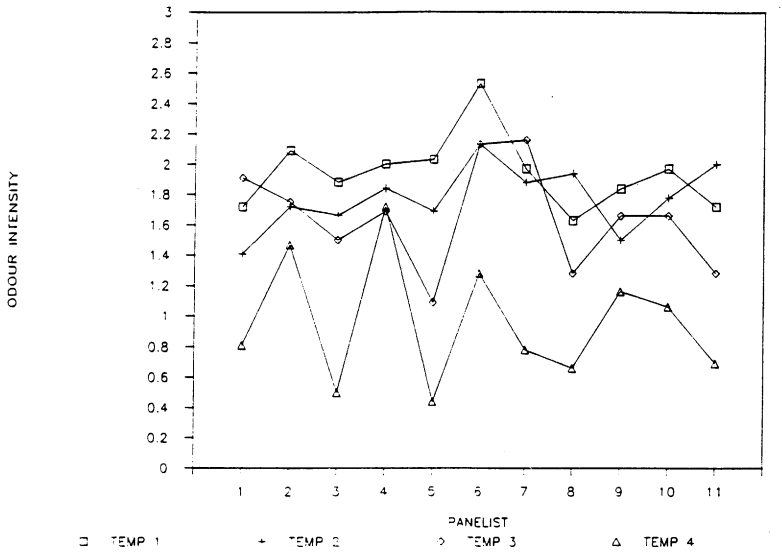

Fig. 2.7 The odour intensity levels for the panelists at different temperatures.

Multiple regression analysis of the data

The use of stepwise multiple linear regression (SMLR) in finding the best fit between sensory and instrumental data, gives a logarithmic model with two significant chemical compounds pentanal and hexanal. The statistical analysis has been run both forward and backward yielding the same result. The model found is:

$$I = 1.225 + 0.073*\text{Log}(C5) + 0.121*\text{Log}(C6)$$

where C5, and C6 are the concentrations of pentanal and hexanal respectively in ppb (v/v) in air. This model is naturally valid only above the odour threshold values of the compounds and up to the concentration where the sense of odour is saturated. With this model the prediction error using root mean square prediction with cross validation (RMSCV) was 14 %. The fit of the model is plotted in Fig. 2.8a and 2.8b. The residuals are plotted in Fig. 2.8c showing them to be randomly distributed around zero.

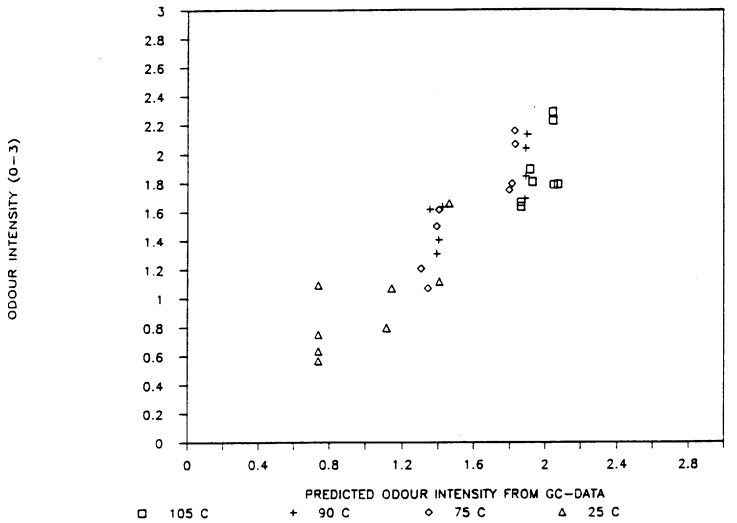

Fig. 2.8a The fit of the derived multiple regression model to the data set, with differentiation of the data points according to evaluation temperature.

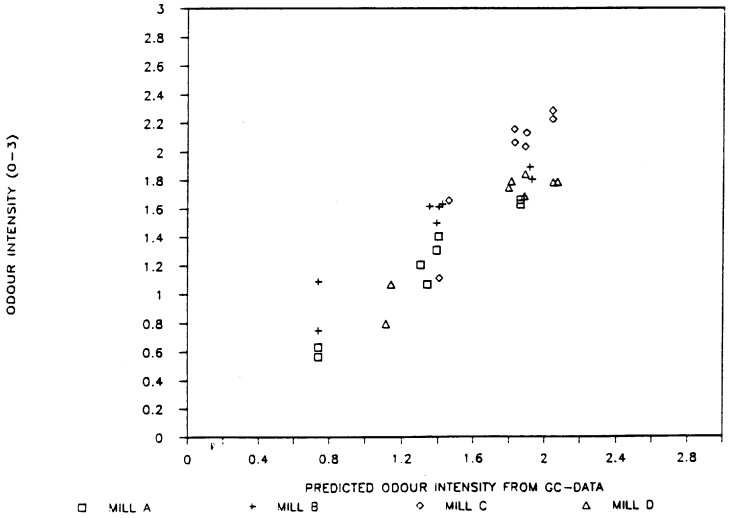

Fig. 2.8b The fit of the derived multiple regression model to the data set, with differentiation of the data points according to sample type.

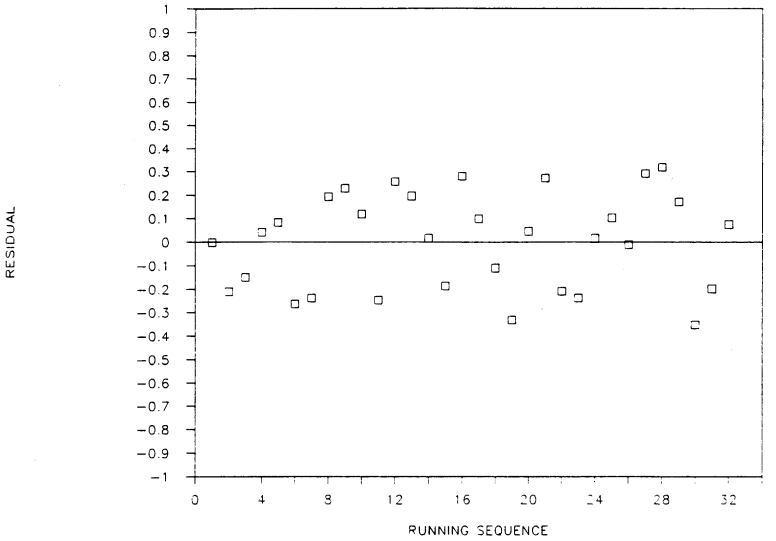

Fig. 2.8c The residual plot of the data set for the derived multiple regression model.

Multivariate calibration using PLS regression

The PLS algorithm developed by Wold in 1982 and later upgraded to the PLS2 algorithm in the SIMCA-3 B (37) programming package was next used to find the best fit between instrument and sensory data. The working principles of the PLS algorithm is given in (37) and (38).

In Fig. 2.9 the present data set is shown in terms of principal components against prediction error, analysed with PLS-regression. The data set has been run both in its original form and after log transformation both weighted (to unit variance) and unweighted. The model was tested for significance by cross-validation after the addition of each principal component. A principal component was considered significant when the CSV/SD was < 0.95. For the logarithmic models three significant principal components were found, for the linear models only one significant component could be found. Considering a three principal component model it can clearly be seen that the log transformation is to be preferred.

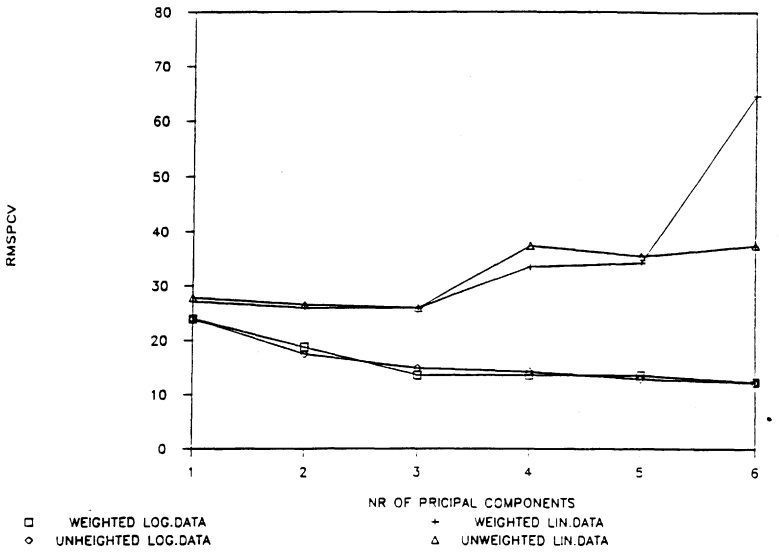

Fig. 2.9 The prediction error % plotted against the number of principal components.

The weighted model gives a lower prediction error for three principal components than the unweighted model and is therefore the optimal one of these.

The scoreplots for the two first principal components of the log transformed 1/SD weighted model are given in Fig. 2.10a and 2.10b. From these figures it can be concluded that the first principal component separates the data more or less according to temperature and the second principal component separates the data mainly according to sample type. These two components explain about 81 % of the predictivity of the model.

The loading plot for the two first principal components is given in Fig. 2.11. The compounds numbered 1, 3, 6, 10 and 14 i.e. pentanal, hexanal, heptanal, octanal and nonanal are most related to the panel mean (PM).

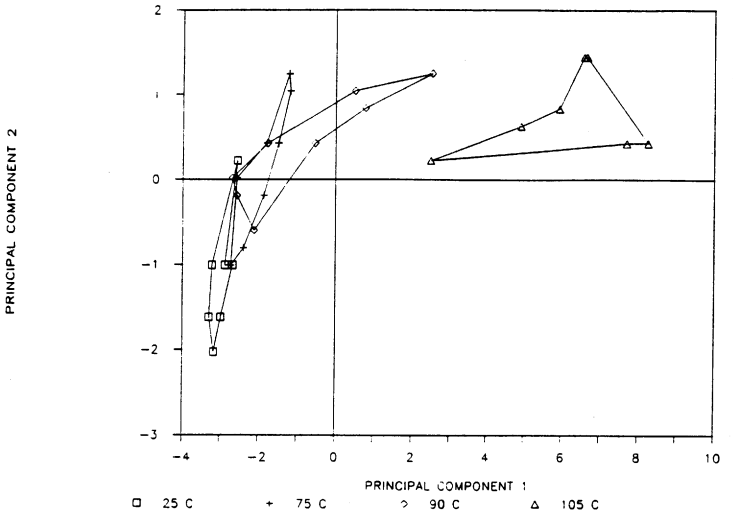

Fig. 2.10a The scoreplot of the first and second principal component with separation of the data points according to temperature.

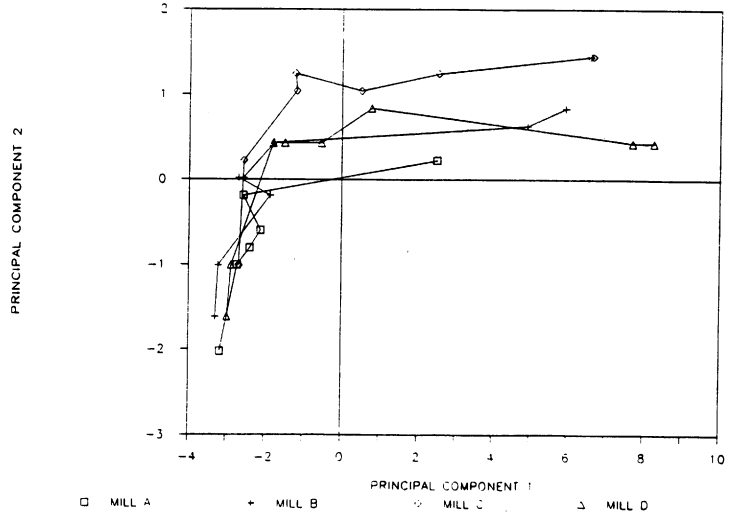

Fig. 2.10b The scoreplot of the first and second principal component with separation of the data points according to sample type.

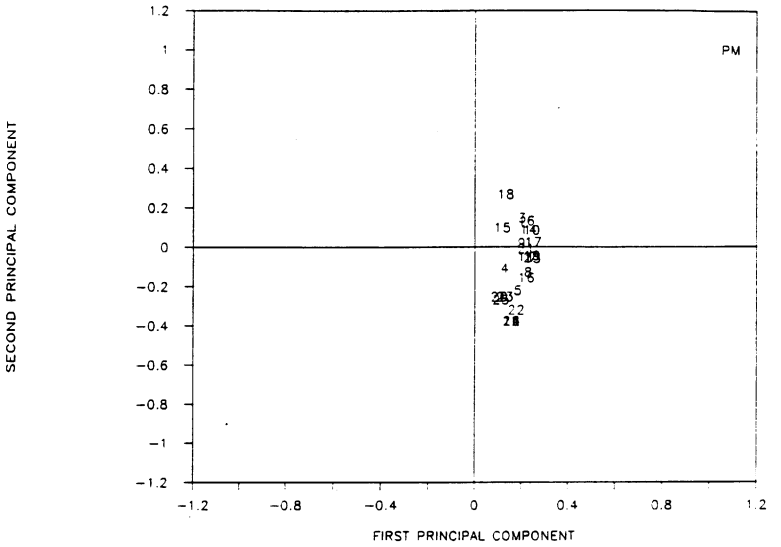

Fig. 2.11 The loading plot of the first and second principal component. PM = panel mean.

The optimal three principal component model that gives a 13.54 % prediction error (RMSPCV) is presented in Fig. 2.12a and 2.12b. The residuals are plotted in Fig. 2.12c. In comparison with the SMLR model, see Fig. 2.8, this PLS2 model distributes the observation more over the scale than in the SMLR model in which the observations are divided into three classes.

By increasing the number of principal components to 6 the model could be improved to give a 12.25 % prediction error for both the logarithmic weighted and unweighted data in this particular case. For an external data set containing 20 samples from 9 different production plants, representing 16 different board product types the optimal three component model gave a prediction error of 29.1 %.

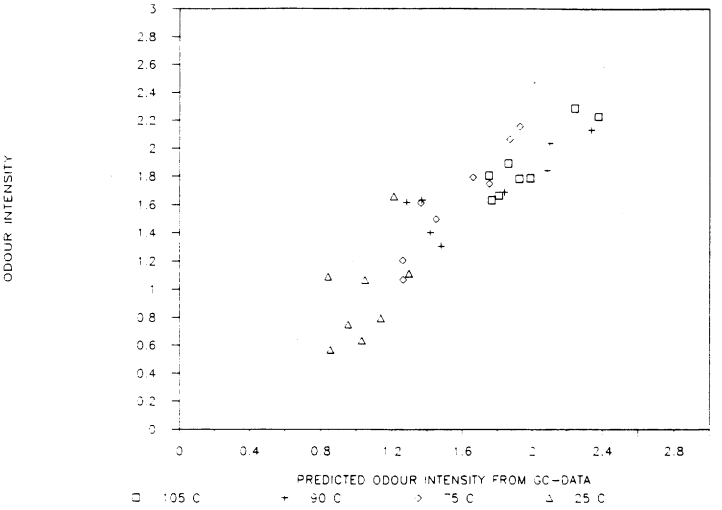

Fig. 2.12a The fit of the derived 3 principal component PLS model to the data set with differentiation of the data points according to evaluation temperature.

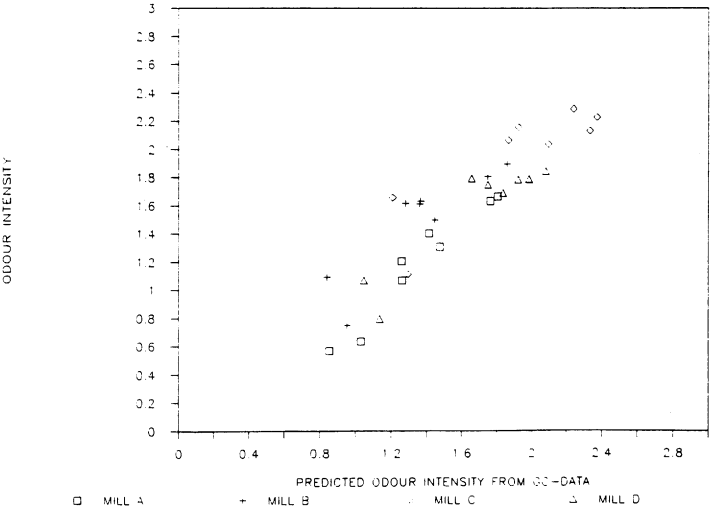

Fig. 2.12b The fit of the derived 3 principal component PLS model to the data set with differentiation of the data points according to sample type.

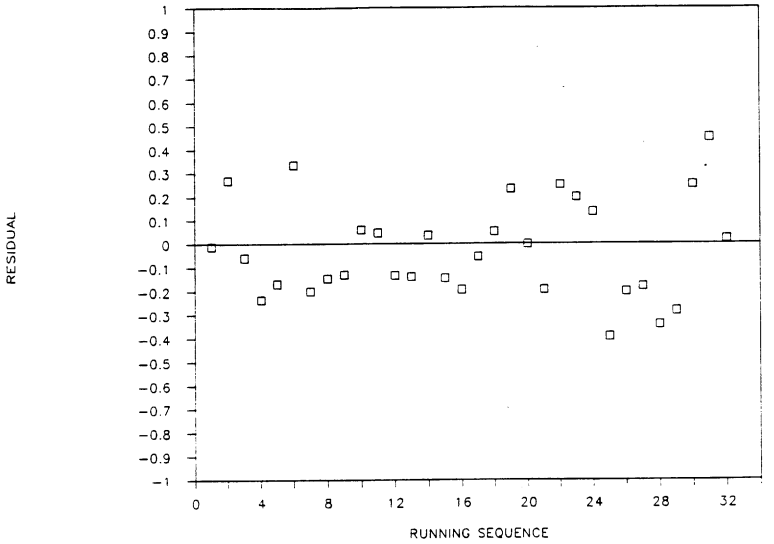

Fig. 2.12c The residual plot of the data set for the derived 3 principal component PLS model.

The data was re-examined using the PLS2 algorithm with a more user friendly multivariate program package UNSCRAMBLER. In this case the individual scores were inserted in the Y-matrix instead of the panel mean values. The loading plot for the two most significant principal components are presented in Fig. 2.13. From this plot it can be seen that some of the panelists B, H, I and K are oriented more in the direction of principal component 2 and the majority of the panelists A, C, D, E, F, G and J in the direction of principal component 1. As component 2 is related more or less to the sample temperature it is likely that the panel members B, H, I and K have judged the sample giving more weight to the perceived vapour heat impact while the others have concentrated more on the character of the gas phase.

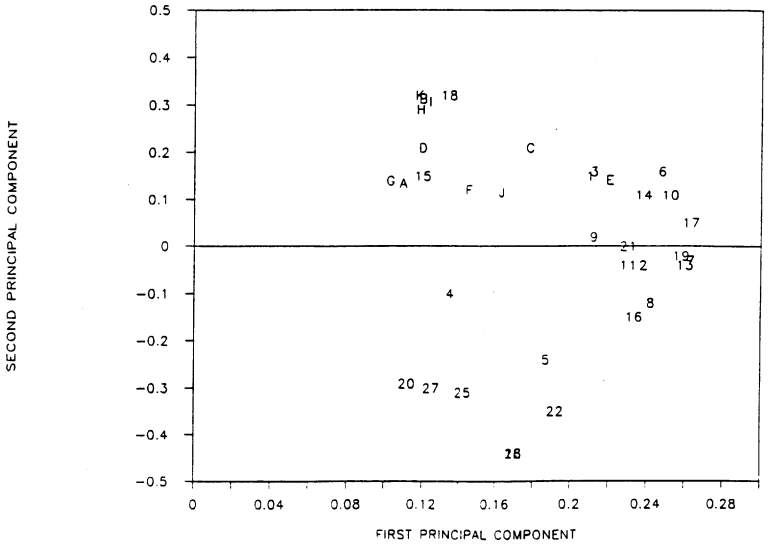

Fig. 2.13 The loading plot of the first and second principal component using the PLS2 algorithm.

2.2 Analysis of taste in water originating from food packaging board

In this main section the analysis of taste (flavor) in water originating from packaging material will be treated. The question that rises immediately is whether it is correct to analyse taste in water originating from the raw board while the board is coated with polymers to enable the packing of liquids. In many of the packaging systems there still exists a raw edge inside the package which allows the board to be in direct contact with the packed liquid thus allowing an extraction of the board. Further the polyethylene as such is not a barrier for all volatiles and migration through the polyethylene may occur. In cases of cup board the polymer coating is often only on one side. In the machine rolls the coated and uncoated side are in contact with each other enabling thus a transfer of compounds from the board to the surface of the polymer which will be the inside of the cup. Further the ready cups are stored stapled on each others to minimize the storage space, enabling in this way a transfer of compounds from the outside to the inside of the cup.

For these reasons it is desirable to have a knowledge of the taste originating from uncoated board. Using the methods described in the following sections it is possible to evaluate how much the board contributes to the packed product. The treatment in this main section is in the same order as in section 2.1. The chromatographic method mainly used is now high performance liquid chromatography (HPLC). The potential of new recently introduced hyphenated techniques is demonstrated in section 2.2.4.

2.2.1 HPLC-analysis of the samples

A requirement for odour is that the odorant is volatile. Taste can be caused both by volatile and non-volatile compounds. One way to analyse non-volatile compounds or compounds with limited volatility is by liquid chromatography (LC). In this case the taste-causing compounds are extracted with water from the sample and then analysed by LC. An ideal situation would be if an aliquot of the same sample that is evaluated by a sensory panel could be analysed by LC. This was done with reverse phase (RP) liquid chromatography during the main studies of the samples without success. The response from the LC-system was too small when the taste experiences were clear. The LC experiment was later carried out as a separate study on a separate set of material from the same batch. An ion-chromatograph (IC) was available one year after the main study and a sample set of the same batch which had been freeze stored was then analyzed.

IC-analysis

The samples were analysed by means of IC after a storage period of one year at - 18 °C. During the storage the samples were wrapped as separate packages in aluminium foil. Before the analyses, the samples were allowed to stand at ambient temperature on a laboratory bench for 24 hours. After this period the board samples were soaked in tap water brought from Ruokolahti Village (the same water that is frequently used in taste analyses). Aliquots of these water samples were sealed in bottles and stored in a refrigerator at +4 °C for 1-2 days until analysed. The analysis were carried out using a Waters IC system consisting of a a Waters Model 510 pumping unit provided with a Rheodyne injection valve, Waters Model 430 Conductivity Detector and a Hewlett Packard 3390 integrator.

Experimental conditions

Samples: 10 g board samples, soaked in 1 l of water for 3 h at 25 °C, injected amount 200 ul
Eluent: borate buffer eluent (anions), 0.5 mM EDA (divalent cations) or 2 mM nitric acid (monovalent cations), flow rate 1.2 ml/min (39).
Column: Waters IC-Pak A (07355) for anions, Waters IC-Pak C (07354) for divalent cations and the Waters Guard Column (07356) in combination with (07354) for monovalent cations
Separation temperature: 25 °C
Detector sensitivity: 2.5 uSfs for the anions, 20 uSfs for the cations

With the Waters IC-Pak A (07355) the anions F^-, Cl^-, NO_2^-, Br^-, NO_3^-, PO_4^{3-} and SO_4^{2-} can be separated (39).

With the Waters IC-Pak C (07354) the divalent cations Mg^{2+}, Ca^{2+}, Sr^{2+} and Ba^{2+} can be separated and the use of Cation Guard Column (07356) in combination with the IC-Pak C makes the separation of the monovalent cations Li^+, Na^+, NH_4^+ and K^+ possible (39).

Results and discussion

The quantification of the results was done using the external standard method. Standards 1 mg/l were prepared in deionized Elgastat UHP water. Each sample was analysed twice with the system and two setups of samples were analysed. The results are presented in Table 2.8.

The determined inorganic ions can hardly be responsible for the off-taste detected in the water due to the low concentration in which they were found and which are far below the detection thresholds for potential inorganic salts (34). From this study it can further be seen that the sample board acts as an ion-exchanger changing monovalent ions to divalent, preferably sodium to calcium. The variation in the sodium values released from the boards might be explained by the amounts of chemical pulp used for the product. The different behaviour of sample D with respect to Ca^{2+} is probably due to the fact that this board is run on a machine which frequently runs clay coated board qualities and the board is thus already loaded through the water system of the machine with Ca^{2+}.

Table 2.8 The detected amounts of cations and anions in the ion-chromatographic study of the taste test water.

Results mg/l

SAMPLE	Na+	NH4+	K+	Mg++	Ca++	CO3--	Cl-	NO3-	SO4--
BLANK WATER	7.27	0.02	0.68	0.88	0.89	0.41	0.41	0.12	0.46
MILL A	7.68	0.02	0.69	0.75	0.81	0.39	0.45	0.11	0.43
MILL B	7.59	0.02	0.61	0.71	0.76	0.35	C.39	0.11	0.46
MILL C	7.74	0.04	0.72	0.75	0.79	0.39	0.38	0.11	0.42
MILL D	7.38	0.06	0.76	0.78	0.92	0.34	0.37	0.10	0.57

SD mg/l

	Na+	NH4+	K+	Mg++	Ca++	CO3--	Cl-	NO3-	SO4--
BLANK WATER	0.07	0.01	0.05	0.00	0.01	0.10	0.08	0.02	0.05
MILL A	0.11	0.00	0.01	0.02	0.02	0.08	0.04	0.01	0.02
MILL B	0.09	0.00	0.02	0.00	0.02	0.05	0.00	0.00	0.02
MILL C	0.11	0.00	0.05	0.04	0.01	0.03	0.01	0.00	0.05
MILL D	0.18	0.00	0.04	0.01	0.01	0.06	0.01	0.01	0.01

RP-analysis

The RP-chromatography analysis was tried on an aliquot of the taste test water sample without success. The sample/water ratio was then increased and the soaking was carried out over one night. After this the samples were immediately analysed by RP liquid chromatography. This study was done in the same week as the taste test evaluation was carried out.

The equipment used for this study was a Hewlett Packard 1090 LC system equipped with an auto-injector, diode-array detector and a DPU 8-channel integrator. The controller of the system was an HP-85 BASIC-computer.

Experimental conditions

Samples: 10 g board samples soaked in 100 ml water for 16 h at 25 °C, injected amount 20 ul
Eluent: ethanol/water 10/90, flow rate 0.5 ml/min
Column: C18, narrow bore
Separation temperature: 40 °C
Detection wavelength: 260 +/- 80 nm.

Results and discussion

In Fig. 2.14 the chromatograms from the different samples are compared.

The ethanol/water eluent system was chosen for a possible sensory identification of the compounds. In a separate run where the samples had been further concentrated by evaporating a part of the water, the UV-spectra were collected in the range 210 - 410 nm. The spectra of the 4 biggest peaks were recorded. The fractions giving rise to the peaks were tasted and sniffed for a positive qualitative identification. The most efficient method was to heat the collected sample on a plate and sniff the evaporated vapour. By this method a clear smell of vanillin in the fraction giving rise to peak 4 could be perceived. Further verification was carried out by obtaining a UV-spectrum of a pure vanillin and comparing the spectrum and the retention time with those of the sample. The amounts of vanillin observed in the analysis by HPLC were < 0.7 mg/l. There was a concentration factor of 10

and a 13 h longer water extraction in the preparation of the chromatographic samples compared to the preparation of taste test samples. The present amounts of vanillin in the taste test are thus < 0.07 mg/l, which is below the detection threshold values of vanillin found in the literature (40).

In the spectra of the peaks 1, 2, and 3 a strong absorbance in the region < 220 nm is seen. Spectrum 1 and 2 show an absorption at 275 nm, spectrum 3 shows absorptions at 250 and 280 nm. Sensory identification was not successful for these compounds.

2.2.2 Sensory evaluation of the taste of the samples by an analysis of variance of the taste test data

Besides the main task in this study (analysis of volatiles and analysis of odour intensity) a smaller study of the taste originating from board, by examinating the water which had been in contact with the board, was carried out.

For this purpose 10 g of board was soaked in 1 l of tap water brought from Ruokolahti Village. This water was considered by the panel to be of good quality for the taste tests with no interferring background taste. The soaking of the board was carried out for 3 h at 25 °C. After this each panelist was served a set of 4 samples and a hidden blank in randomized order and was then instructed to judge the samples on the following scale:

0 = no off-taste
1 = weak off-taste
2 = clear off-taste
3 = strong off-taste

The use of +, - and 1/2 was allowed giving a linear 13 point interval scale. In this case each panelist was also given a blank as reference which is the reason why the scale is in off-taste units and not in total taste impact units.

The test was carried out twice using the same panel and the same board samples as in the odour test.

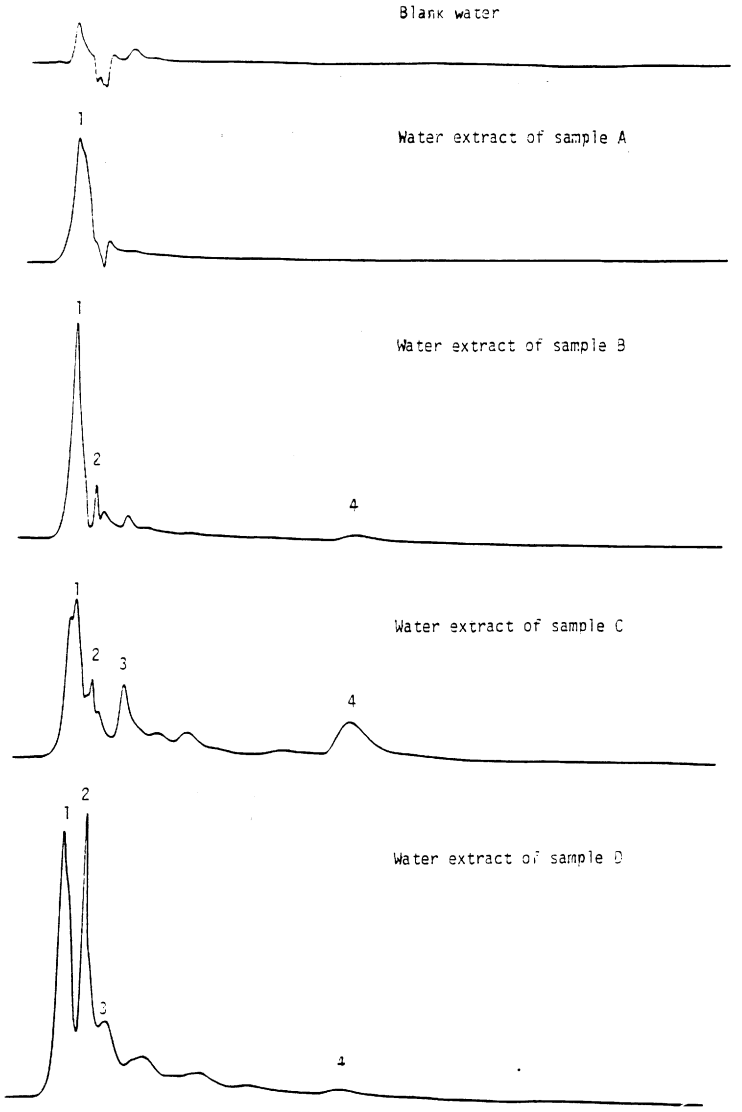

Fig. 2.14 The RP liquid chromatograms of the water extracts from the different board types

In this study the panelist (P) was again chosen as a random variable and the mills as a fixed variable with reference to the discussion in section 2.1.2.

The distribution of the raw data from 88 points of the taste test, after the elimination of the influence of the fixed variable P is presented in Fig. 2.15. The distribution is approximately normal.

The results from the analysis of variance of the material are presented in Table 2.9.

Table 2.9 Analysis of variance of the taste test data.

Source	df	SS	MS	F-ratio	
Total	87	61.92	.71		
M(mill)	3	25.59	8.53	31.593	***
P(panel-ist)	10	19.62	1.96	10.316	***
MP	30	8.25	.27	1.421	
Sampl. error	44	8.47	.19		

Both main effects have a significant influence on the variance. The main effects are plotted in Fig. 2.16 and 2.17 with 95 % confidence intervals. In Fig. 2.16 the influence of the sample type on the off-taste of water is shown. The x-axis classification is in this case the same as in section 2.1.2. The plotted values are arithmetic means of one sample judged by 11 panelists in two replicates.

A comparison of Fig. 2.16 with Fig 2.6 shows a similar profile which demonstrates the relation between the senses of taste and smell. The same discussion about the storage of samples as in section 2.1.2. applies also here.

In Fig. 2.17 the individual levels for off-taste intensity are plotted. The data values are arithmetic means of 4 samples judged in two replicates.

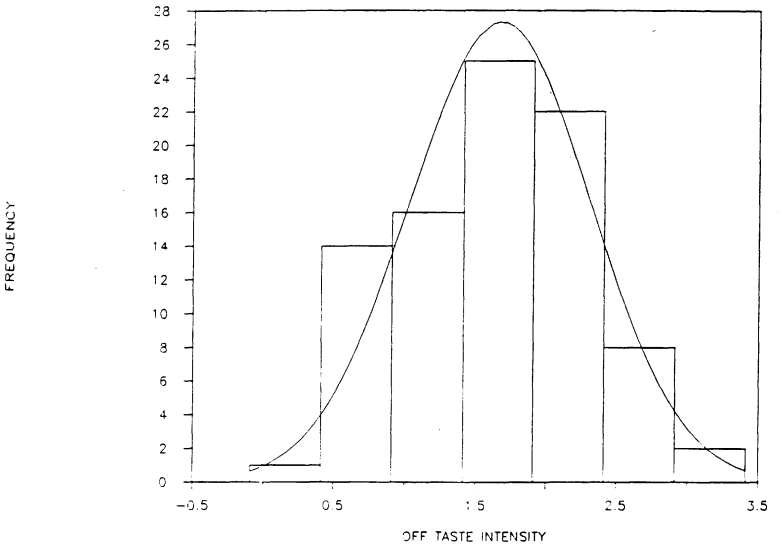

Fig. 2.15 The distribution of the taste test raw data after the influence of the fixed variable M has been eliminated.

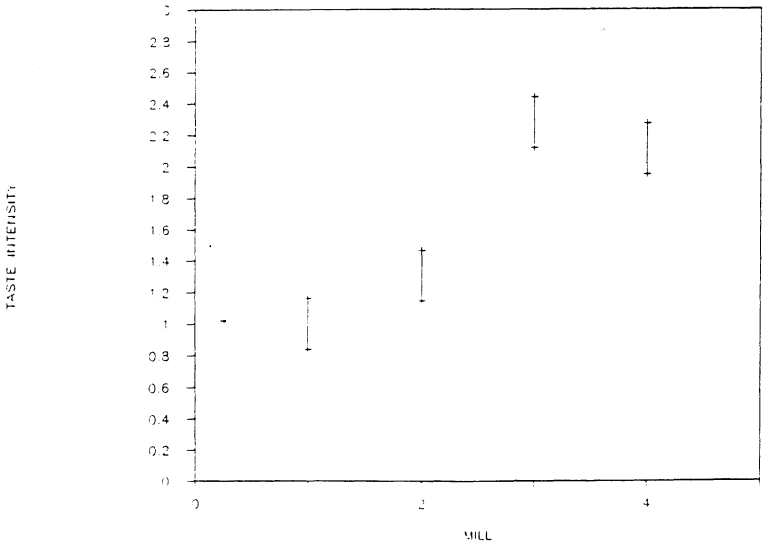

Fig. 2.16 The influence of sample type on the off-taste intensity.

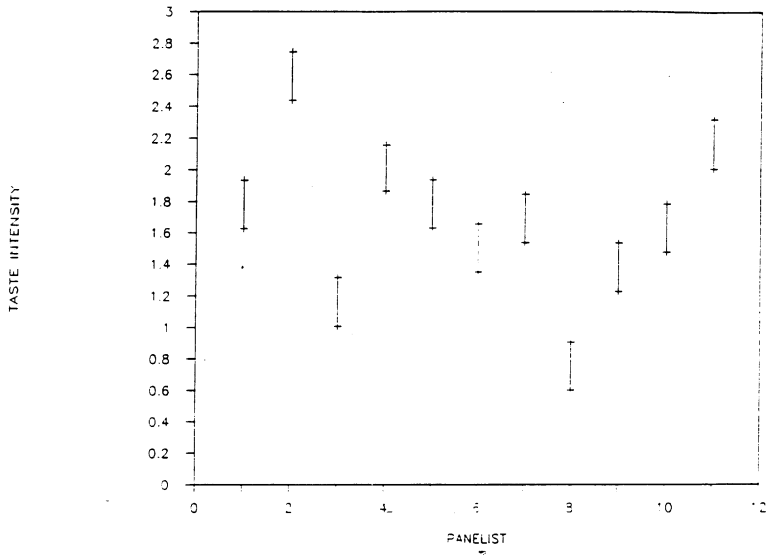

Fig. 2.17 The individual off-taste intensity levels for the panelists.

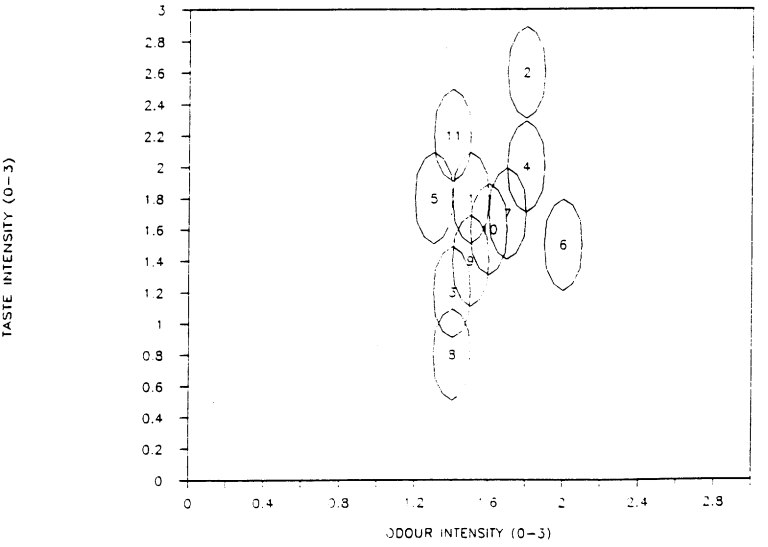

Fig. 2.18 The plot of taste scores against odour scores with 95 % confidence elipsoids for the individual panelists.

By comparing the taste test results with odour test results it can be be seen that there exist differences in the modality intensities between panelists, see Fig. 2.18. The members 6 and 8 are thus oriented more in the direction of odour.

From this analysis it can be concluded that there are significant differences in the taste level in different boards caused by different raw materials and different production processes. There are also significant individual differences in the response level between the panelists. This effect can be overcome by using a panel instead of a single judgement as discussed in section 2.1.2.

2.2.3 A correlation study of the data using multiple regression analysis

As the interpretation of the RPLC peaks is not complete the quantitative calculations of the components were omitted. Instead the absolute mAu signal values obtained from the diode-array detector were compared with the panel scores.

Using multiple linear regression analysis the best fit between the data was;

$$I = -0.4236 + 0.026*(p_1) + 0.073*(p_2) + 0.038*(p_4)$$

where p_i stands for the peak area for component i (Fig. 2.14) in mAu:s run under the conditions given in 2.2.1. The values obtained and predicted by the model are plotted against each other in Fig. 2.19. The sample material is too small to make any firm conclusions about the possibilities to use RPLC as an instrumental method for the study of taste. However, this example shows promising results.

2.2.4 The use of hyphenated techniques in analyses of taste compounds

A recent development in analytical instrumentation has given new possibilities to trace the origin of taste and odour. By the combination of fourier-transform infrared spectroscopy and mass spectrometry reliability in the identificationof the compounds is enhanced. By adding a pyrolyzer to the system, conclusions about compounds which cannot be separated by GC can be drawn.

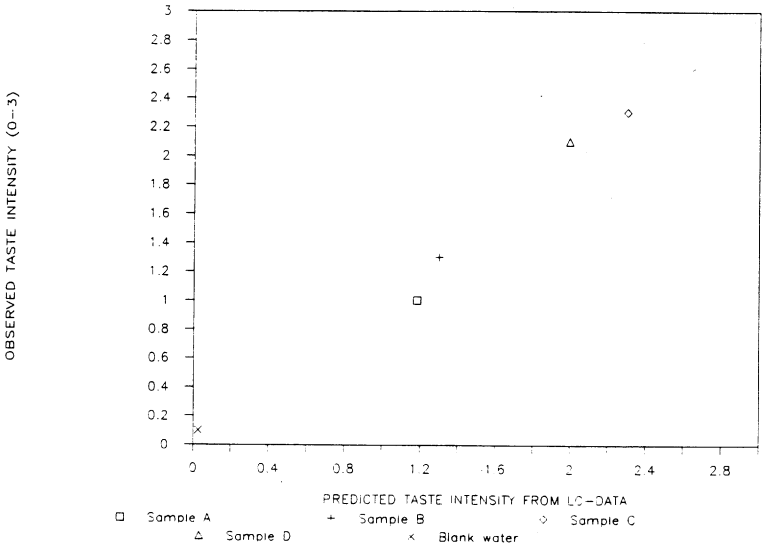

Fig. 2.19 The fit of the derived multiple regression model to the taste test data set.

The following equipment was used in this study. A fourier-transform infrared detector IRD model 5965A from Hewlett Packard designed to be located between the mass selective detector and GC with the Hewlett Packard chem. station model 59970C as a controller equipped with the EPA/IR library. The mass spectrometer was a MSD 5970 upgraded with a chem. station model 59970C equipped with the Wiley/MS library. As a separator a Hewlett Packard GC model 5890 was used, to which the IRD and MSD were coupled in a serial configuration. The injections were partly made using a split/splitless injector in the conventional way and partly using a PYROLA 85 platinum foil pyrolyzer added to the split/ splitless injector.

The following separation technique can be used to separate the water soluble compounds from board which may contribute to the taste sensation; 1) soak the board in water, 2) filter the water to get rid of possible fibres and particles in the water, 3) collect the volatiles in charcoal from the water for identification, 4) vacuum evaporate the rest of the water for the identification of non-volatile compounds.

Analysis of the volatile fraction of the water soluble compounds originating from board

The volatile fraction of the water soluble compounds was collected into charcoal using Grob's closed loop stripping system (41). The compounds were then eluted from the charcoal using CS_2 and injected to the system using a classical injection technique.

Experimental conditions

Sampling conditions

Samples: 20 g of board was soaked in 200 ml of water for 16 h, after this the water was filtered, an internal standard of 11 ng 1-chloro-nonane was added to the water
Stripping: the water was stripped into charcoal according to (41), stripping time 3 h, stripping temperature 40 °C
Fraction collection: into 15 ul of CS_2 from the adsorbent

GC-conditions

Injector:split/splitless, operated in split mode, splitting ratio 1/30 temperature 200 °C
Column: fused silica SE-30 Ø 0.32 mm 60 m Orion
Carrier gas: He purity 99.9995 %
Oven temperature: 50-200 °C 4 C/min
Transferline and light pipe temperatures: 200 °C
Detector:IRD, 4000-750 cm^{-1}, resolution 8 cm^{-1}, MSD, 70 eV scanned from 20-350 amu.

Results and discussion

The results are shown in the chromatograms of Fig. 2.20a and 2.20b. The chromatograms have been scaled on the basis of the ISTD total ion chromatogram peak intensity. The total response in the chromatograms was scaled using the same ratios with reference to sample D, to which ordinate the full scale value 1000 response units was given. The identified compounds have earlier been found as potential odourants above board 2.1.1. This result indicates that the taste observed in water originating from board may partly originate from the volatiles.

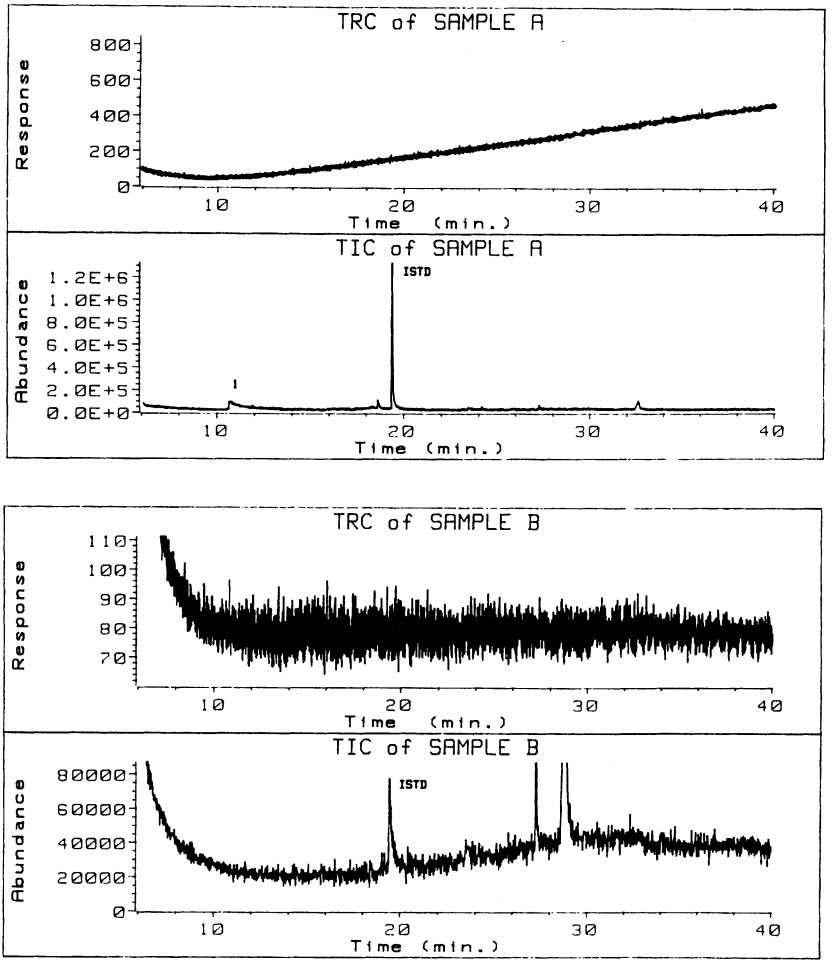

Fig. 2.20a The total response and total ion chromatograms for the water soluble volatile fraction of samples A and B (1 = hexanal, ISTD = 1-chloro-nonane).

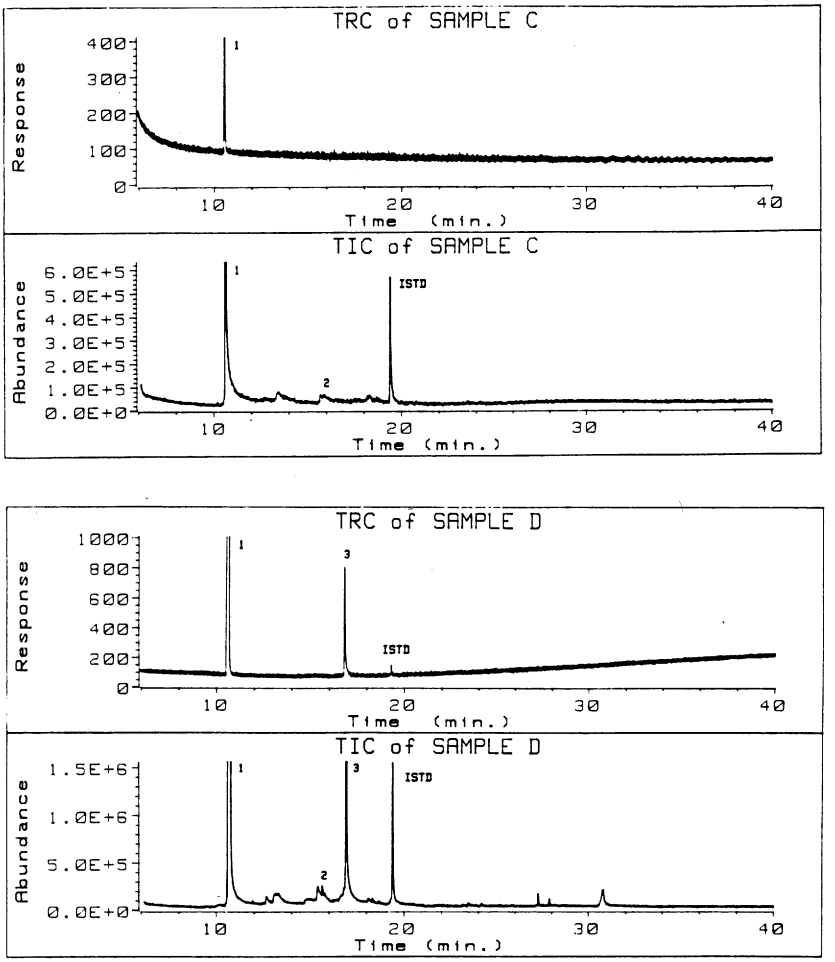

Fig. 2.20b The total response and total ion chromatograms for the water soluble volatile fraction of samples C and D (1 = hexanal, 2 = 2-pentyl-furan, 3 = tr-2-octenal, ISTD = 1-chloro-nonane).

Analysis of the non-volatile fraction of the water soluble compounds originating from board

The stripped water from the previous analyses was freeze dried. The amounts of non-volatile water soluble material per 20 g board were the following:

Sample	Amount of water soluble material/mg
A	128
B	51
C	57
D	93

A way to classify this fraction is by LC/MS or by using direct inlet probe with the MS. As these possibilities were not available the use of a platinum foil pyrolyzer was tried.

Experimental conditions

Samples: amounts in the range 0.36-0.65 mg of freeze dried non-volatile fraction of water soluble compounds originating from board

Pyrolysing conditions

Chamber temperature (T_c): 150 °C
Current pulse 1 time (t_1): 8 mS
Current pulse 1 amplitude (I_1): 41 A
Current pulse 2 time (t_2): 2000 mS
Current pulse 2 amplitude (I_2): 9 A
Pyrolysis temperature (T_p) 950 °C

GC-conditions: as above.

Results and discussion

The pyrograms of the samples are shown in Fig. 2.21a and 2.21b. Both the total ion chromatogram from the MSD and the total response chromatogram from the IRD are shown. With both detection systems 18 compounds were identified. These are listed in Table 2.9.

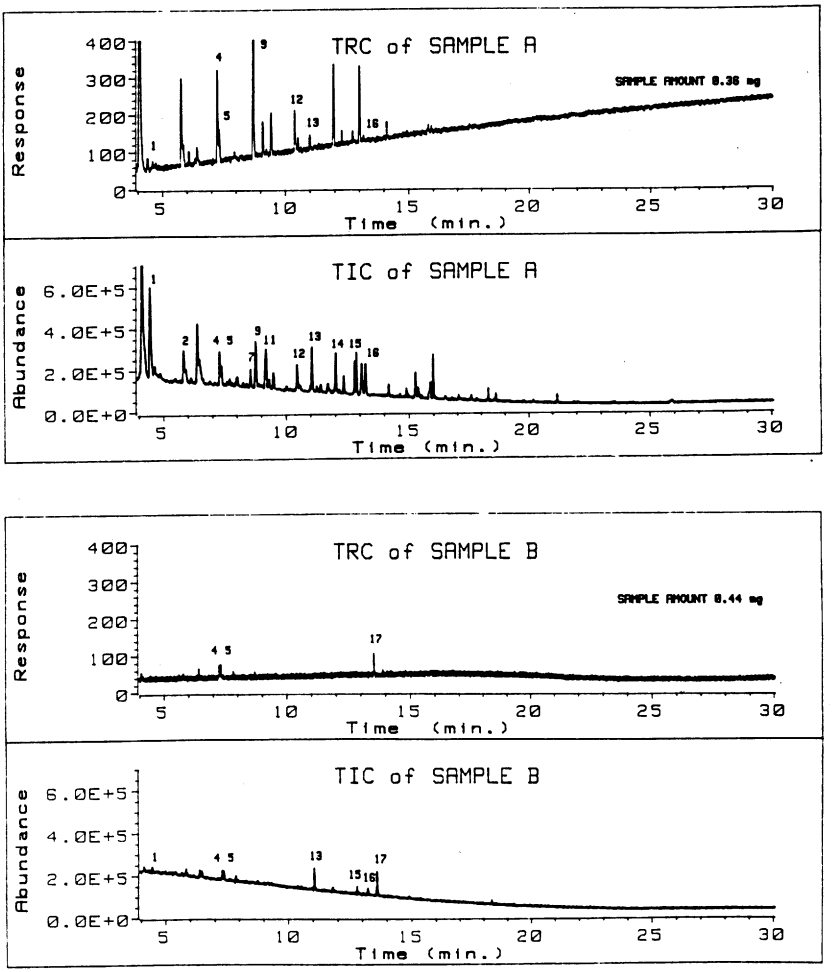

Fig. 2.21a The total response and total ion pyrograms for the water soluble non-volatile fraction of samples A and B.

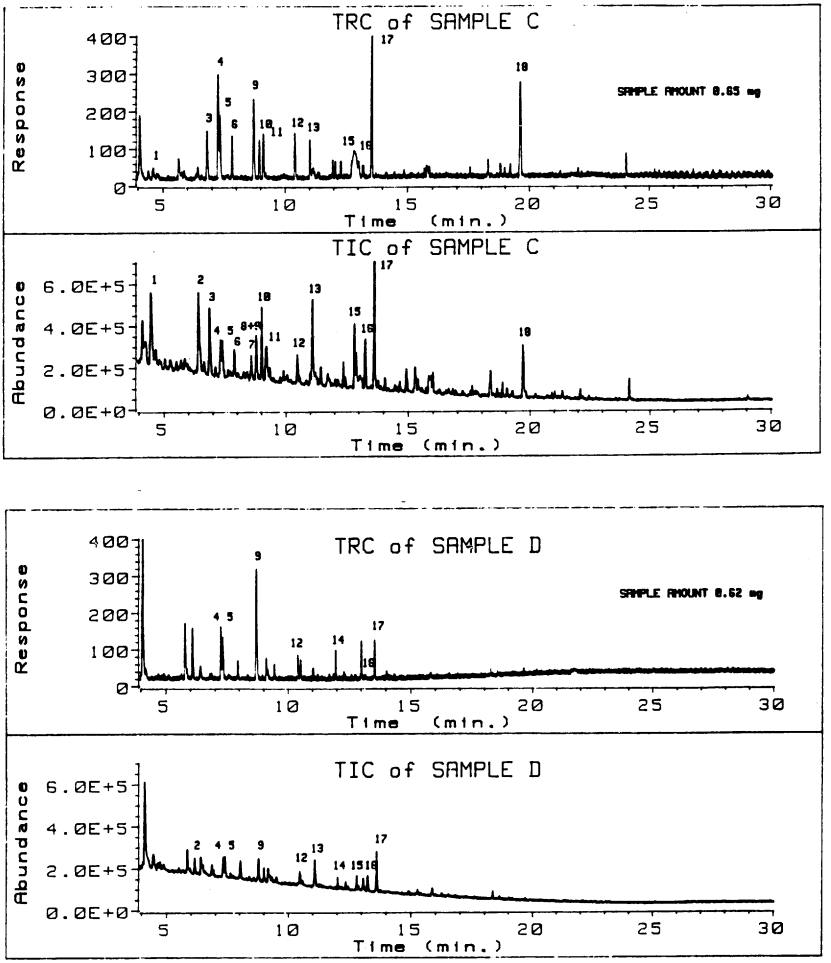

Fig. 2.21b The total response and total ion pyrograms for the water soluble non-volatile fraction of samples C and D.

Table 2.9 Identified peaks with MSD and IRD from the pyrograms of the water soluble non-volatile material originating from packaging board.

Compound	Name
1	Benzene
2	Methylbenzene
3	Hexanal
4	2-cyclopenten-1-one
5	2-furaldehyde
6	Siloxane
7	Ethylbenzene
8	Xylene
9	2(3H)-dihydro-furanone
10	2-heptanone
11	Styrene
12	3-methyl-2-cyclopenten-1-one
13	Phenol
14	2-hydroxy-3-methyl-2-cyclopenten-1-one
15	2-methylphenol
16	4-methylphenol
17	2-methoxyphenol
18	4-hydroxy-3-methoxybenzaldehyde

From these results one can work out the original materials. Thus, compounds 1, 2, 7, 8, 11, 13, 15, 16, 17 and 18 are likely to originate from lignin. Compounds 4, 5, 9, 12 and 14 are likely to originate from carbohydrates. Compounds 3 and 10 are likely to originate from the extractives (fatty acids) and compound 6 from the stationary phase of the column.

From these studies the conclusion can be drawn that the taste in the water test is partially caused by lignin compounds, carbohydrate residues and wood extractives and their oxidation products.

2.3 APPLICATIONS

In this section two cases will be demonstrated in which the odour level in boards are estimated on the basis of instrumental means. The need to estimate the odour level on instrumental basis are at hand when there is not enough sample available to carry out sensory evaluation. Typically this is the case when competitive samples are analysed. Another situation when there is a need estimate odour on the basis of instrumental analysis is when process samples from the pulping plant are analysed. The production of handsheets for a sensory test involves many sequences in which valuable information about volatiles may be lost. Further, this way of analysing is expensive and time consuming involving many manual steps. The alternative method is to filter off the water from the pulp, let the samples dry over night at ambient temperature run chromatograms on them and calculate the estimated odour levels. The calculations of the odour intensities have been carried out using the multiple regression analysis model described in section 2.1.3.

Case 1

In this case boards containing fibres from various sources are compared. All boards have been produced on different board machines. The chromatograms have been run at different temperatures to give a more reliable total view of the odour originating from the samples. The samples are the following ones:

Sample	Description
I	Liquid packaging board 320 g/m2. Bleached chemical pulp of Finnish virgin fibres.
II	Same as above produced on a different board machine.
III	Linerboard 300 g/m2. Based on Finnish recycled fibres.
IV	Linerboard 300 g/m2. Based on middle european recycled fibres.

The static head-space method described in section 2.1.1 has been used for the analyses of the samples. In fig. 2.22 the chromatograms obtained at 90 °C for the different samples and in fig. 2.23 the chromatograms for sample IV obtained at different temperatures are presented. From fig. 2.23 it is obvious that the preheating temperature increases the number of obtained compounds in the chromatogram and the amounts of the compounds obtained. In Table 2.10 the number of recorded peaks for the different sample chromatograms is given. In Table 2.11 and 2.12 the pentanal and hexanal concentrations obtained in the head-space above samples are tabulated. Table 2.13 is a tabulation of the total amount of the compounds found in the head-space above board. For this calculation hexanal has been used as an external standard. Further, in Table 2.14 the predicted odour intensities at different temperatures are given.

Table 2.10 The number of recorded peaks in the head-space FID chromatograms of the samples at different temperatures.

Sample	60 °C	75 °C	90 °C	105 °C
I	0	4	20	46
II	0	4	15	54
III	2	12	33	105
IV	3	21	53	122

Table 2.11 The amount of pentanal in the head-space/ppb at different temperatures.

Sample	60 °C	75 °C	90 °C	105 °C
I	0	0	5	10
II	0	0	5	44
III	5	57	146	455
IV	11	70	200	370

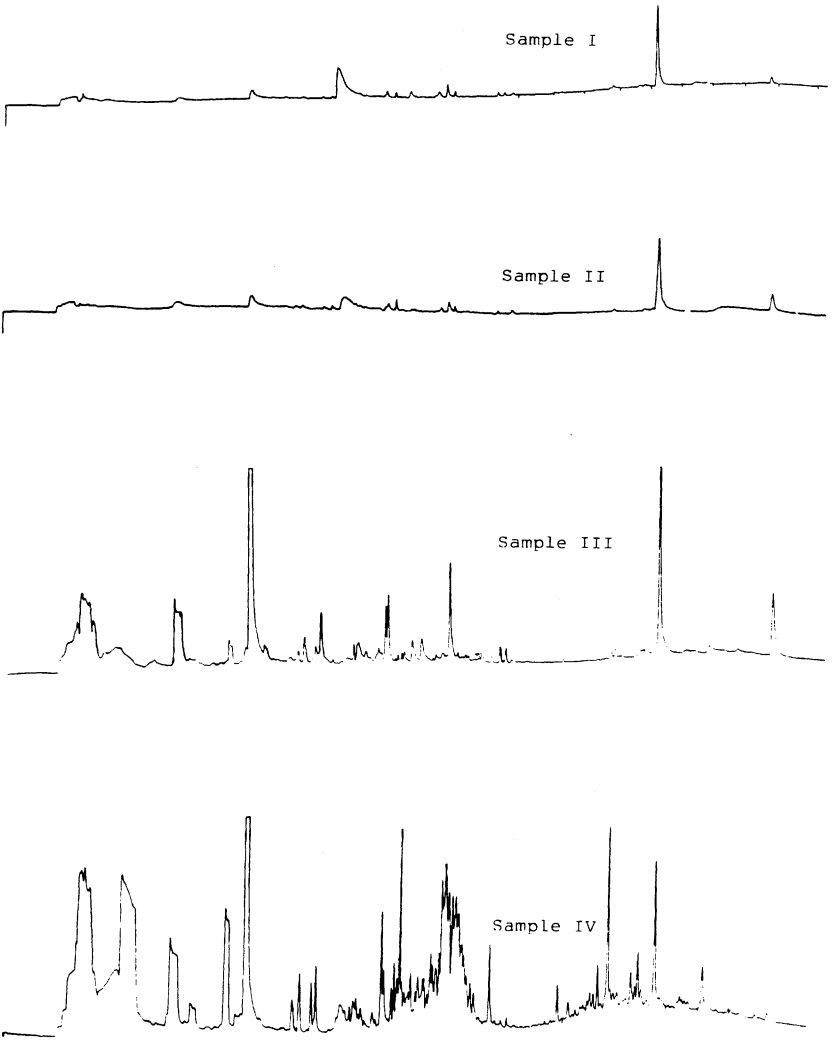

Fig. 2.22 Head-space FID chromatograms of samples I - IV. Preheting conditions 90 °C, 15 min.

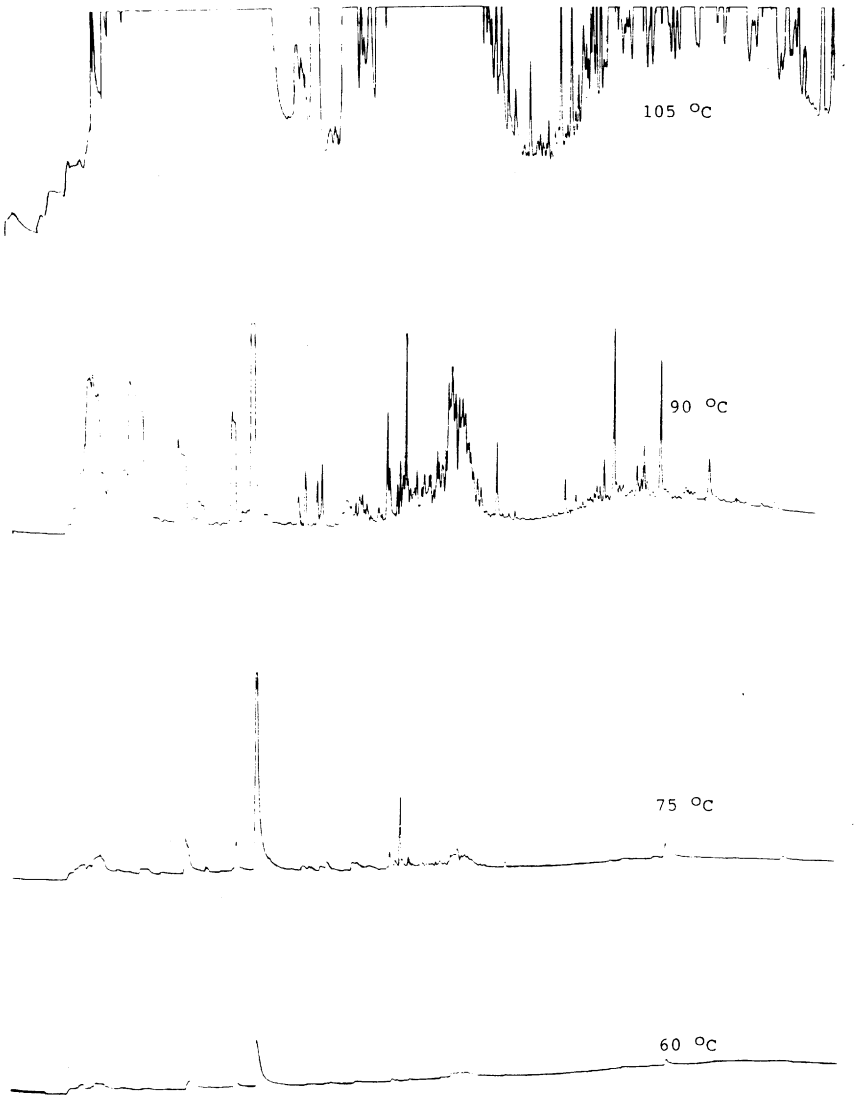

Fig. 2.23 Head-space FID chromatograms of sample IV run using different preheating temperatures, preheating time 15 min.

Table 2.12 The amount of hexanal in the head-space/ppb at different temperatures.

Sample	60 °C	75 °C	90 °C	105 °C
I	0	5	13	78
II	0	5	55	110
III	10	283	775	6027
IV	77	391	975	7948

Table 2.13 The predicted odour intensity of the samples at different temperatures (0-3).

Sample	60 °C	75 °C	90 °C	105 °C
I	0.4	1.2	1.7	1.8
II	0.4	1.2	1.7	1.8
III	1.7	1.9	2.0	2.1
IV	1.8	1.9	2.0	2.1

From these results it can be concluded that it is possible to produce food-packaging boards of similar quality regarding odour impact using different board machines. Further it can be concluded that the use of recycled fibres represents a potential hazzard to increase the volatiles and odour level when used in food packaging board.

Case 2

A total of 55 tons of liquid packages collected from two different areas in 1991 and 1992 in West Germany has been pulped and analysed. The raw material was used for coreboard. To be able to judge the quality of the fibre raw material for other production purposes samples were taken after the pulping stage. The pulping of the present batches was started after the removal of the PE-coating with fresh water in the pulper. The pulp samples are noted as I and II representing the different collection districts. As a reference sample

for these samples a mill waste sample of bleached liquid packaging board (rejected PE-coated waste of sample I in case 1) was used. This was pulped in the same way with fresh water in the pulper at the initial stage. The samples were filtered giving fibre cakes of approx. 10 g in dry mater. These were air dried over night and a part of them were analysed immediately after the drying while other parts were analysed after one and two weeks storage storage periods when the fibre cakes had been exposed to laboratory air at 23 °C and visible light. The chromatograms of the samples recorded at 90 °C are presented in fig. 2.24. The DCM-extract contents and the metal contents of the samples are given in Table 2.14 The behaviour of the predicted odour levels for the samples are plotted in fig. 2.25.

Table 2.14 The DCM-extract and metal contents of the samples.

Sample	DCM-extract %	Fe ppm	Cu ppm	Mn ppm
Reference	0.29	224	< 1	3
I	0.34	206	7	7
II	0.38	305	14	17

From these results, considering the reference sample it can be observed that recycling of mill waste does not lower the odour level of the board, (compare to sample I in case 1 above). In all chromatograms we can see a background compound 2-methyl-propene, trimer which is a compound originating from atactic polypropylene used as hot melt wax. This is a background compound related to the process and should not be interpreted to belong to these batches of recycled fibres. The growth of the aldehyde amounts during the oxidation differs between the reference and collected waste samples. The reference sample represents the typical pattern observed in oxidized board samples indicating that the precursors are the wood extractives, preferentially linolic acid. In the collected waste samples nonanal is the dominating aldehyde suggesting that the distribution of the precursors or the oxidation mechanism differs from those observed for the reference sample. Even if the DCM-extract content presented in Table 2.14 is only a sum parameter, it is likely to conclude that the amount of precursors varies between the batches.

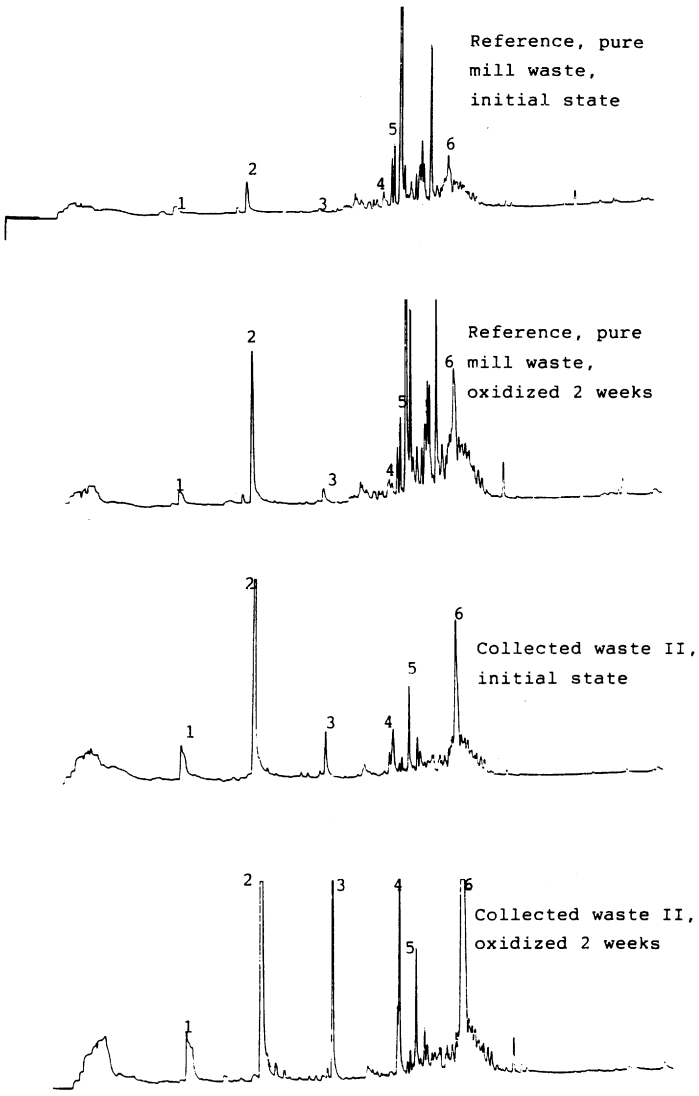

Fig. 2.24 The head-space chromatograms of reference pulp and collected waste II in their initial state and after 2 week autoxidation. Preheating conditions 90 °C 15 min.

The variation in extract contents might also indicate some residues of milk fat in the recycled pulp, giving thus another distribution of aldehydes. Further the metal contents between the reference sample and the collected waste samples differ probably due to printing ink residues giving possibilities to other autoxidation mechanisms of the precursors.

Although the calculations of the predicted odour intensities relies only on pentanal and hexanal the recycled waste gives higher odour levels. The use of recycled pulp for food-packaging board includes thus several potential risks to raise the odour level of the board.

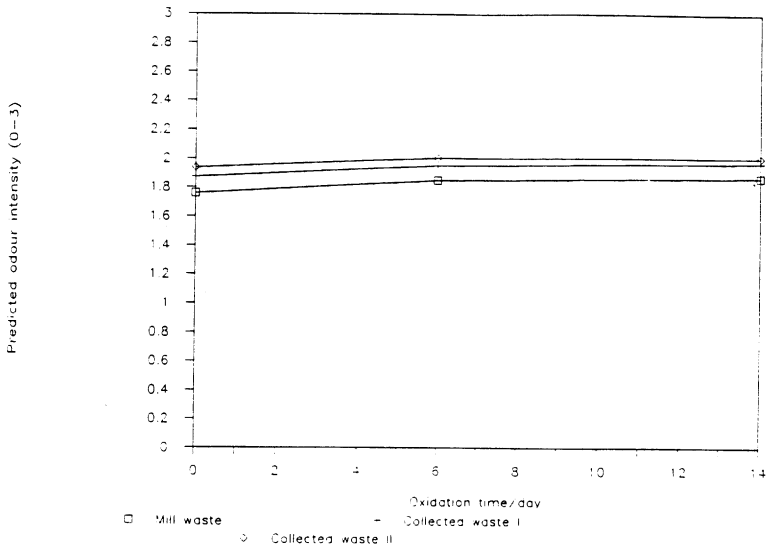

Fig. 2.25 The behaviour of the predicted odour intensity for the Collected waste I, Collected waste II and Reference samples as a function of oxidation time.

3 SUMMARY

Information about volatile compounds originating from board can be obtained by both instrumental and sensory means.

With instrumental analysis the most favorable methods are based on static and dynamic head-space chromatography.

The gas-phase composition above the boards varies widely depending on temperature and time exposition to the sample.

The characterization of the volatile compounds can be qualitatively done by GC/MS studies using as an aid the retention index identification.

The detection and quantification of the chromatograms were made using an inexpensive flame ionization detector. Calibration of the presented head-space method was performed by external on column injection.

Of the board types studied the general trend considering the total amount of volatiles at elevated temperatures was the following; board of bleached chemical and bleached mechanical pulp > board of unbleached and bleached chemical pulp > board of bleached chemical and unbleached chemi-mechanical pulp > board of bleached chemical pulp.

Of the volatiles studied hexanal was the dominating compound in all board types. Other volatiles of significant amount were pentanal heptanal, octanal, and nonanal.

The test design for the evaluation of odour intensity from board was a three dimensional analysis of variance, in which the main effects were board type, evaluation temperature and the judges. All main effects were found to influence the variance significantly. The addition of unbleached chemical pulp, bleached mechanical pulp or unbleached chemimechanical pulp to bleached chemical pulp tended to raise the odour level in the given order. An elevation of the evaluation temperature raised also the odour intensity.

Besides this the judge/sample and judge/temperature interactions were

found significant. These were interpreted as differences in the ways in which panelists experienced the samples.

Attention was paid in this study to the combination of instrumental and sensory results. Thus for both the instrumental analyses and the panel the samples were presented in similar glassware, in equal amounts and after the same heat treatment.

Using multiple linear regression, a mathematical function for the dependence of sensory data on instrumental data was found with a prediction error of 14.0 %. Using the PLS algorithm a prediction error of 12.3 % was found. It was also demonstrated that by properly weighting the data the model could be improved.

The evaluation of taste by instrumental means was approached by liquid chromatography. By ion-chromatography mono- and divalent inorganic cations and anions were analysed from water, exposed to board by soaking. The amounts that were found were small and can hardly be considered to be responsible for the recorded off-taste intensity.

The board was found to act in this test as an ion exchanger changing monovalent ions to divalent, preferably Na^+ to Ca^{2+}.

Reverse phase liquid chromatography with UV-detection and sensory evaluation, revealed vanillin to be one of the components extractable with water from board. From a two dimensional analysis of variance of the taste test data it could be concluded that the addition of unbleached chemical pulp, bleached mechanical pulp or unbleached chemimechanical pulp to bleached chemical pulp tended to raise the off-taste level in water in the given order. Further it could be concluded that differences in the response to taste intensity existed between panelists.

Promising results were found in combining sensory taste results with RPLC data. The resolution power of LC and the sensitivity of UV-detection were the limiting factors that should be overcome for a more efficient use.

The potential of hyphenated chromatographic techniques was demonstrated in the identification of water soluble compounds

originating from the board. The head-space GC method was used in two cases to estimate the odour level of recycled material compared to reference material. In both cases the recycled pulp or boards produced of recycled pulp possessed a higher odour level than the reference samples.

4 FINAL CONCLUSIONS

Board samples, identically prepared and pretreated have been analysed both using instrumental methods and by sensory evaluation. Multiple regression analysis and PLS-regression models were used to compare head-space GC and sensory results. Correlations were achieved with prediction errors of 14.0 % and 12.3 % respectively. In these models aliphatic aldehydes, preferentially pentanal and hexanal, were of significant importance. As they also have low odour threshold values and appear in the gas phase above board at ambient temperatures in concentrations near their threshold values and at elevated temperatures in concentrations above their threshold values, it can be concluded that they are to a significant degree responsible for the odour impact perceived from board. Further it can be concluded that hexanal is the dominating volatile aldehyde originating from pulp and paper produced for food packaging purposes. The use of recycled fibres may change the composition of volatiles originating from the packaging board considerably.

Although a sensory panel is a very efficient tool for evaluating odour in pulp and board, there may exist situations when there is no panel at hand for e.g. in small production units, evening and night shifts in continuous production, during summer vaccation periods etc. There may also exist cases when not enough sample is available for carrying out sensory evaluation properly for e.g. comparisons of competitive samples. The good correlation found in the present study between the performance of the sensory panel and the instrumental methods suggests that instrumental methods can be used.

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ODOUR AND TASTE ORIGINATING FROM FOOD PACKAGING BOARD

H Lindell

B Mulder, TNO, Netherlands
Did you study the temperature effect and the odour arising from paper products that are wetted, for example, paper towels and have you any ideas to bring the odour down from these towels if they are made from recycled fibres?

H Lindell
In this comparison I had 95% dry matter for all boards so that was very easy to do but if you have wetted samples it will of course enhance odour and you should bear this in mind when you interpret the results to mean that this may be a contributing effect.

P de Clerck, Avebe (Far East) Pte Limited, Singapore
I would like to ask a question about the effects of the additives that are used in paper and board. You identified that the fatty acids are a major source of hexanals and pentanals and that quite small presences of these materials cause significant taste and odour from the board. One of the major additive applications used in making this board is surface sizing. The type of starch applied can be a cereal starch or root or tuber starches. When you use cereal starches they contain about 0.8% fat by weight and 50% of it is unsaturated. These materials will naturally absorb and hold a lot of the odour from the compounds when used for food packaging. Also cereal starches contain a significant amount of protein, 0.4% by weight which gives these products their characteristic smell and this we find often reflects back in board treated with such materials.

Have you done any work on the aspect of surface applications, coatings, surface sizings on the odour levels of the board?

H Lindell

In this study I did not focus on it but I have checked different chemicals that we use in our company, among them starch. I find that starch from potatoes is quite good. You can see a difference in the taste level between starch based on corn and potato. In chromatograms this effect is more difficult to see. We have run chromatograms of different kinds of sized board the variation in the aldehydes from case to case might of course partly come from the fat in the starch.

Dr W Raverty, AMCOR Research, Australia

Our results suggest that aliphatic aldehydes are also a significant product of ozone bleaching. Have you any data that suggests that ozone bleached pulps going into papers will be a more significant source of odour and taint for food packaging grades.

H Lindell

The bleaching breaks down the fatty acids and we have the aldehydes at the bleaching stage. They are removed by washing after bleaching and it hasn't caused any rise of the odour level that I have knowledge of but, if they are not washed away they might contribute.

C Soremark, Assi Kraftliner, Sweden

Does the pH of the paper influence the taste or the odour or rather the pH at the moment of production of the paper?

H Lindell

If you think of neutral sized and rosin sized papers then there isn't a big difference between them. If you think of using CTMP the production at lower pH values will contribute much more with extractives to the system then if you produce CTMP at a higher pH values with which you are removing your extractives and gain more favourable results