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Food Chemistry 71 (2000) 319–326

Food Chemistry

www.elsevier.com/locate/foodchem

Comparison of methods of analysis of time-intensity data: application to Scotch malt whisky

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Received 4 January 2000; received in revised form 12 April 2000; accepted 20 April 2000

Abstract

Time-intensity (TI) data typically consist of assessments at 1 s intervals of a set of samples by a panel of assessors. Some means of summarising the data must be found, and the data must be represented in a way which allows comparisons between samples. Parameters such as maximum intensity, time to maximum, and total duration are typically used and samples compared by analysis of variance. Thirteen assessors scored sweet taste intensity for 60 s, for 20 whisky samples drawn from 5 types of cask after 4 maturation times. Scaling and then averaging of the transformed data provided the best summary, and ante-dependence modelling showed that the intensity value at each time-point of the curve was dependent on the previous five time-points. Small effects of cask type and maturation time were found on sweet taste in whisky samples.

Keywords: Ante-dependence modelling; Cask; Flavour; Maturation; Oak; Sweetness

1. Introduction

In time-intensity (TI) measurement a single sensory characteristic, or occasionally two characteristics (Zimoch & Findlay, 1998), is tracked as it changes over a period of time. The technique was primarily developed as a means of studying the persistence of tastes, such as sweetness, but has since been applied to many other characteristics (Cliff & Heymann, 1993). In a recent application, an increase in maximum intensity and duration of sweet taste has been reported in TI studies of Brazilian sugar cane spirit (cachaça) maturing in American oak for four years (Cardello & Faria, 1999). The spirit showed correlated decreases in intensity and duration of alcoholic and aggressive flavours, and an increase in wood flavour. For sweet taste, however, there was relatively little increase in intensity after 2 years, but a substantial increase in duration. It appeared, therefore, that a change in the spirit was causing an increase in the persistence of the sweet taste, without substantially increasing its maximum intensity.

Distilled alcoholic beverages are commonly matured in wooden casks before sale and consumption, and in some cases this may be a regulatory requirement (e.g. EC, 1989). In the case of Scotch whisky, a minimum maturation period of 3 years is required in wooden casks not exceeding 700 l capacity (SI, 1990). Scotch whisky is not normally matured in new casks, and it is now common practice for Scotch whisky distillers to use casks of American oak (*Quercus alba* and other species) which have previously held Bourbon whiskey. Regulations in the USA specify that Bourbon whiskey must be matured in new charred oak casks (Booth, Shaw & Morhalo, 1989), and so there is a ready supply of used casks. These casks are then used several times for the maturation of Scotch malt and grain whiskies. As casks are successively used, less material is available for extraction, and the effect on the spirit is reduced (Piggott, Conner, Paterson & Clyne, 1993) until they eventually fail to provide a satisfactory maturation (Philp, 1986, 1989). Many changes take place in the spirit during maturation, and the final product has a very different flavour and aroma from the newly distilled spirit (Nishimura & Matsuyama, 1989). Some materials reduce in concentration in the spirit, through losses by evaporation through the cask wood; there is a general

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PII: S0308-8146(00)00188-6

increase in concentration as water and ethanol are lost by evaporation; material is extracted from the wood into the spirit; and reactions occur within and between components of the distillate and compounds extracted from the wood (Philp, 1986; Nishimura & Matsuyama, 1989). Increases in spicy, smooth, vanilla, woody and sweet aroma notes are among the sensory changes (e.g. Piggott et al., 1993), caused primarily by materials such as vanillin, other aromatic aldehydes and other materials extracted from the wood (Conner, Paterson, Birkmyre & Piggott, 1999).

The special features of TI data are that: the same variable is measured many times at short time intervals (repeated measures data); the measurement instruments are sensory assessors. For both credibility and precision, many (8 or more) assessors are required to measure differences between samples, yet there are large differences between assessors; however, assessors are remarkably consistent over an experiment.

In other areas of application, e.g. research on nutrition using laboratory animals, the data are often analysed at every time point using analysis of variance. This often leads to a large volume of output which gives an entirely false impression of the strength of the results since the data at each time point are clearly not independent of previous time points.

A simple, effective and frequently-used method of analysing this type of "repeated measures" data is to calculate summary measures and to analyse these by analysis of variance (see e.g. Tuorila, Sommardahl, Hyvonen, Leporanta & Merimaa, 1995). Other methods of dealing with this range from the "split-plot in time" form of analysis (when there is only a small number of points) to modelling the correlational structure either directly or by a data driven method such as ante-dependence modelling (Kenward, 1987). Ante-dependence modelling estimates the correlations between the measurements at different time-points, and can then allow for the extent of the correlation. The ante-dependence structure of data is described in terms of the order, which is the number of preceding measurements upon which a measurement depends. This allows both the incremental changes between adjacent time points and the cumulative changes to a time point to be properly analysed.

Only summary measures are directly applicable to TI data of the form seen (for example, in Fig. 2 of MacFie & Liu, 1992). Statistics such as maximum intensity and time to maximum intensity, and landmarks such as start time and time to return to zero, can easily be determined for each curve of each assessor. Other measures such as plateau time depend on being able to identify appropriate landmarks in the data and, therefore, assume that the TI curves have a similar shape for each assessor and each sample. However, this is often not the case (e.g. MacFie & Liu, 1992). It is arguable that more intensive training might reduce the differences between individuals, but individuals have their own "signatures" and it is unlikely that these could be completely eliminated (van Buuren, 1992). A comprehensive list of summary measures is given by Cliff and Heymann (1993).

Summary measures cannot always fully describe small and subtle differences between samples. It is, therefore, natural to wish to obtain an "average" curve for each sample and to compare these average curves. It was clearly shown by Overbosch, van den Enden and Keur (1986) that taking averages over samples leads to curves that do not properly represent the data.

Van Buuren (1992) proposed the use of a principal components analysis (PCA) of TI curves as a method of producing weighted averages of the curves based on a statistical criterion. MacFie and Liu (1992) suggested that the data should not be centred and might usefully be analysed separately for each replicate of each treatment thus providing many estimates of the variability in the mean curves. Dijksterhuis, Flipsen & Punter (1994) compared three different methods of PCA: (1) based on correlation (data centred and scaled), (2) based on the covariance matrix (data centred but not scaled) and (3) based on the original data (data neither centred nor scaled). They found that the analysis based on the non-centred, non-scaled data was most informative and that the analysis based on correlations provided information on the "shape" of the curve only.

Overbosch et al. (1986) devised a method of deriving an average curve which makes use of three simply determined landmarks in the data. It involves scaling the data on the intensity axis to a common intensity and on the time axis separately before and after the maximum. This method has subsequently been improved by Liu and MacFie (1990) and MacFie and Liu (1992).

Dijksterhuis and van den Broek (1995) explored the use of one scaling factor affecting both the time and intensity directions simultaneously to match the shape of TI-curves. They concluded that the method was of value but that it pointed towards two separate scaling factors, one for each of the time and intensity axes. Dijksterhuis and Eilers (1997) proposed TI curves be modelled using a prototype curve for each treatment, that is an underlying smooth curve. The observed curves are then assumed to be based on the underlying smooth curves, but to be changed in both time and intensity directions in different ways by the different assessors. This approach must determine the underlying curves, optimally constructed from 6 translated copies of a B-spline, and two sets of scale factors. The model gave a good fit to test data, but is difficult to implement and software is not readily available.

The aims of the work described here were to compare methods of analysis of time-intensity data, and to test whether the change in duration of sweet taste occurred on maturation of Scotch malt whisky in the casks typically used.

2. Materials and methods

2.1. Samples

A batch of newly distilled Highland malt spirit was filled into casks at 67.5% ethanol (Chivas Brothers, Paisley PA3 4DY, Scotland). There were five types of cask: made from new charred oak (Type 1), new plain oak (Type 2), casks previously used in the USA for the maturation of Bourbon whiskey (Type 3), Bourbon casks previously used at least once for the maturation of Scotch malt whisky (Type 4), and casks used several times for the maturation of Scotch whisky and judged to have little remaining maturation potential (Type 5). Three casks of each type were used. Samples were taken after 24, 30, 42 and 60 months, and equal volumes from the three casks of each type were combined at each time point. The corresponding 15 year old whisky bottled for sale (45% ethanol) was used as a reference.

2.2. Sensory analysis

A panel of 13 assessors scored the samples in duplicate for intensity of sweet taste using the TI module of the PSA-System (OP&P Product Research BV, 3508 SG Utrecht, The Netherlands). The scale was vertical, anchored at the foot with "not present" and at the head with "very strong". The 20 samples were randomly allocated to 4 sessions (5 samples per session), and together with the sample of 15 year old whisky were tasted in different orders balanced for order of presentation in the session and for the interference effect of the previous sample (MacFie, Bratchell, Greenhoff & Vallis, 1989) in individual booths under red light. Spirits were diluted to 23% v/v ethanol, and 10 ml was presented to assessors in 30 ml polypropylene pharmaceutical cups (Vicarey Davidson, Glasgow G5 9QJ, Scotland). Assessors were instructed to take the sample into the mouth, start the data collection run, and swallow after 10 s of a total run of 60 s.

2.3. Statistical methods

The first step in the analysis was to plot the data for each session for each assessor. This provided detailed information on each run and allowed minor aberrations in the data to be corrected (such as failure to zero the device before and after testing). It also permitted the unique patterns for each assessor to be revealed.

The first summary of treatment effects carried out was to plot both "age" and "cask" means without aligning or scaling the data.

The 624 curves for this study were summarised by the mean (equivalent to the area under the curve), the maximum, the time to first realisation of the maximum, the last zero before the maximum and the first zero after the maximum. The times from zero to maximum and from maximum to the return to zero were analysed as well as the total times. Although the analysis of variance technique is appropriate, it does not allow correction for "order of presentation" effects, nor does it allow for session effects. A more general method of analysing univariate data, the mixed model, can now be formulated and estimated in readily available computer packages. This allows assessor, replicate within assessor and sessions within replicates within assessors to be regarded as random effects. Thus, proper provision was made for the session effects noted in the preliminary graphing of the data. The fixed effects included "order of presentation" as well as the treatment effects.

The data for both replicates of the 20 treatments and for the eight replicates of the control treatment were analysed separately using non-centred, non-scaled PCA. The principal component scores for the first component thus became estimates of the treatment profile.

Using the method due to MacFie and Liu (1992) the curves were scaled on the intensity axis and on the time axis using simply obtained landmarks. For this analysis the data have been separately scaled for each session of each assessor. Thus, within a session of an assessor the same scaling factors have been used, preserving differences within a session but largely eliminating differences between sessions.

In order to obtain some information about differences between the treatment curves, it is usual to carry out analysis of variance at selected points on the scaled data. A more sophisticated technique for analysing such data is ante-dependence modelling. The correlational structure of the data is used to determine a suitable covariate model using data from previous time points. Once the order of the covariate is determined an analysis is carried out which gives for each time point the treatment effect at that time point adjusting for all previous time points and a cumulative analysis to that time point. The results were also summarised by carrying out an analysis of variance at appropriate time points at intervals determined by the order of the covariance structure such that the information from the times analysed was substantially independent.

All analyses were carried out with Genstat 5 (Numerical Algorithms Group Ltd., Oxford OX2 8DR, UK) except PCA, for which Unscrambler v 5 (CAMO ASA, N-0115 Oslo, Norway) was used.

3. Results

The aim of the analysis was to estimate the differences between the age and cask treatments free of the effects of assessors. Any information on assessors is entirely secondary though it can prove useful for training and recruiting new assessors for TI studies.

Fig. 1 shows the mean effects of assessors. There are clearly many different patterns of response. Most assessors perceived a peak 10–20 s after the start whilst others did not perceive the peak until much later. Some assessors rose very quickly whilst others rose more slowly. The intensity of the peak varied greatly from assessor to assessor, from as high as 350 to as low as 50. Most assessors declined slowly from the peak but others declined more sharply.

Figs. 2 and 3 give the first crude estimates of the treatment effects. The twin peaks are an artefact and are due to the fact that the maximum was reached at different times by different assessors. Also, since the assessors showed large differences in the maximum intensity, these curves give large weights to the information from assessors with high peaks. Consequently, these aggregate curves were not truly representative of the individual curves.

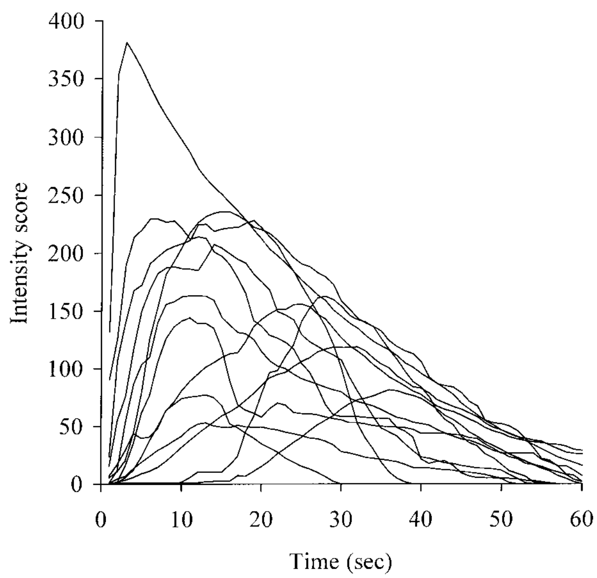

Fig. 1. Mean TI curves for sweet taste for eight replicates of control whisky sample for each assessor.

Table 1 gives the results of fitting a mixed model to sensory data from each individual TI profile. For all variables reported in this table there were large differences between assessors and some evidence of smaller differences between sessions within assessors. There was no statistical evidence of differences between age and cask treatments for the mean (area under the curve), the maximum or the time of first maximum. For subsequent analysis landmarks were used: time to last zero before the peak (T_0~below) and time to first zero after the peak (T_0~after) so these variates were also analysed. Again there was no evidence of treatment effects but a great deal of evidence of assessor and session effects within assessor. There was slight statistical evidence of a difference between age treatments for the time differences. The 60 months value was greater than the control and 30 months values which were greater than the 24 and 42 month values.

The mean curves after PCA are given in Figs. 4 and 5. The lack of smoothness evident in Figs. 2 and 3 is less evident, but still a noticeable feature of these graphics. An analysis of variance was carried out on the first component scores (Table 2). Only at 8 s is there evidence of statistical differences for a cask. When the results for the casks most like those used by Cardello and Faria (1999) (new charred oak, Type 1, and new plain oak, Type 2) were considered separately, the PCA analysis (Table 2 and Fig. 5) gave a statistically sig-

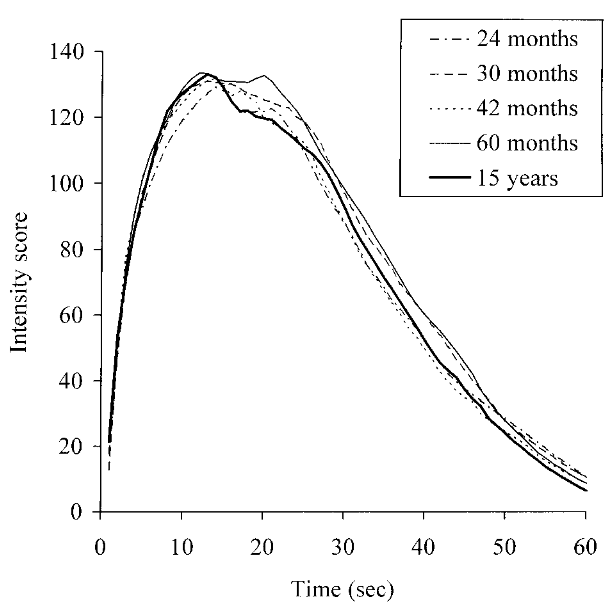

Fig. 2. Mean TI curves for sweet taste for whisky samples of different ages before scaling.

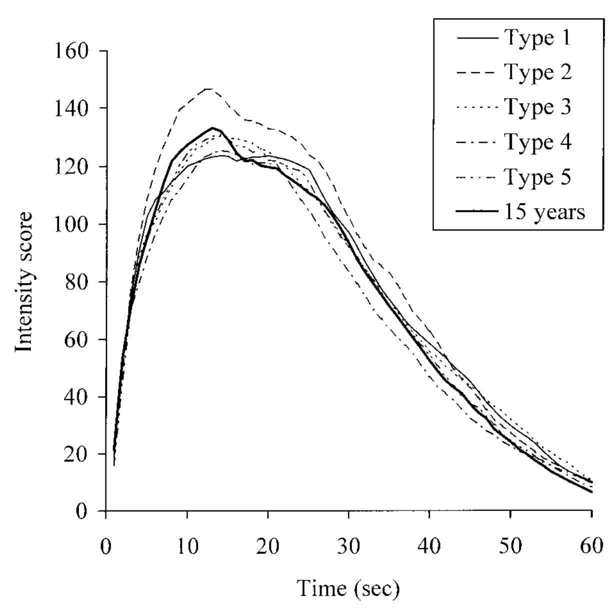

Fig. 3. Mean TI curves for sweet taste for whisky samples from different cask types before scaling.

Table 1
Means and standard errors of differences (S.E.D.) for (a) age and (b) cask effects from fitting a mixed model to data before scaling

(a)		Age (months)					
	Control	24	30	42	60	S.E.D	
Summary statistics							
Mean	74.0	72.7	79.0	73.9	79.6	3.60	ns[a]
Maximum	192	187	199	188	193	6.7	ns
Time of maximum	16.7	17.6	17.4	17.7	18.0	0.63	ns
Landmarks							
T_0~before	4.21	4.12	4.15	4.38	3.94	0.343	ns
T_0~after	45.2	43.9	45.2	44.0	46.1	0.97	ns
Derived variates							
T_{max}–T_0~before	12.5	13.4	13.2	13.4	14.0	0.64	ns
T_0~after–T_{max}	28.5	26.2	27.8	26.3	28.1	1.02	ns
T_0~after–T_0~before	41.0	39.7	41.0	39.7	42.1	1.01	*

(b)		Cask						
	Control	New charred	Plain	Once used	More than once used	Downgraded	S.E.D.	
Summary statistics								
Mean	74.0	77.1	83.5	76.2	69.6	75.2	4.09	ns
Maximum	192	192	202	192	181	192	7.7	ns
Time of maximum	16.7	17.8	16.9	17.8	17.8	18.0	0.72	ns
Landmarks								
T_0~before	4.21	4.45	4.23	4.08	3.97	4.01	0.394	ns
T_0~after	45.2	45.9	45.1	44.4	43.6	44.9	1.11	ns
Derived variates								
T_{max}–T_0~before	12.5	13.4	12.7	13.7	13.8	14.0	0.72	ns
T_0~after–T_{max}	28.5	28.0	28.3	26.5	25.8	27.1	1.14	ns
T_0~after–T_0~before	41.0	41.5	40.9	40.3	39.5	41.0	1.15	ns

[a] ns = not significant.
* = $P < 0.10$.

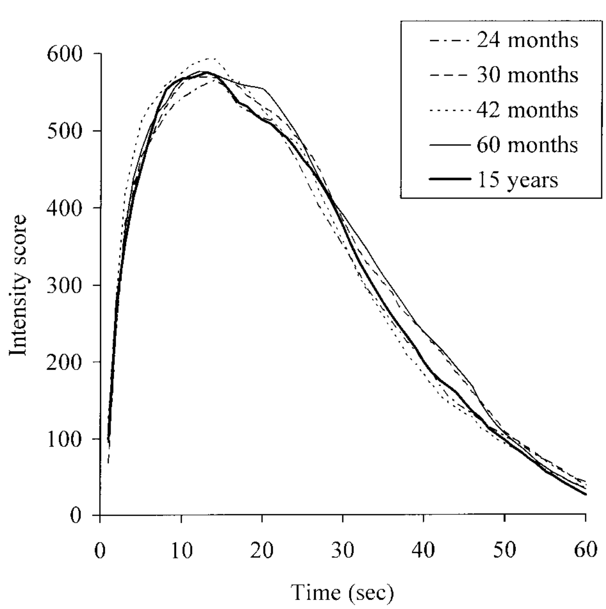

Fig. 4. Mean TI curves for sweet taste for whisky samples of different ages after averaging across panel by PCA.

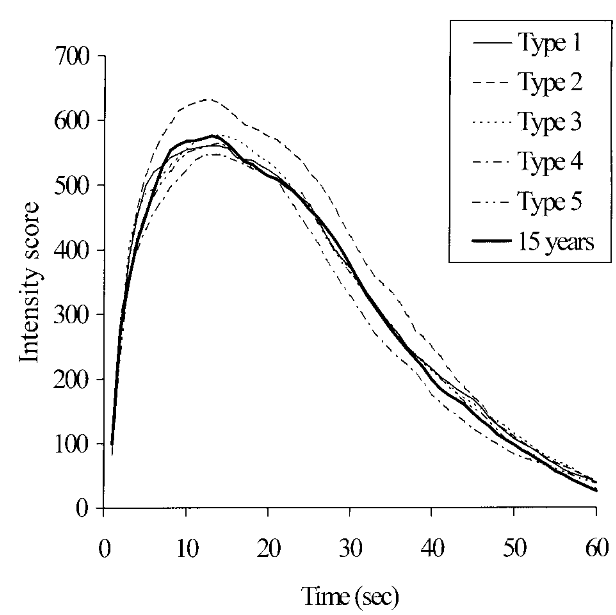

Fig. 5. Mean TI curves for sweet taste for whisky samples from different cask types after averaging across panel by PCA.

nificant difference at 13 s (561 v 631, sed = 25.2), 18 s (538 v 589, sed = 17.6) and 23 s (493 v 548, sed = 20.1) but not at 28 s. However, a statistically significant age difference did not emerge until 18 s — 501, 601, 580 and 573 (sed = 24.8) for 24, 30, 42 and 60 months ageing. At 23 s it was 465, 571, 514 and 533 (sed = 28.5) and at 28 s

Table 2
Means and standard error of differences (S.E.D.) for (a) age and (b) cask effects from ANOVA of selected points on adjusted data using scores from PCA

(a)	Age (months)						
Time	Control	24	30	42	60	S.E.D.	
8	553.7	521.1	531.6	559.9	536.7	27.28	ns[a]
13	575.9	561.4	569.8	593.3	576.4	27.78	ns
18	531.7	526.5	546.3	553.3	558.3	24.66	ns
28	418	384	426	399	421	36.20	ns
38	231.5	224.8	260.2	207.4	265.8	30.01	ns

(b)	Cask							
Time	Control	New charred	Plain	Once used	More than once used	Downgraded	S.E.D.	
8	553.7	542.6	595.8	530.5	496.7	521.2	28.75	*
13	575.9	561.2	631.5	574.6	547.0	561.9	29.28	ns
18	531.7	538.3	589.0	552.8	524.6	525.7	25.99	ns
28	418	402	468	404	366	399	38.20	ns
38	231.5	237.4	275.2	237.6	209.8	237.7	31.63	ns

[a] ns = not significant.
* = $P < 0.10$.

the effect was not statistically significant. Thus the peak intensity developed from 24 to 30 months ageing and then fell slowly away. There was also evidence that the peak intensity was more persistent at 30 months ageing.

The method described by MacFie and Liu (1992), which was developed from earlier work by Overbosch et al. (1986), was used to standardise the curves. The curves were centred using the maximum intensity and scaled on the intensity axis, and also on the time axis by two scaling factors: one from the time of last zero before the peak to the time of the peak (T_{max}–T_0~before) and one from the time of the peak to the first zero after the peak (T_0~after–T_{max}). For this analysis each individual's data were separately scaled for each session. The cost of so doing is that information between sessions is largely lost, whilst information within sessions is preserved. The resulting curves are given in Figs. 6 and 7. The graphics give information about the response from approximately 10 s before the peak until 20 s after the peak. The time scale is thus different from the previous figures, which have an expanded time scale to cope with different patterns of response between assessors. As would be expected, the shapes of the curves are slightly different from the shapes shown after the data treatments previously discussed. This is not necessarily a disadvantage of the method, but it is a consequence of choosing a different way of aggregating the individual curves.

Differences between treatments were tested by carrying out an analysis of variance on the centred and scaled data (Table 3). However, multiple analyses of variance of repeated measures data such as these carries the risk of over-interpreting the results. A convenient way of handling the correlations is to use ante-dependence modelling (Kenward, 1987). When this was carried out it was found that each measurement was dependent on the previous 5 measurements. When an analysis of variance was done for each time point allowing for the previous 5 time points the cumulative effects of the cask at the peak point were found to approach statistical significance. These results are conveniently examined by an analysis of variance at-5, 0, 5, 10 and 15 s from the peak. Although there were small differences between cask types in the direction which would be expected (i.e. newer casks showed higher intensity scores for sweetness after the peak) they were not statistically significant.

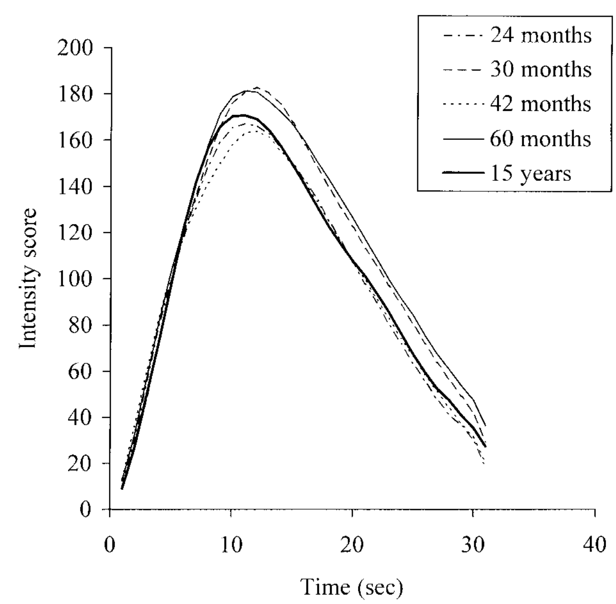

Fig. 6. Mean TI curves for sweet taste for whisky samples of different ages after scaling (MacFie & Liu, 1992).

4. Discussion

There were substantial differences between assessors (Fig. 1), and in most cases these differences swamped any effects which might have been due to the treatments (maturation period and cask). However, all the methods of analysis used here showed some minor differences between casks and between maturation periods. The analysis of summary statistics is the conventional approach to TI data, and is the simplest to implement, though as discussed above many authors have pointed out potential deficiencies. From the literature the PCA method, which scales only in the intensity direction, appeared to be the most promising but we were disappointed that it did not produce clearer results. The major problem was the variation in time to maximum intensity, which cannot be accommodated by this method. The MacFie and Liu (1992) method produced more credible response curves than simple averaging or PCA, without the artefact of the twin peaks shown by some of the samples. The apparent difference in the shape of the curves is misleading, since they appear different in comparison with simple averaging or PCA, neither of which provides a very good representation of the individual curves. However, the results depend on the selection of landmarks in the data, which may be rather arbitrary. Further research into this method would be useful, possibly using an iterative process for selecting the scaling factors using the existing average curves as first iterations. Only the method proposed by Dijksterhuis and Eilers (1997) appears to offer any advantage, but in the absence of reliable readily available software its utility could not be tested.

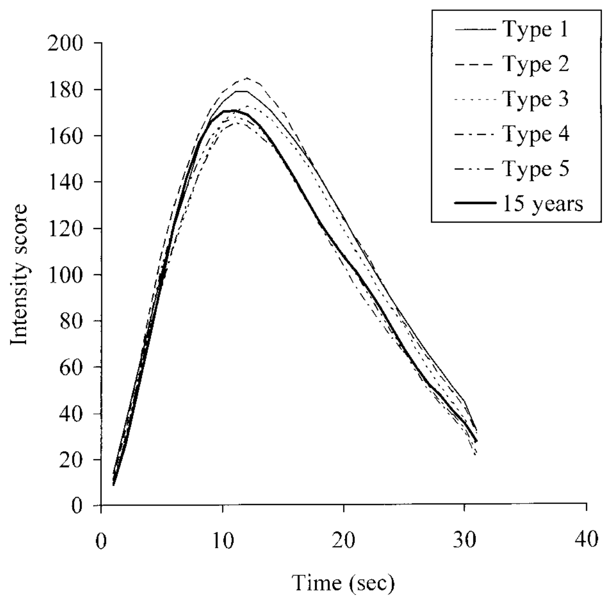

Fig. 7. Mean TI curves for sweet taste for whisky samples from different cask types after scaling (MacFie & Liu, 1992).

The origins of a sweet taste in matured spirits to which sugars have not been added are problematic, since the new distillate contains none of the non-volatile materials normally regarded as conferring a sweet taste. Small amounts of sugars are extracted from the wood during maturation, and after 12 years a glucose concentration of approximately 120 mg l^{-1} proof spirit has been reported (Reazin, 1983) in whisky stored in new charred casks, with arabinose at a slightly higher concentration and others at lower levels. Total sugars in this case seemed to be around 0.5 g l^{-1}, but detection thresholds for sugars in water are typically around 5 g l^{-1} (Amerine, Pangborn & Roessler, 1965) and so it

Table 3
Means and standard error of differences (SED) for (a) age and (b) cask effects from ANOVA of selected points on adjusted data using MacFie and Liu's (1992) method

(a)	Age (months)						
Time from peak	Control	24	30	42	60	S.E.D.	
-5	121.9	118.1	119.8	119.0	122.4	8.20	ns[a]
0	170.4	167.1	180.9	163.2	181.3	8.53	*
5	140.5	142.6	158.1	141.4	159.5	9.25	ns
10	101.2	97.8	113.0	99.7	117.1	9.43	ns
15	59.6	55.9	72.0	58.6	75.4	8.37	*

(b)	Cask							
Time	Control	New charred	Plain	Once used	More than once used	Downgraded	S.E.D.	
-5	121.9	120.8	130.0	112.7	113.7	122.1	8.64	ns
0	170.4	179.0	182.8	170.3	165.7	167.9	8.99	ns
5	140.5	157.4	160.2	154.2	140.8	139.4	9.75	ns
10	101.2	114.8	115.4	110.0	94.2	100.1	9.94	ns
15	59.6	72.6	70.4	67.1	59.8	57.5	8.82	ns

[a] ns = not significant.
* = $P < 0.10$.

seems unlikely that the sugar content can make any direct contribution to flavour. It seems more plausible that a sweet sensation, due to volatile aromatic aldehydes such as vanillin or other compounds, is perceived as ''flavour by mouth'', and that assessors recorded it as a sweet taste when asked to do so.

In the present work, samples of the new distillate and of the 12 month matured spirit were not available for comparison, but Cardello and Faria's (1999) results showed a substantial increase in sweet taste duration between 24 and 48 months in oak casks. The increase in intensity halted at about 24 months, so the 24–60 months period examined here should correspond to the increase in duration previously found. Small differences were shown by analyses of raw data (effect of age on total duration, Table 1), of principal curves (cask effect on intensity at 8 s, Table 2) and of scaled data (effect of age on intensity at peak and 15 s later, Table 3), but the results were not as striking as those presented by Cardello and Faria.

5. Conclusion

Differences between assessors presented a major problem for the methods of analysis used here, none of which was clearly superior. There is a clear need for further methodological studies to identify the most appropriate method, or methods, of analysis which can then be incorporated into software, but the software will not be developed until there is some agreement on the best method to use. In this case, MacFie and Liu's (1992) method produced more credible response curves than others, but it depends heavily on the selection of appropriate landmarks. In combination with ante-dependence modelling it provides a powerful method of summarising and analysing the data. There was some indication that the spirit samples which had been in cask for a longer time, or in the casks which provided higher levels of extracted materials, showed higher maximum sweet taste intensities, and longer durations of sweet taste.

Acknowledgements

The work of BioSS is supported by funding from the Scottish Executive, Rural Affairs Department (SERAD). The authors wish to thank Chivas Brothers Ltd for financial support and for the provision of samples.

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