Application of Weibull Hazard Analysis to the Determination of the Shelf Life of Roasted and Ground Coffee

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Roasted and ground coffee was stored at constant O_2 partial pressure (0.5–21.3 kPa), a_w (0.106–0.408) and temperature (4–35 °C). Product acceptability was monitored by use of a modified Weibull Hazard sensory method where the end of shelf-life was the time at which 50 % consumers found the product unacceptable. The effect of O_2, a_w and temperature was studied from a kinetics standpoint. Oxygen increase from 0.5 to 21.3 kPa accelerated deterioration 20-fold. A water activity increase of 0.1 led to a 60 % increase in deterioration suggesting non-enzymatic browning activity, while a temperature increase of 10 °C rose the rate of deterioration about 15–23 %. The activation for shelf life was ≅ 13 kJ/mole indicating diffusion within the glassy matrix is controlling deterioration.

Keywords: Weibull Hazard; shelf-life; coffee; staggered design

Introduction

Shelf-life can be defined as the time until a product becomes unacceptable to consumers under a given storage condition. Consumer acceptability is usually determined by means of 'consumer tests' using a large number of untrained tasters in which samples with various ages are tasted together (Meilgaard *et al*., 1991). This is accomplished by using samples of product manufactured at different times or by freezing aged samples to stop deterioration. Both techniques introduce undesired error, i.e. batch to batch variability or the continuation of the deterioration during the frozen storage.

Another technique used in industry for shelf-life determination is team judgment. It is frequently applied in the early stages of product development, where prototypes are evaluated during the storage by a team familiar with the product. The main drawback is that results are limited to the tasting team making necessary the validation with consumers prior to launching.

The use of correlation between trained panel evaluation and consumer response has been reported in the literature (Fritsch *et al*., 1997). First, a team trained to recognize deteriorating attributes quantifies changes in their intensity during storage. Samples are frozen and then used in a second test where consumer acceptability is determined. Consumer response is correlated to trained panel data to determine the limits for the intensity of deteriorating attributes in product at the end of consumer shelf-life. The shelf-life of new samples is found equal to the time when intensity limits for deteriorating attributes are detected by the trained panel. The main advantage of the technique is that one set of consumer data is used multiple times when correlating to trained panel assessment. The main disadvantages are that results depend on the type of the scale used for measuring the deterioration (Shepherd *et al*., 1988) and that sample storage introduces error which in turn is amplified when coupling two sets of sensory data. The technique has created intense debate in the literature. Fritsch *et al*. (1997) and sensory psycho-physicists support the use of trained tasters to predict consumer sensory responses (Moskowitz, 1996, 1997), where as statisticians do not (Dugle, 1996).

An ideal method would be to use samples of one batch of product and evaluate them at various times during the storage with a large number of consumers. This is too costly and time consuming. One way to simplify this is to

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use staggered sampling designs in which reduced testing is done in the beginning of the storage focusing on samples close to the end of shelf-life. This approach has been extensively used in the mechanical and electrical industries (Johnson, 1964).

Gacula (1975) proposed the use of staggered sampling designs for shelf-life studies in foods. The technique consists on evaluating an increasing number of samples as storage time progresses. The main advantages of a staggered design over the methods described above are that all samples come from one batch, product is tasted throughout the whole storage period until unacceptable and that untrained consumers can be used for the evaluation.

Staggered sampling methods have been used to determine the shelf life of luncheon meats (Gacula, 1975), cassava flour (Shirose *et al*., 1978), breakfast cereal (Pickering, 1984), ice cream (Wittinger & Smith, 1986), refrigerated meats (Andujar & Herrera, 1987), frozen foods (Tomasicchio, 1989), cottage cheese (Schmidt & Bouma, 1992), pasteurized milk (Duyveste, 1997) and sausages (Thiemig, *et al*., 1998). Data analysis involves plotting hazard values versus time and using the Weibull distribution to determine shelf life equal to the time at which 50% of consumers find product unacceptable. Labuza and Schimdl (1988) and Fu and Labuza (1993) called the technique the 'Weibull Hazard Method'. More details of this method can be found in the literature (Nelson, 1969, 1972; Gacula & Kubala, 1975; Gacula & Singh, 1984).

The objective of this paper is to present shelf life results for roasted and ground (R/G) coffee obtained with the Weibull Hazard method and to determine the effect of O_2 partial pressure, a_w and temperature on shelf-life.

Material and Methods

Coffee samples

Freshly roasted Colombian Arabica coffee beans were ground in a McGarveys Commercial Grinder to 'drip brewing size' (McGarveys, Minneapolis, MN). **Table 1** shows storage conditions used for the study. Levels of O_2 partial pressure, T °C and a_w were selected to be representative of real world situations frequently found in the storage of R/G coffee. Storage conditions were combined in a full factorial design.

Table 1 Storage conditions for sensory shelf life determination in R/G coffee

Factor	Levels
O_2 partial pressure (kPa)	0.5, 1.0, 3.0, 5.1, 10.1, 21.3
a_w	0.106, 0.248, 0.408
T (°C)	4, 22, 35

Full factorial design: $6 \times 3 \times 3 = 54$ treatments.

Storage and sampling

Samples of 120 g of R/G coffee were weighed to 0.1 g in a Mettler PC 4400 balance (Mettler Toledo Inc., Hightstown, NJ) and placed into 1 L amber bottles. The bottles were hermetically sealed with rubber stoppers. The head space of bottles was replaced once a week with 20 L (NPT) of an O_2/N_2 primary standard mixture at a specific concentration (Genex, Minneapolis, MN). The gas mixture was humidified by bubbling through saturated solutions of NaOH, $MgCl_2$ and K_2CO_3, which have a_w similar to that of coffee with an a_w of 0.106, 0.248 and 0.408, respectively. After flushing, the internal pressure of bottles was equilibrated for 1 min to atmospheric pressure. Bottles were kept horizontally in incubators at controlled temperatures of 4 ± 0.5 °C; 22 ± 0.5 °C and 35 ± 0.5 °C. The depth of the R/G coffee layer was approximately 20 mm.

Samples were initially taken once every 2/6 of the shelf-life predicted from data published by Clinton (1980) using algorithms developed by Cardelli (1997). The accelerated part of the study started after 50% of samples were found unacceptable. Then, samples were taken once every 1/6 of predicted shelf life. The experiments were conducted until six sample times were evaluated or the storage time reached 7 months, whichever came first.

Sensory evaluation

Samples of 20 g of R/G coffee were removed at each sampling time, weighed to the 0.1 g on a Mettler PC 4400 balance (Mettler Toledo Inc., Hightstown, NJ) and brewed with 1 L of bottled drinking water (Kandhyoi, Minneapolis, MN) using standard coffee filter paper (Mr. Coffee Inc., Bedford Hts., OH) in a table top coffee maker (Mr. Coffee Inc., Bedford Hts., OH). The brewed coffee was kept in 1 L Thermos jars and then poured into pre-warmed ceramic cups (200 mL capacity) and covered with aluminum foil. Samples were then identified with random numbers and served to untrained tasters.

Tasters were recruited among students (50% male, 50% female, 20–31 years of age) and staff (50% male, 50% female, 23–58 years of age) of the University of Minnesota and allowed to participate in the study only if they regularly drank one or more cups of black coffee per day, without sugar or cream. Three tasters were used at the beginning of the test. The number of tasters was increased by $C = 1$ at each sampling time until half of the tasters found the samples unacceptable. After that the number of tasters for each period was increased by $C + U$, with U = number of unacceptable responses for the previous test time.

No more than eight samples per session were presented to tasters who were asked to grade the brew as being acceptable or unacceptable. Tasters were also asked to rinse their mouth with water and wait for 2 min between samples.

Data analysis

The results were transferred to a master spreadsheet for hazard calculation and then to a Weibull Hazard plot as described by Gacula and Singh (1984). The criteria for the end of shelf life was set as the time for 50% probability of untrained tasters to grade the samples as being unacceptable. This probability corresponds to a cumulative hazard of 69.3. The shape factor (β) was determined as 1/slope. The Weibull distribution is unskewed for $2 < \beta < 4$, which leads to better shelf life estimates. When the test is extended beyond shelf life, most of samples are judged unacceptable shifting β outside the optimum range. In these situations, data were re-plotted up to a cumulative hazard of 100.

Results and Discussion

Typical results and hazard values are shown in **Tables 2** and **3**, respectively. **Figure 1** shows a Weibull Hazard plot for time versus cumulative hazard. A first attempt resulted in $\beta > 4$, therefore only cumulative hazard values < 100 were plotted with $\beta = 3.4$. Shelf-life resulted in 22.5 weeks with 95% confidence limits of 20.6–24.6 weeks.

Shelf life results were analysed from a kinetics standpoint with a focus on the effect of O_2 partial pressure, a_w and temperature on the acceleration of deterioration. **Figures 2** to **4** show all the shelf life data collected in our experiments. As expected the sensory shelf life decreases with the increase in O_2 partial pressure, a_w and temperature. Among these factors, oxygen has the most critical role in the deterioration with an approximately 20-fold acceleration when increasing pressure from 0.5 to 21.3 kPa. Most of R/G coffee in cans is vacuum packaged at about 1–5 kPa of O_2. Oxygen is rapidly consumed, which provides a shelf life of approximately 2 years at room temperature (Clinton, 1980; Radtke & Piringer, 1981). But once opened, the shelf-life is less than a month (Clarke, 1993). The effect of oxygen was noticed by the tasters who were invited to add comments in the score sheets. Terms like 'fresh', 'oxidized' and 'stale' were frequently used to describe the evolution of the flavor of R/G coffee during the study.

One technique to evaluate the effect of water activity is to calculate Q_a which is the ratio of shelf life two water activities 0.1 a_w units apart (Loncin *et al.*, 1968). For R/G coffee, a_w had the second most important effect with a Q_a value of about 1.6 over the whole experimental range, i.e. an approximately 60% decrease in shelf life per 0.1 a_w increase as seen in **Table 4**. The strong a_w effect on the kinetics of shelf life deterioration may be related to the contribution of non-enzymatic browning.

The effect of temperature on shelf life was studied by determining Q_{10} values and the energy of activation (E_a) for kinetics of deterioration. **Table 5** shows that the results for Q_{10} ranged from 1.15 to 1.23, i.e. a 15 to 23% acceleration per 10 °C increase in temperature. The magnitude of E_a was $\cong 13$ kJ/mole, indicating that sensory shelf life deterioration is controlled by a diffusion process which has little temperature sensitivity (Labuza & Schmidl, 1985). Even with this low temperature sensitivity, refrigerated storage in air at 4 °C versus room temperature would give about a 44% increase in shelf

Table 3 Weibull hazard ranking table for R/G coffee at 1.0 kPa O_2, 35 °C and $a_w = 0.248$

Rank	Weeks	H value[a]	$\sum$ H
25	7.1	4.0	4.0
24	12.1	4.2	8.2
23	17.3	4.3	12.5
22	17.3	4.5	17.1
21	17.3	4.8	21.8
20	17.3	5.0	26.8
19	20.1	5.3	32.1
18	20.1	5.6	37.6
17	20.1	5.9	43.5
16	20.1	6.3	49.8
15	20.1	6.7	56.4
14	20.1	7.1	63.6
13	20.1	7.7	71.3
12	23.3	8.3	79.6
11	23.3	9.1	88.7
10	23.3	10.0	98.7
9	23.3	11.1	109.8
8	23.3	12.5	122.3
7	23.3	14.3	136.6
6	23.3	16.7	153.3
5	23.3	20.0	173.3
4	23.3	25.0	198.3
3	23.3	33.3	231.6
2	23.3	50.0	281.6
1	23.3	100.0	381.6

[a]H = Hazard value = 100/rank.

Table 2 Typical Weibull sensory data for R/G coffee (1.0 kPa O_2, 35 °C and $a_w = 0.248$)

Weeks	Acceptability																		
0.0	+	+	+																
7.1	+	+	− *25*	+															
12.1	+	+	− *24*	+	+														
17.3	− *20*	− *21*	+	− *22*	− *23*	+													
20.1	+	− *13*	+	− *14*	+	− *15*	− *16*	+	− *17*	− *18*	− *19*								
23.3	− *1*	+	− *2*	− *3*	+	+	− *4*	− *5*	− *6*	− *7*	− *8*	− *9*	− *10*	− *11*	+	+	− *12*	+	+

+: acceptable sample as assessed by an untrained taster.
−: unacceptable sample as assessed by an untrained taster.

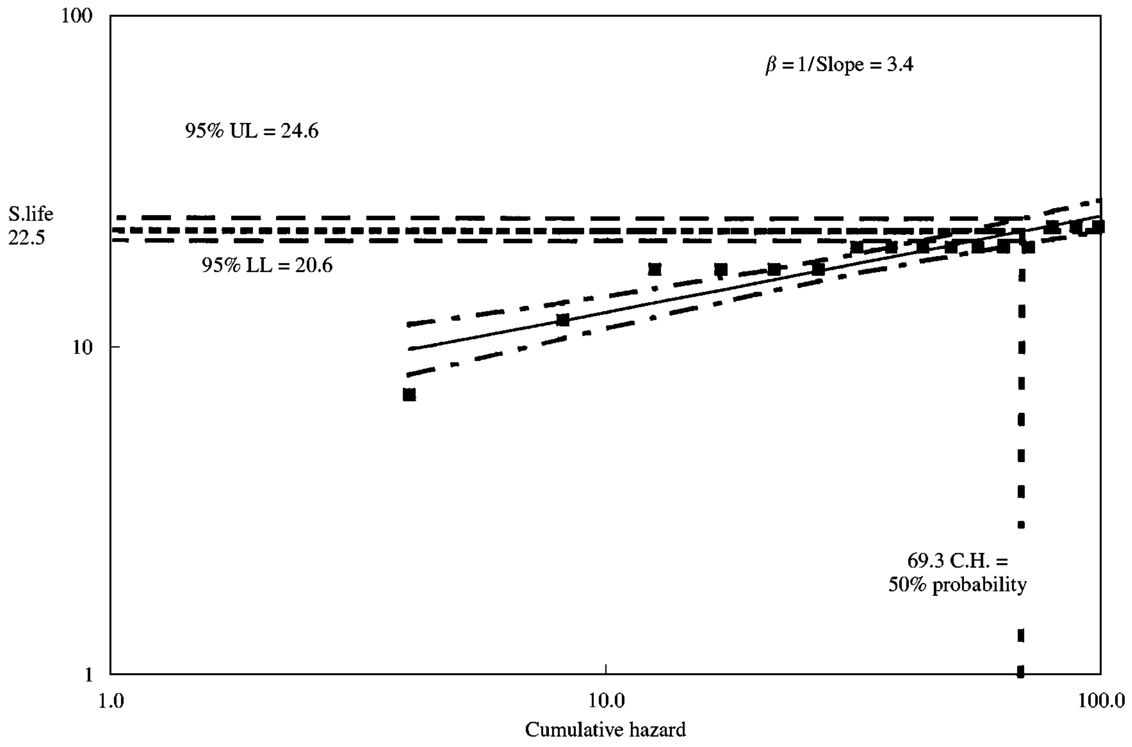

Fig. 1 Weibull hazard plot for R/G coffee (1.0 kPa O_2, 35 °C and a_w = 0.248). Cumulative hazard < 100

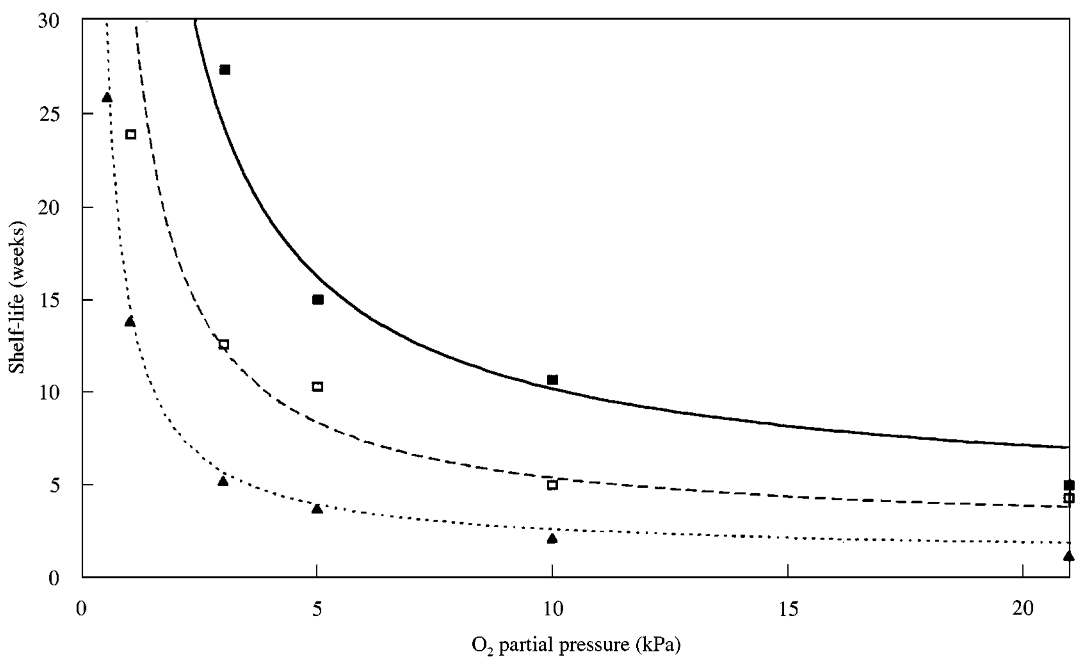

Fig. 2 Sensory shelf life of R/G coffee at 4 °C, a_w: ■ 0.106, □ 0.248, ▲ 0.408

life. Storage of an open can in the freezer (≅ − 15 °C) would increase shelf life over that at room temperature by about 70%.

The low E_as determined in our experiments could be a consequence of the fact that coffee is in the glassy state for all storage conditions (Cardelli, 1997). In this state, the limiting factor is that oxygen needs to diffuse through the matrix before it can react with the coffee lipids. Even though lipid oxidation has a high temperature dependence, diffusion controls the rate at which O_2 can diffuse to the lipid. Thus, when temperature is increased, the amount of oxygen available for reaction is what limits the reaction rate.

Conclusions

The impact of the storage factors on shelf life decreased in the following order O_2 partial pressure, a_w and temperature. Shelf life was reduced in about 20 times when

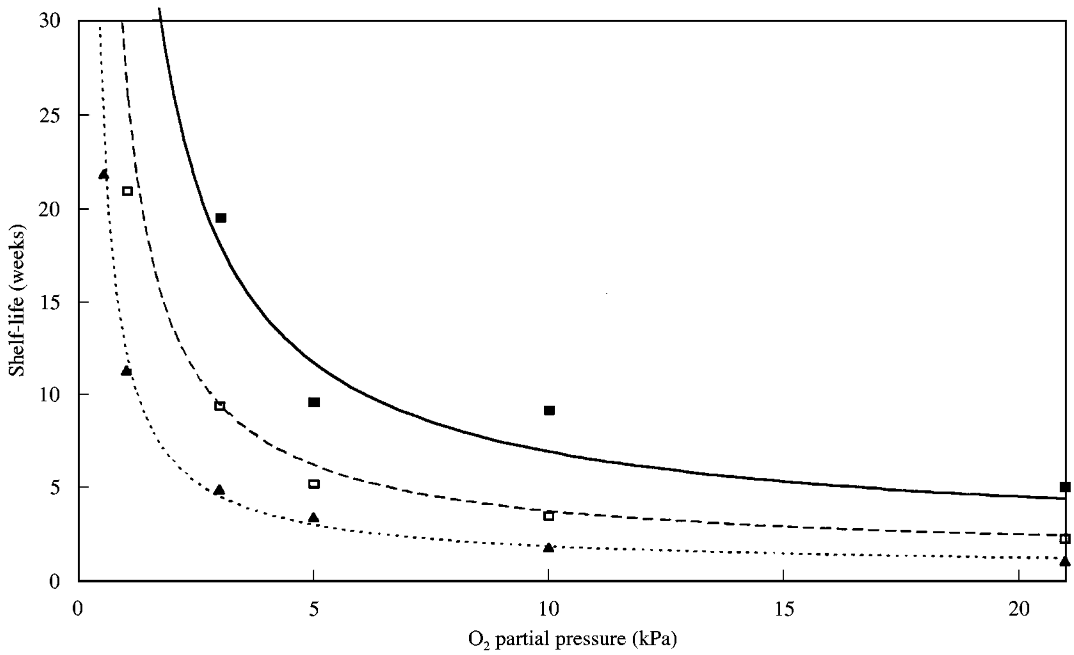

Fig. 3 Sensory shelf life of R/G coffee at 22 °C, a_w: ■ 0.106, □ 0.248, ▲ 0.408

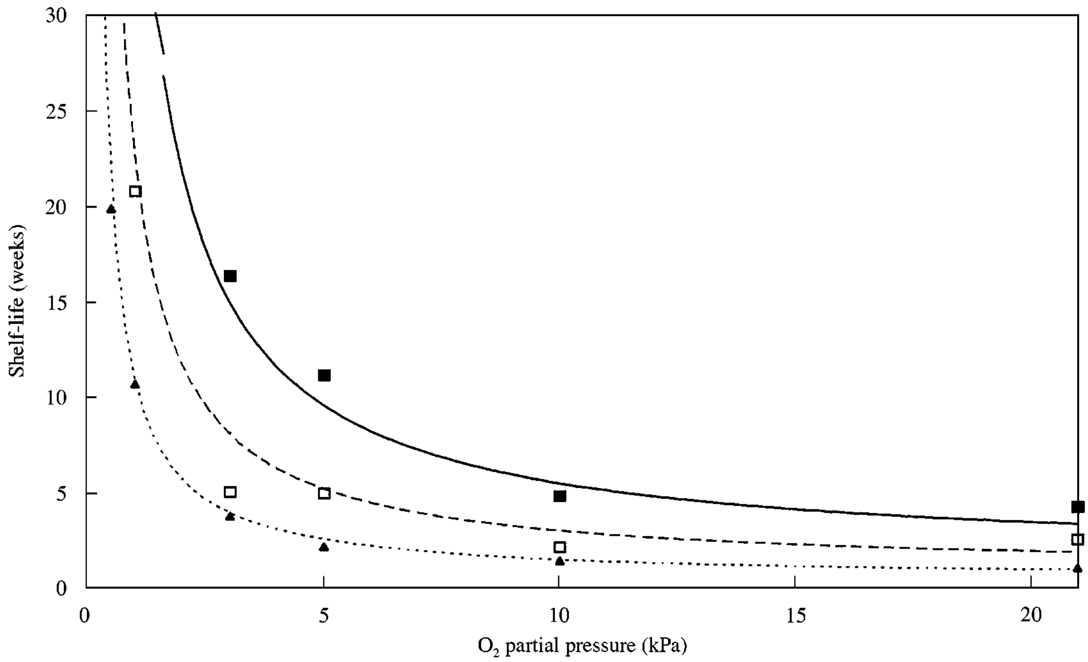

Fig. 4 Sensory shelf life of R/G coffee at 35 °C, a_w: ■ 0.106, □ 0.248, ▲ 0.408

Table 4 Water activity dependence, Q_a, for the shelf life of R/G Coffee stored at 10.1 kPa O_2 and 22 °C

a_w	Q_a
0.106	1.6
0.248	1.6
0.408	1.6

Table 5 Temperature sensitivity, Q_{10}, and energy of activation for sensory shelf life of R/G coffee stored at 10.1 kPa O_2 and $a_w = 0.248$

T (°C)	Q_{10}	E_a kJ/mole
4	1.23	14
22	1.18	13
35	1.15	12

O_2 level increased from 0.5 to 21.3 kPa, only 60% when a_w rose in 0.1 and approximately 20% per each 10 °C increment. The use of staggered tasting combined to Hazard analysis and Weibull probability distribution provided a simple approach to shelf life determination as perceived by consumers.

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