Retardation of Rancidity in Deep-Fried Instant Noodles (Ramyon)[1]

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The storage stability of instant fried noodles (ramyon) was determined by accelerated aging at 63 C with organoleptic evaluation of the onset of rancidity. Three methods of extending the shelf-life of ramyon were examined: (a) addition of 200 ppm antioxidant, butylated hydroxyanisole (BHA), t-butylhydroquinone (TBHQ), or a polymeric antioxidant (Poly-A) to the frying (palm) oil; (b) coating the inner surface of the polyethylene package with TBHQ equivalent to 200, 500 and 1000 ppm based on the oil in the ramyon; and (c) addition of a mixture of 200 ppm TBHQ and 200 or 500 ppm disodium ethylenediaminetetraacetate (EDTA) to the frying oil. When the antioxidants were added to the oil, BHA and Poly-A approximately doubled while TBHQ tripled the shelf-life of ramyon. The mixture of TBHQ (200 ppm) and disodium EDTA (500 ppm) in the frying oil quintupled the shelf-life. The inner surface application of TBHQ (200 ppm) extended shelf-life twice that of an equal amount of TBHQ in the frying oil. Rancid off-flavors developed slowest in noodles with a_w 0.3. Hexanal concentration in ramyon was a good indicator of the development of oxidative rancidity. Organoleptic evaluation showed the flavor of ramyon was objectionable when hexanal concentration reached 3.5 ppm based on the weight of ramyon (as is). The relative effectiveness of antioxidants in preventing off-flavor in ramyon could be determined from the hexanal concentration in stored ramyon.

Instant fried noodles (ramyon) are an important staple food in Asia and are growing in popularity in the Western world. The shelf-life of packaged ramyon noodle is limited generally to 4–5 mo due to oxidative rancidity, which is generally referred to as autoxidation. In the free radical mechanism of lipid autoxidation, the peroxides are fragmented into aldehydes, ketones, acids and other degradation products which are responsible for rancid off-flavors. Frankel et al. (1) analyzed the volatile carbonyl compounds in the autoxidation of linoleic acid and identified 66 mole % hexanal as a major off-flavor compound. 2-Octenal, 2-heptenal and 2,4-decadienal were minor components. In accord with other foods that autoxidize, the shelf-life of ramyon is increased by using frying oils with a high concentration of saturated fatty acids, adding antioxidants and preventing contamination with copper and iron salts. After packaging, other factors that affect ramyon stability include light, temperature, humidity and oxygen-permeability of the packaging material (2–5).

The objectives of this study were: (i) to test the influence of water activity (a_w) on the stability of ramyon during storage; (ii) to compare the shelf-life of ramyon fried in oil containing butylated hydroxyanisole (BHA), t-butylhydroquinone (TBHQ), or polymeric antioxidant (Poly-A); (iii) to compare the shelf-life of ramyon when TBHQ was applied to the inner surface of the package as opposed to its addition to the frying oil; (iv) to determine the concentration of copper and iron in ramyon and to test the effect of a chelating agent, disodium ethylenediaminetetraacetate (EDTA), together with TBHQ in retarding the autoxidation of ramyon, and (v) to determine whether hexanal can be used as an index of oxidative rancidity in ramyon.

MATERIALS AND METHODS

For ramyon production a 1:1 blend of hard wheat flour (Ross Industries, Cargill Inc., Wichita, Kansas) and soft wheat flour (King Milling Co., Lowell, Michigan) was used. Analyses of the blend gave 13.2% moisture, 11.8% protein (N × 5.7, d.b.) and 0.53% ash (d.b.) (6). Palm oil with no additives was obtained from Palmco Inc., Portland, Oregon. Sodium chloride, acetone and disodium EDTA were products of Fisher Scientific Co., Fairlawn, New Jersey. Hexanal (99%), 4-heptanone (98%) and TBHQ (97%) were obtained from Aldrich Chemical Co., Milwaukee, Wisconsin. BHA was purchased from Sigma Chemical Co., St. Louis, Missouri. Poly-A was 10% POLY-AO™-79 in corn oil with the commercial name ANOXOMER, and was obtained from Dynapol Co., Palo Alto, California.

Preparation of ramyon. Wheat flour (1 kg, 14% m.b.) was placed in a Hobart A-200 mixer (Hobart Corp., Tory, Ohio), and 330 ml of a 5.7% brine solution was added over a period of 1 min while mixing at low speed. Mixing was continued 4 min at medium speed. The mixture was given a 10-min rest period at room temperature. A laboratory noodle machine (Ohtake Noodle Machine Co., Tokyo, Japan) was used to sheet and cut the dough. The initial dough sheet was formed by passing the dough through the rolls (150 mm width, 180 mm diameter) set at a gap of 4 mm. The sheet was passed through the rolls a total of eight times in the same direction. The sheet was wound on a spool between sheeting steps, and the dough sheet was reduced to a final thickness of 1.5 mm. The final dough sheet was cut by hand into 30 cm long sheets that were fed into the machine cutter, which consisted of two pairs of slotted brass rolls with 1.5 mm gaps. The noodle strands were directed from the cutter onto a slow-moving belt so the strands formed a sinusoidal wave in the long dimension.

The wavy noodle strands (140 g, wet basis) were placed randomly in a wire basket (115 × 115 × 40 mm) fitted with a lid, and the noodles were steamed for 5 min on each side in an aluminum steamer. Immediately after steaming, the baskets were immersed in 4 kg palm oil for 70 sec at 180 C in a Belshaw 611 Fryer (Belshaw Bros. Inc.,

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Seattle, Washington). Each batch of oil (4 kg) was discarded after a single frying operation in which four baskets, each with 140 g of wet noodles, were immersed in the oil. The fried noodles were removed from the fryer and allowed to drain excess oil for 20 sec.

After cooling to room temperature, the ramyon was crushed into pieces approximately 2-5 mm long by a mortar and pestle. Ten grams of crushed ramyon were placed in glass jars (32 ml headspace), and capped for stability testing at 25 C and 63 C. The crushed ramyon showed 6.5% moisture, 10.5% protein (N × 5.7, d.b.), and 0.51% ash (d.b.) by the AACC methods, and 20.0% crude fat extracted with petroleum ether by a soxhlet for 16 hr. Hereafter, the weight of crushed ramyon is expressed on an "as is" moisture basis.

Trace metals in ingredients and ramyon. One-half gram of palm oil, wheat flour, salt or crushed ramyon was placed in a micro-kjeldahl flask for wet combustion. Concentrated sulfuric (3 ml) and nitric (8 ml) acids were added, the mixture boiled gently for three hr, cooled and diluted to volume (100 ml) with deionized water. Copper and iron were determined in the digests using atomic absorption spectroscopy (Perkin Elmer Model 603, Norwalk, Connecticut) at 324.7 and 248.3 nm, respectively. Standard solutions of copper and iron were obtained from J. T. Baker Chemical Co., Phillipsburg, New Jersey.

Effect of ramyon moisture on rancidity development. Ramyon samples with different moisture levels were prepared by equilibration with salt solutions of known water activity (a_w = 0.1, 0.3, 0.4, 0.5 and 0.6) as described by Lange (7). A cotton bag containing 30 g of crushed ramyon was suspended from a rubber stopper atop a 500-ml filtering flask that contained 200 ml of salt solution. The stopper was also fitted with a stopcock that was used in conjunction with the side tube to purge the flask with oxygen-free nitrogen after the flask was loaded with crushed ramyon. Each flask was kept at 25 C for two weeks, as recommended by Katz and Labuza (8), and then the side-arm was opened for 24 hr to allow oxygen to enter the flask. The side-arm was closed and the sample evaluated every five days by a taste panel for the onset of rancidity at 25 C. The onset of rancidity was determined organoleptically by using the threshold method (9) directly on the crushed ramyon. Five panelists compared the taste of treated ramyon with that of a control sample which had been stored at −20 C.

Accelerated aging (Schaal oven) tests on ramyon. The effect of adding antioxidant (200 ppm of BHA, TBHQ or Poly-A) to the frying oil was investigated. After frying, samples of crushed ramyon (10 g each) were placed in capped glass jars (headspace 32 ml) and stored at 63 C (10), and the onset of rancidity was determined by the taste panel.

In a second experiment, the effect of a combination of chelating agent and an antioxidant was evaluated. The control sample was fried in oil containing neither antioxidant nor chelating agent. In the treatment, the samples were fried in oil containing 200 ppm TBHQ, a mixture of TBHQ and disodium EDTA, 200 ppm each, or a mixture of 200 ppm TBHQ and 500 ppm disodium EDTA. The ramyon samples, which were aged by the accelerated oven test, were evaluated by the taste panel.

In a third storage experiment, the effect of TBHQ applied to the inner surface of the storage bag was investigated; no antioxidant was used in the frying oil. TBHQ (100 mg) was dissolved in 100 ml of acetone, and aliquots (0.4, 1.0 or 2.0 ml) of this solution were applied evenly over the inner surface of a polyethylene bag ("Ziploc" 5 × 8 cm) to give a quantity of TBHQ on the surface equal to 200, 500 or 1000 ppm, respectively, based on the oil (20%) in the ramyon. Samples (10 g) of crushed ramyon were placed in the treated polyethylene bags, and the filled bags kept inside capped 2-oz glass jars (2.54 cm i.d. and 6.35 cm height). The double-packaged noodles were stored at 63 C and the onset of rancidity determined by the taste panel.

Thin layer chromatography (TLC) was used to determine if TBHQ was oxidized during its application to the surface of the package. Silica gel plates GHLF 21521 "Uniplate" (Analtech Co., Newark, New Jersey) were used for TLC with methanol-chloroform (1:9, v/v) as developing solvent. The components were located by spraying the plates with aqueous sulfuric acid (50%) followed by ashing on a hot plate for 3 min.

Hexanal determination. Hexanal was determined in the head space above noodles by using the chromatographic method of Fritsch and Gale (11) with a slight modification. The gas chromatograph (GC) was a Hewlett Packard model 5750 equipped with a flame ionization detector. 4-Heptanone was used as the internal reference standard, and separation was done on an aluminum column (10 ft × 1/4 in. i.d.) packed with 10% silicone OV-101 on acid-washed 60-80 mesh chromosorb W (Supelco Inc., Bellefonte, Pennsylvania). The GC conditions were: column temperature, 100 C; injection port temperature, 200 C; detector temperature, 150 C, and nitrogen flow rate, 24 ml/min.

Five standard mixtures containing 2.5, 5.0, 7.5, 10.0 and 12.5 µl of hexanal and 5 µl of internal standard were prepared in 1000 ml of deionized water. An aliquot (10 ml) of each solution was added to 15 g of sucrose in a 250-ml centrifuge bottle, and boiling water was added until the total volume reached 150 ml. The bottle was capped immediately with a septum cap and swirled for 60 sec. Then 5.0 ml of the head-space gas was taken with a gas-tight syringe and injected into a GC at a rate of approximately 1 ml/sec. Areas of peaks were calculated as the product of the width at half-height times the height. A linear standard curve was obtained by plotting the concentration of hexanal (0-7 ppm) based on sucrose vs the ratio of the response area for hexanal and 4-heptanone.

To determine hexanal in the aged ramyon, the crushed ramyon was ground on a Wiley mill (through 60 mesh), and 15 g of ground ramyon was weighed into a centrifuge bottle in place of sucrose. The hexanal concentration in the ground ramyon was obtained from the ratio of the response area for hexanal and 4-heptanone by the method of least squares.

RESULTS AND DISCUSSION

Water activity of ramyon vs rancidity development. Ramyon was fried in palm oil containing no antioxidants, and the ramyon was equilibrated to constant moisture over various salt solutions with a_w 0.1-0.6. Table 1 shows the moisture contents of ramyon at different water activities, and the days for the ramyon to develop a rancid flavor at 25 C. The reciprocals of the time for onset of rancidity are plotted against water activity in Figure 1. The rancid taste developed most slowly in ramyon at a_w 0.3,

TABLE 1

Effect of Water Activity on the Moisture Content and Time to Develop a Rancid Taste in Fried Noodles[a]

	Water activity on equilibrated salt solution				
Property	0.1 Sat. $ZnCl_2$	0.3 44.4% $CaCl_2$	0.4 39.6% $CaCl_2$	0.5 35.6% $CaCl_2$	0.6 31.7% $CaCl_2$
Noodle moisture, wet basis, %	3.56 ± 0.01	5.16 ± 0.03	6.12 ± 0.03	6.83 ± 0.05	8.60 ± 0.06
Noodle moisture, fat-free wet basis, %	4.45 ± 0.01	6.45 ± 0.04	7.65 ± 0.04	8.54 ± 0.06	10.75 ± 0.07
Time to rancid taste, day[b,c]	76 ± 2	142 ± 4	117 ± 3	91 ± 2	36 ± 1

[a] Average of 3 determinations.

[b] Rancidity development at 25 C.

[c] LSD of time to rancid taste at $p = 0.05$ is 12.7.

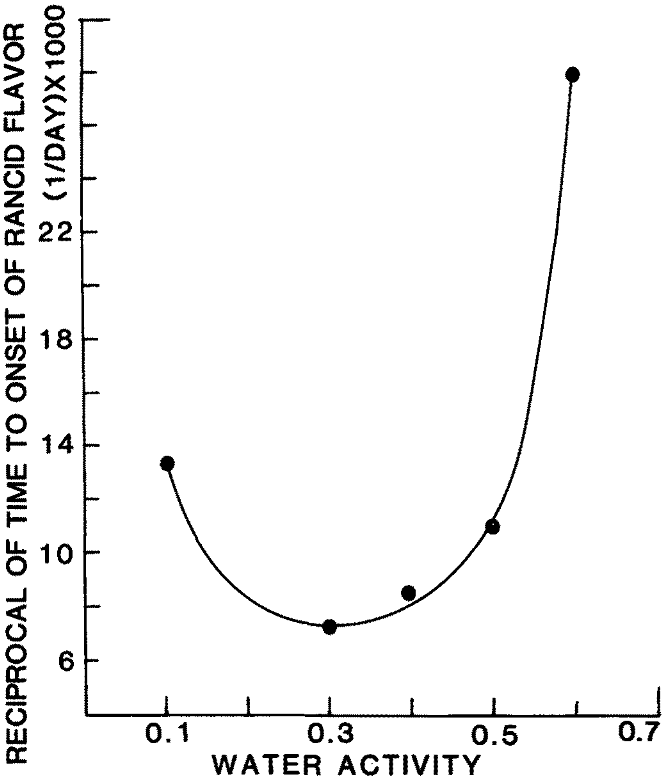

FIG. 1. Onset of rancid flavor in ramyon noodles stored at different water activities at 25 C. The average standard deviation was 2.8 (l/day × 1000).

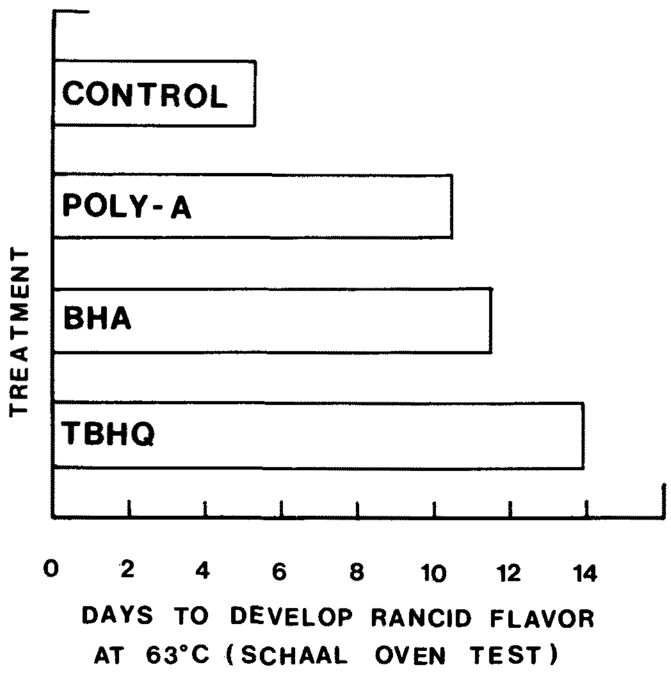

FIG. 2. Time (day) of rancid flavor development in ramyon fried in oil containing 0 or 200 pm antioxidant. The average standard deviation was 1.1 day.

which was equivalent to 5.16% moisture. The result agrees with theoretical considerations, which predict a minimum rate of rancidity development when water forms a monomolecular layer covering components in foods at a_w 0.2–0.3 (12,13). Okada (2) reported that a monolayer of water occurred in ramyon at a_w 0.16–0.24 (3.8–5.3% moisture), and that ramyon with a_w 0.25–0.8 varied little in peroxide or carbonyl value over the first 60 days of storage at 30 C. After 100 days storage, peroxide and carbonyl values varied markedly. On the other hand, we found that ramyon at a_w 0.6 was rancid after 36 days at 25 C (Table 1, Fig. 1).

Effect of antioxidants in frying oil on the shelf-life of ramyon. Ramyon was prepared in palm oil containing 200 ppm of three different antioxidants, and the onset of rancidity in the noodles was determined organoleptically after accelerated aging at 63 C. The results in Figure 2 show that 200 ppm of the antioxidant TBHQ, BHA and Poly-A extended the shelf-life of ramyon at 63 C by nine, six and five days, respectively, beyond the five-day shelf-life of the control. Thus, TBHQ approximately tripled and Poly-A and BHA doubled the shelf-life of ramyon com-

TABLE 2

Copper and Iron in Ingredients and Ramyon[a]

Ingredient	Trace metal concentration[b] (ppm) Copper	Iron
Blended wheat flour	0.18 ± 0.02	1.97 ± 0.14
Salt	0.32 ± 0.01	0.92 ± 0.04
Palm oil	0.15 ± 0.01	1.23 ± 0.08
Fried noodle	0.19 ± 0.01	1.71 ± 0.13

[a]Average of 3 determinations.

[b]Reported on a dry basis.

TABLE 3

Time to Develop a Rancid Taste in Ramyon Fried in Oil Containing a Mixture of Disodium EDTA and TBHQ

Treatment	Days at 63 C (145 F)[a]
Control[b]	4.8 ± 0.5
200 ppm TBHQ in oil	15.4 ± 1.1
200 ppm TBHQ and 200 ppm disodium EDTA in oil	18.6 ± 1.9
200 ppm TBHQ and 500 ppm disodium EDTA in oil	20.6 ± 2.3

[a]Average of 5 determinations. LSD at p = 0.05 is 1.67.

[b]Oil containing neither TBHQ nor disodium EDTA.

TABLE 4

Time to Develop a Rancid Taste in Ramyon Stored in a Polyethylene Bag with Its Inner Surface Coated with TBHQ

Treatment[a]	Days at 63 C (145 F)[b]
Control[c]	18.4 ± 0.9
200 ppm TBHQ in frying oil	27.2 ± 1.8
200 ppm TBHQ on package[d]	37.0 ± 3.1
500 ppm TBHQ on package[d]	39.8 ± 2.1
1000 ppm TBHQ on package[d]	44.6 ± 3.1

[a]Crushed ramyon (10 g) was placed in a 5 × 8 cm zip-lock bag, and the bag was stored in a capped 32-ml jar.

[b]Average of 5 determinations. LSD at p = 0.05 is 2.83.

[c]No TBHQ in oil or on package surface.

[d]Concentration of TBHQ based on 20% of crude fat in noodles.

pared to an antioxidant-free sample. By using the Schaal oven test (10) with pure oil, we ranked the antioxidants in the same order of effectiveness as the ramyon test, i.e., TBHQ > BHA > Poly-A. However, it was reported that Poly-A has a relatively high functional carry-through capability (14). On a cost comparison basis, TBHQ and BHA were equally effective in this study. The additional cost in 1983 was 0.15 cents per day of extension at 63 C, which is equivalent to approximately 20 days at 25 C (Tables 1 and 3), per 120 g of ramyon, which is a common weight of a single-serving package.

Effect of a chelating agent on the shelf-life of ramyon. Copper ranged from 0.15 to 0.32 ppm and iron from 0.92 to 1.97 ppm in the ingredients and in the ramyon (Table 2). The ramyon-making process increased the levels of copper and iron by 19 and 12% above the total amounts in the ingredients, respecctively. Copper and iron have been reported (5,15) to promote rancidity development in ramyon.

Table 3 shows that addition of disodium EDTA prolonged the shelf-life of ramyon. When compared to ramyon fried in oil containing 200 ppm of TBHQ, which extended the shelf-life by 11 days past the control, frying in oil containing a mixture of 200 ppm of TBHQ with 200 ppm or 500 ppm of disodium EDTA extended the shelf-life by 14 or 16 days, respectively. It is well known that a synergist, such as disodium EDTA, has very little effect on the oxidation of foods in the absence of a primary antioxidant. However, a synergist greatly enhances the effectiveness of a primary antioxidant by forming inactive complexes with pro-oxidant metals.

Effect of TBHQ coated on the inner surface of the package on the shelf-life of ramyon. The inner surface of the polybag was coated with TBHQ equivalent to 200, 500 and 1000 ppm based on the oil in the noodle. TLC was used to determine if the thin film of TBHQ inside the bag had oxidized or reacted with any additives in the plastic. Figure 3 shows that TBHQ gave two spots with R_f 0.39 and 0.79 (Lane 2), while an acetone rinse of the polyethylene film gave two faster moving spots (Lane 1), which were not identified. Lane 3, which was the acetone extract of the inner surface of a TBHQ-coated bag, shows TBHQ was recovered unchanged along with the unknown components from the polyethylene.

Compared to the control containing no antioxidant, the presence of 200 ppm in the frying oil extended shelf-life nine days at 63 C, while the same quantity of TBHQ on the inner surface of the bag extended shelf-life 19 days (Table 4). Increasing the quantity of TBHQ on the surface of the bag to 500 and 1000 ppm gave 22 and 27 more days without rancidity compared to the control. These results may be explained by transfer of TBHQ from the inner surface of the bag (compare Lanes 4, 6 and 8 with 5, 7 and 9, Fig. 3) to the surface of the noodle. It seems likely that the fat on the surface of the noodle initiates rancidity development in noodles because the surface is exposed to the most light and oxygen. Lanes 4, 6 and 8 in Figure 3 contained fast-moving spots of which a major fraction was triglycerides and a minor fraction was unknowns from the plastic. Triglycerides were translocated from the ramyon onto the inner surface of the polyethylene bag.

The control sample given in Table 4 had a shelf-life of 18 days, while the control sample shown in Figure 2 and given in Table 3 had a shelf-life of ca. 5–6 days. The difference is due to the double packaging used to obtain the data in Table 4. In the experiment reported in Table 4, the noodles were sealed in small polyethylene bags which were then placed in capped glass jars. Without the jars, the rancid by-products might have escaped from the bags. The data shown in Figure 2 and Table 3 were for noodles packaged in capped jars with ca. three volumes of headspace. The double packaging of ramyon, with the small head space in the bags, tripled the shelf-life of control ramyon in Table 4 compared to the control ramyon

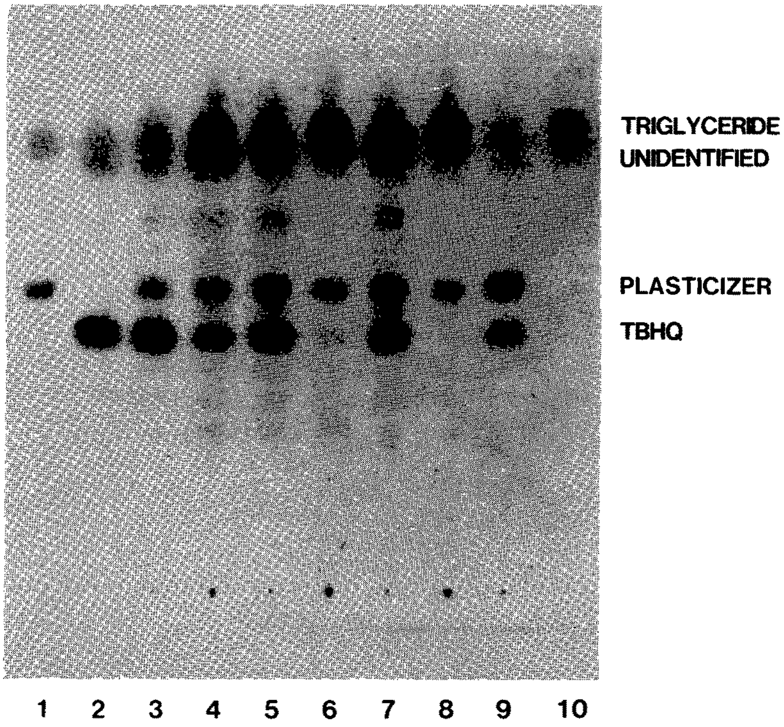

FIG. 3. Fate of TBHQ applied to the inner surface of polyethylene bags used to package ramyon. Fifty μl of solution was applied to each lane. Lane 1. Acetone (1 ml) rinse of the inner surface of polyethylene bag; Lane 2. Acetone (1 ml) containing 0.4 mg TBHQ; Lane 3. Acetone (1 ml) rinse of polyethylene bag previously coated with 0.4 mg TBHQ; Lane 4. Acetone (1 ml) rinse of polyethylene bag initially coated with 0.4 mg TBHQ, then loaded with 10 g ramyon and stored 12 hr at 63 C (145 F); Lane 5. Acetone (1 ml) rinse of polyethylene bag initially coated with 0.4 mg TBHQ, followed by 12 hr storage at 63 C (145 F); Lane 6. Same as Lane 4 except stored 24 hr; Lane 7. Same as Lane 5 except stored 24 hr; Lane 8. Same as Lane 4 except stored 48 hr; Lane 9. Same as Lane 5 except stored 48 hr; Lane 10. Acetone (1 ml) solution containing 0.4 mg palm oil.

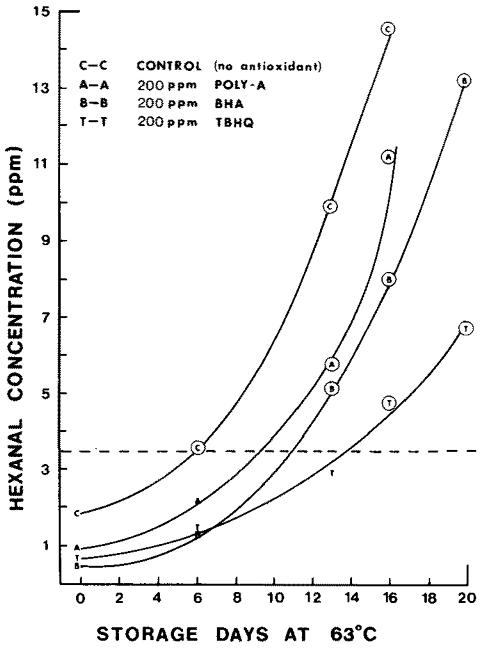

FIG. 4. Hexanal concentration vs storage days of ramyon at 63 C (145 F). The circled points on the curves have a rancid taste as determined organoleptically. The dotted line denotes the concentration of hexanal which can be detected organoleptically in ramyon. The average standard deviation of hexanal in the ramyon was ±0.54 ppm.

kept in a capped jar. Alternatively, the plastic bag insulated some of the noodles from the storage temperature of 63 C. The noodles packaged doubly had a lower average temperature and developed rancidity more slowly than in the singly packaged noodles in glass jars.

Hexanal as a measure of rancidity in ramyon. When the hexanal concentration of the ramyon stored in the capped glass jars exceeded 3.5 ppm, a rancid taste was detected by the panel (Fig. 4). Furthermore, the efficiencies of the antioxidants determined by the rate of hexanal release in stored ramyon were in the exact order as determined by the organoleptic test (Fig. 2).

From the off-flavor points (circles) shown in Figure 4, it can be concluded that the onset of rancidity as determined organoleptically by the Schaal oven test occurred when the concentration of hexanal in ramyon exceeded 3.5 ppm. At 3.5 ppm hexanal in ramyon the curves in Figure 4 predict the onset of rancid taste at 6, 9, 11 and 14 days for the control, Poly-A, BHA and TBHQ noodles, respectively. The values found by the taste test (Fig. 2) were 5.2, 10.4, 11.4 and 13.8 days, respectively. The detection of a rancid taste in ramyon occurred at a lower hexanal concentration (⩾3.5 ppm hexanal) than that found for dry oat cereal (5-10 ppm hexanal), which was reported by Fritsch and Gale (11). Hexanal in ramyon appears to be a good indicator of the development of oxidative rancidity. The relative effectiveness of antioxidants in preventing off-flavor development could be determined by the amount of hexanal released per unit weight of the stored ramyon.

It is widely accepted that polyunsaturated fatty acids are largely responsible for rancid flavor. Palm oil has less than 10% polyunsaturated fatty acids (16), while wheat-flour nonstarch lipids contain more than 60% (17). The main source of polyunsaturated fatty acids in ramyon may at first glance appear to be the frying oil, but wheat flour contains 1.5–2.0% lipids, of which 64–71% is polyunsaturated fatty acids (17). Approximately 25% of the polyunsaturated fatty acids in ramyon arise from wheat flour. Therefore, even if noodles were fried in fully saturated fat, oxidative rancidity would still limit the shelf-life of the noodle.

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