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Determination of Optimum Sterilization Condition for the Production of Retort Pouched Curry Sauce

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Abstract

In order to optimize sterilization conditions of retort pouched curry sauce, sterilization processes for eighteen conditions by varying temperature, time and method were conducted through $3^2 \times 2^1$ experimental factorial design. Quality evaluations before and after sterilization included measurements of vitamin (niacin) retention, pH and color differences, and organoleptic test(taste, color and viscosity). F_0 values were also measured at each condition. Product qualities were mainly affected by sterilization temperature and time, whereas sterilization method had no significant effect. Effect of sterilization time on product qualities was higher than that of sterilization temperature. From the response surface analysis, an optimum range of sterilization condition simultaneously satisfying desired specifications was determined to be 123.5℃, 21.5 min to 127.5℃, 17.0 min. In this range, the sterility(F_0 value) at a cold point during sterilization was approximately 15.0 min.

Key words: sterilization, retort pouched curry sauce, response surface analysis

Introduction

Sterilization is a characteristic process in the production of thermally processed foods such as canned or retort pouched foods. The effect of time-temperature treatments on nutrients and other quality attributes such as color, texture, flavor, viscosity and taste has been considered to be an important aspect for the sterilization of foods. Lund *et al.*,[1] Harris and von Loeseche,[2] Schroeder,[3] and Goldblith *et al.*,[4] and many other researchers have discussed about the thermal process simultaneously satisfying commercial sterilization of microorganisms and maximum retention of nutritional and quality attributes. However, little work has been done to evaluate the quality of particular retort pouched foods when subjected to various thermal processing levels.

The objective of this study was to determine the influence of heating temperature and time, and the type of heating medium on the sterility and the quality factors during sterilization of retort pouched curry sauce. In this study, the response surface methodology was applied to find an optimum sterilization time-temperature range not only to accomplish commercial sterility but also to provide products having better nutritional and quality attributes than product from the reference time-temperature condition(120 ℃, 33 min).

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Materials and Methods

Experimental design

Table 1 shows $3^2 \times 2^1$ factorial experiment in which there were two quantitative variables(sterilization temperature and time) and a single qualitative variable(sterilization method). For quantitative variables, temperature was varied with 3 levels(120℃, 125℃ and 130℃) and time was also varied with 3 levels(20 min, 25 min, 33 min). These were coded by the following equations:

$$X1 = \frac{T - 125}{5} \quad (1)$$

where T is sterilization temperature (℃),

$$X2 = \frac{t - 25}{5} \quad (2)$$

where t is sterilization time(min).

For qualitative variable(sterilization method) two levels were also conveniently coded by minus one and plus one for steam/air mixture and hot water with air or steam overpressure as a heating medium, respectively. Coded sets of reference conditions selected in this study were(-1, $+1.6$, -1) and (-1,

+1.6, +1) since these conditions have been commercially used for sterilization of common curry sauce with meats and vegetables in Korea.

Sterilization

Sterilization process was conducted according to the experimental design planned above with randomized run of order. The high temperature and high pressure sterilizer for ready-to-serve foods(Hisaka Laboratory Sterilizing Machine, Model; RCS-40 RTGN) was used for this study. During sterilization at each condition, heat penetration temperatures and F_0 values at the coldest point in the retort pouch were recorded by a F_0 monitor(Ellab Temperature Recorder, Model; Z4FD). Conducting thermal process at each condition, 20 retort pouches of curry sauce were sterilized at one time. In experiments using steam/air mixture as the heating medium, 4 pouches each were placed on 5 trays without overlapping pouches. On the other hand, when using hot water with air or steam overpressure as the heating medium, only 3 trays were used about 6 to 7 pouches were placed on each tray with 1/3 overlapping pouches. This was for minimizing the difference of intensity of heat transfer affected each pouch between two sterilization methods. In order to find the optimum overlapping condition resulting in the same sterility at both reference conditions(120℃, 33 min at both sterilization methods), preliminary sterilization experiments were conducted by varying degree of overlapping(3/4, 1/2, 2/3 and 1/4), and then the optimum condition was determined to 1/3 overlapping. The coldest point during sterilization was assumed as the center of diced potato having the lowest thermal conductivity, considering whole contents of curry sauce used in this study. The retort pouch monitored by F_0 monitor was placed at the middle part of retort. Pouches used in this study were formed from casting polypropylene 70 μm, aluminum foil 9 μm, and polyethylene telephtalate 12 μm laminate. The total fill weight was about 190 g, and the pouch size was 5 in×6.6 in.

Measurements of pH and color values

Retort pouched curry sauces sterilized in each condition were sampled randomly with 4 replications. The pH of curry sauce was measured by the pH meter(HANNA Instruments, Model; B521). The color values(L, a, b, and ΔE) were measured by color and color difference meter(Nippon Denshoku, Model; 1001DP), and color difference(C_d) between samples produced in the reference condition and other conditions were defined by the following equation:

$$C_d=\sqrt{(L-L_0)^2+(a-a_0)^2+(b-b_0)^2} \qquad (3)$$

where L, a, b are color parameters for various conditions, and L_0, a_0, b_0 are color parameters for the reference condition.

Quantatative analysis of vitamins

Ten grams of samples were mixed with 100 ml of 0.1 N-H_2SO_4 and were homogenized by blade homogenizer(Ace Homogenizer, Model; AM-11). These were autoclaved for 40 min at 1.5 kg_f/cm^2 according to the procedure described by Kirk.[5] After cooling to room temperature, the contents were adjusted to pH 4.5-4.6 with 2 M sodium acetate, 5 ml of diastase (5 %w/w)(Merck) and 5 ml of papain(10 %w/w)(Merck) were added to the contents. These were incubated overnight at 37±1℃. The sample was filtered through(ash-free) filter paper using the aspirator(Eyela, Model; A-3S). 5 ml aliquots of sample were refiltered through a clarification filter, 0.25 mm, with 0.5 micron porosity(Waters) using a 25 μl syringe. 5 μl replicates of the filterate were injected into the U6K injection loop and vitamin concentrations for each sample was measured by UV aborbance at 254 nm. The peaks were graphically produced by a single pen recorder(Young-In, Model; D520).

The high performance liquid chromatogrpahy (HPLC) was equipped with a pump(Waters, Model; 510), a U6K septumless injector and a UV absorbance detector(Waters, Model; 481). The chromatography column was a Waters 30 min×4 mm(i.d.) micro Bondapak C18. Mobile phase, which was a mixture of nanograde methanol(Waters; 399 ml) 1 ml glacial acetic acid in 600 ml distilled deionized water, was pumped through the column at the rate of 1 ml per min.

Standard calibration curves for water soluble vitamins(thiamine, riboflavin, niacin, folic acid, pyridoxine an ascorbic acid) were obtained before analyzing samples.

Experiments were carried out in quadruplicate and concentration of vitamins was determined from the standard curves.

Sensory evaluation

Sensory evaluations were conducted by 7 to 10 trained panel members. In this study, the multiple comparison test was used for evaluating samples from the reference condition and other conditions. Panel members rated the preference of samples for taste, color and viscosity using a nine-point preference scale(1=worst, 9=best) in which the score for the reference sample was assumed as 5.

Statistical analysis

The multiple regression analysis was conducted by using SAS/STAT(Software, SAS Institute Inc.). From this anlaysis, main, interaction and quadratic effects of three independent variables on product qualities were determined and optimum second order equations were obtained through the stepwise procedure. In order to show the relationships between input variables and measured responses schematically, response surface methodology was employed and contour plots for each product quality were generated by SAS/STAT.

Results and Discussion

Seven dependent responses, namely, vitamin(Niacin) retention, pH, difference of color, sensory evaluated values(taste, color and viscosity) and F_0 value obtained by varying three independent variables, temperature, time and method during sterilization were shown in Table 1. Values in Table 1 were determined by the following manners: (1) Vitamin retention(Y1); Niacin was the standard material for predicting nutrient destruction during heat processing in this study (Contents of other vitamins were trace). Values indicated in Table 1 were the mean of retained niacin percentage after sterilization at each condition: (2) pH and color difference (Y2 and Y3); pH and C_d values were figured out by averaging four replications. Values for color difference were calculated by Eq. (3). If the average ΔE value of sample from each condition was higher than that from the reference condition, positive sign was taken. Otherwise, negative sign was taken: (3) Sensory evaluated values (Y4, Y5 and Y6); these were average scores evaluated by 7-10 panelists when the score of sample produced at the reference condition was let by five: (4) F_0 values (Y7); these were the mean values of 5-10 replications. Quality differences between samples from two reference conditions were negligible and sensory evaluated scores for the products of these conditions could be assumed the same in Table 1. Considering Table 1 totally, evaluated product qualities (Y1-Y6) were worse and worse on the whole with increasing the sterility (Y7). Comparing product qualities between run 11 having a minimum sterility

Table 1. Product qualities evaluated after various conditions of sterilization through $3^2 \times 2^1$ factorial design

Order of run	Temperature (X_1)	Time (X_2)	Method (X_3)	Y1	Y2	Y3	Y4	Y5	Y6	Y7
11	−1	−1	−1	86.9	5.55	−1.58	5.50	6.38	5.00	4.2
13	−1	−1	+1	86.8	5.57	−1.52	5.33	5.11	5.67	4.5
8	−1	0	−1	80.2	5.52	−1.84	4.66	5.00	5.50	7.7
3	−1	0	+1	83.7	5.58	−1.91	4.78	4.30	4.60	8.1
R*	−1	+1.6	−1	81.6	5.50	0	5.00	5.00	5.00	15.7
R*	−1	+1.6	+1	79.2	5.49	0	5.00	5.00	5.00	15.6
5	0	−1	−1	88.9	5.53	−0.60	5.12	5.75	5.12	15.0
2	0	−1	+1	90.3	5.56	−2.11	5.22	5.30	5.30	17.0
6	0	0	−1	85.7	5.41	+0.38	5.12	5.25	4.63	26.8
1	0	0	+1	82.0	5.45	−1.99	4.00	5.10	3.90	30.0
9	0	+1.6	−1	73.8	5.37	−1.28	3.66	5.17	4.00	50.4
14	0	+1.6	+1	74.8	5.42	+2.02	4.33	4.56	4.44	−
7	+1	−1	−1	79.3	5.36	+1.03	5.75	5.50	4.50	30.1
4	+1	−1	+1	82.3	5.51	+1.88	4.37	4.37	5.75	38.9
10	+1	0	−1	79.2	5.38	+1.39	3.33	5.17	3.16	−
15	+1	0	+1	84.4	5.44	+2.26	4.33	5.45	5.11	−
12	+1	+1.6	−1	65.0	5.22	+2.82	2.63	2.63	3.12	−
16	+1	+1.6	+1	71.8	5.28	+2.47	3.55	4.56	4.00	−

−; These values were too large to measure, *;Reference conditions, Y1; Vitamin (Niacin) retention (%), Y2; pH, Y3; Color differnce, Y4; Taste (sensory evaluation), Y5; Color (sensory evaluation), Y6; Viscosity (sensory evaluation), Y7; F_0 value at the coldest point in retort pouch. For Y4, Y5, Y6, the range was from 1 to 9.

and run 12 having a maximum sterility, considerable differences were observed.

Results of multiple regression analysis were shown in Table 2. Table 2 shows that coefficients for main effects were generally much greater than those for interaction or quadratic effects. Only the interaction effect of sterilization temperature and time was more or less significant on pH, sensory evaluated taste and viscosity, and F_o value. From these results, quality degradation during sterilization was mainly affected by temperature and time. However, the effects of sterilization method on product gualities was much less significant than that of temperature and time.

Contour plots for each attribute of product quality shown in Fig. 1-Fig. 7 were generated as a function of two independent variables, viz., sterilization temperature and time, while sterilization method was held constant at the point of center (that is, zero). From now, more details of the effect of independent variables on the individual attribute of product quality will be discussed.

Niacin retention

Vitamins in a curry sample detected by HPLC analysis were pyridoxine, thiamine and niacin, and initial concentrations of each vitamin were 2.78, 6.35 and 11.62 μg/g, respectively. For pyridoxine and thiamine, since the initial concentrations were too low and these have been known as relatively heat-liable[6-9] it was impossible to quantify their retentions accurately. On the other hand, since the heat stability of niacin has been known to be relatively higher compared with pyridoxine or thiamine,[6,10] the effect of heat severity on the retention of niacin could be investigated in this study.

The retention of niacin was mainly affected by two independent variables, sterilization temperature and time, i.e., effects of those variables were significant at 5% level and 1% level, respectively, as shown in Table 2. This means that the effect of time was moderately higher than that of temperature. Teixeira *et al.*[11] presented a relationship between various time-temperature treatments providing microbial lethality and retention of nutrients with different z values. They concluded that if any nutrient in foods has a small z value, its retention is favored by low-temperature and long-time process; whereas if any nutrient in foods has a large z value, its retention is favored by high-temperature and short-time process. It would be seen that high-temperature and short-time process slightly favors niacin retention from the result obtained by the present study. Contour plot for niacin degradation is shown in Fig. 1. As shown in this figure, the destruction rate of niacin during thermal processing was varied from 14% to 38% by changing heat severity. Fig. 1 also shows that the effect of the sterilization temperature on niacin degradation was not significant below 25 min of the sterilization time.

Table 2. Effects of independent variables on each product quality evaluated by multiple regression analysis

Evaluated qualities		Main effects[a]				Interaction effects			Quadratic effects[b]	
		X1	X2	X3	X1·X2	X1·X3	X2·X3	X1·X2·X3	X1·X1	X2·X2
Niacin retention	Coeff.	−2.757	−3.943	0.830	−1.382	1.038	0.068	0.641	−2.550	−0.726
	T Stat.	−2.767**	−3.749***	−0.988	−1.512	1.043	0.091	0.701	−1.504	−0.679
pH	Coeff.	−0.080	−0.051	0.027	−0.024	0.018	−0.006	−0.004	−0.007	−0.001
	T Stat.	−7.469***	−4.480***	3.061**	−2.433**	1.631	−0.795	−0.428	−0.365	−0.070
Color difference	Coeff.	1.577	0.432	−0.007	−0.093	0.137	0.250	−0.118	1.013	0.234
	T Stat.	5.016***	1.301	−0.026	−0.324	0.441	1.062	−0.409	1.893	0.693
Taste[c]	Coeff.	−0.466	−0.602	−0.031	−0.301	0.012	0.192	0.186	−0.056	0.243
	T Stat.	−3.555***	−4.353***	−0.287	−2.502***	0.091	1.957*	1.550	−0.250	1.727
Color[c]	Coeff.	−0.226	−0.354	−0.169	−0.164	0.220	0.261	0.170	−0.316	0.003
	T Stat.	−1.241	−1.836*	−1.134	−0.978	1.207	1.970*	1.015	−1.017	0.015
Viscosity[c]	Coeff.	−0.338	−0.540	0.244	−0.237	0.317	−0.041	0.005	0.094	0.283
	T Stat.	−2.630**	−3.976***	2.319**	−2.007	2.460**	−0.420	0.041	0.430	2.049*
F_o value	Coeff.	24.238	13.173	1.493	9.196	1.549	−0.432	−0.497	3.524	0.600
	T Stat.	30.618***	19.867***	2.653*	11.389***	2.362*	−0.680	−0.758	4.809**	1.505

[a]X1 = sterilization temperature, X2 = sterilization time, X3 = sterilization method
[b]Since X3 · X3 is constant, this was eliminated in regression analysis
[c]These were sensory evaluated values
*Significant at 10% level, **Significant at 5% level, ***Significant at 1% level

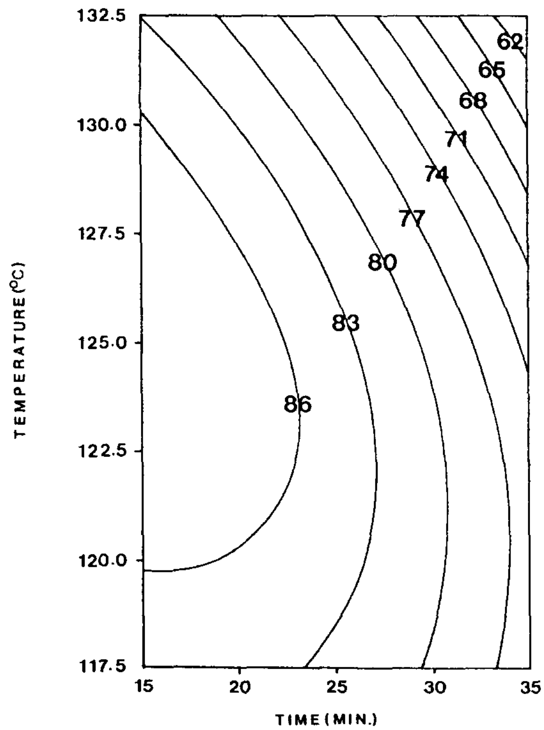

Fig. 1. Contour plotting lines for niacin retention(%) as a function of sterilization temperature and time at the center point (=0) of sterilization method. (response surface equation; $Y1=84.233-2.757\ X1-3.942\ X2-2.550\ X1^2-1.382\ X1\cdot X2\ -0.726\ X2^2$, $R^2=0.820$)

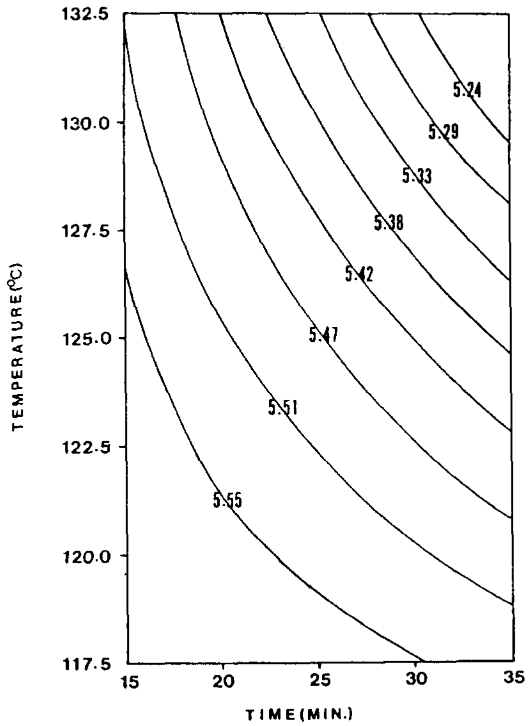

Fig. 2. Contour plotting lines for pH as a function of sterilization temperature and time at the center point (=0) of sterilization method. (response surface equation; $Y2=5.469-0.080\ X1-0.051\ X2-0.007\ X1^2-0.024\ X1\cdot X2-0.001\ X2^2$, $R^2=0.938$)

pH

As shwon in Table 2, pH was affected by sterilization temperature and time adversely. That is, the increase of thermal severity resulted in drop of pH. Table 2 also shows that pH was affected moderately by the sterilization method. However, correlation coefficients (R^2) between pH and organoleptic properties by the linear regression analysis were 0.50, 0.25 and 0.61 for taste, color and viscosity, respectively. That is, the significant effect of pH in the range of 5.22-5.58(see Table 1) on the sensory acceptability was not found. Therefore, the acceptable pH range could not be determined, and so pH term was eliminated for establishing the optimum range of sterilization condition, even if the change of pH by varying the sterilization condition was significant statistically. Fig. 2 shows the contour plot on pH as a function of the sterilization temperature and time. As shown in Fig. 2, pH of product had regularly-increased tendency with increasing the sterilization temperature and time.

Color difference

The change of color according to the change of sterilization condition was positively affected mainly by the sterilization temperature, but was not nearly affected by the sterilization time. That is, ΔE value was increased with increasing processing temperature regardless of processing time in interested region. This suggests that, as the sterilization time was too long, outside part of product in the pouch would receive the excessive heating effect and color deterioration by the browning reaction would be occurred at a much faster rate. Many researchers(12-15) have discussed about the browning reaction of various foods occurring in thermal processes at high temperature and during storage. They have explained that main reason of the browning reaction is the reaction between reducing sugars and amino compounds contained in food ingredients. Nagashima *et al.*(16,17) presented results on the change of color of canned cooked curry occurring during heating and storage. They explained that this would be caused by amino carbornyl

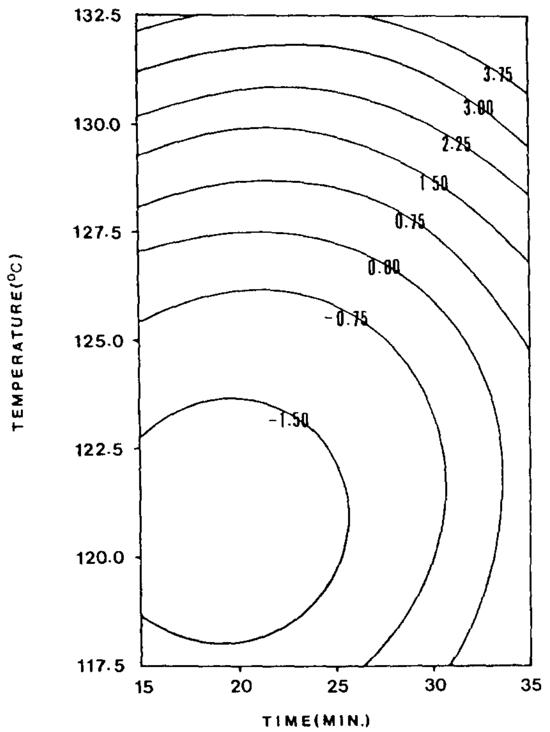

Fig. 3. Contour plotting lines for color difference as a function of sterilization temperature and time at the center point (=0) of sterilization method (response surface equation; $Y3 = -0.961 + 1.577\ X1 + 0.432\ X2 + 1.013\ X1^2 - 0.093\ X1 \cdot X2 + 0.234\ X2^2$, $R^2 = 0.792$)

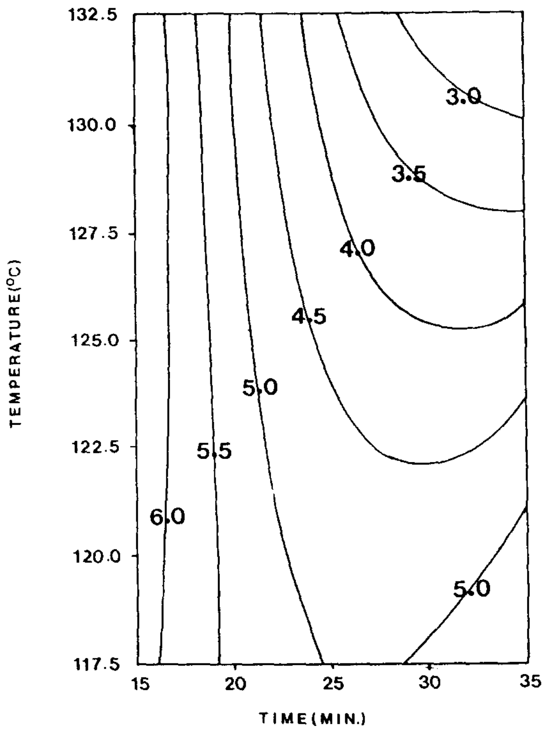

Fig. 4. Contour plotting lines for sensory evaluated teste as a function of sterilization temperature and time at the center point (=0) of sterilization method (response surface equation; $Y4 = 4.407 - 0.466\ X1 - 0.602\ X2 - 0.056\ X1^2 - 0.301\ X1 \cdot X2 + 0.243\ X2^2$, $R^2 = 0.760$)

browning reaction. The result of the present study for the color difference is shown in Table 2 and illustrated with contour plot in Fig. 3. Fig. 3 shows that when the sterilization temperature was below 127.5 ℃, the degree of browning of the product from each condition was similar or lower compared with that from the reference condition.

Sensory values

All tested terms(taste, color and viscosity) were mainly affected by the sterilization time and, to some extent, by the sterilization temperature as similar with the result for niacin retention. That is, the effect of 5 minutes of the sterilization time on reducing the organoleptic qualities was higher than that of 5℃ of the sterilization temperature. This suggests that the sensory properties of product can be also improved by selecting the high-temperature and short-time sterilization condition. Lund[18] presented a comprehensive review on sensory analysis results performed for appearance, texture, odor, flavor, taste and off-taste of various foods as affected by thermal processing. He concluded that the sensory properties have generally not received the same success in quantification as nutrients as mentioned above since these somewhat depend on the mind of the observer, but our knowledge of this area will allow us to design process to accomplish any desired set of quality attributes.

Novais *et al.*[19] investigated the sensory acceptability of several retort pouched foods processed at different F_0 values and found that it was not significantly affected within the range of $F_0 = 5$ min to $F_0 = 15$ min. In the present study, however, it was found that the thermal severity affected sensory acceptability significantly. This may be caused by the application of much wider range of F_0 value in this study.

Contour plots for the sensory values, points for taste, color and viscosity, are shown as a function of sterilization temperature and time in Figs. 4, 5 and 6, respectively. Considering Fig. 4, 6 and Table 1, the statistical results of sensory evaluation for taste and

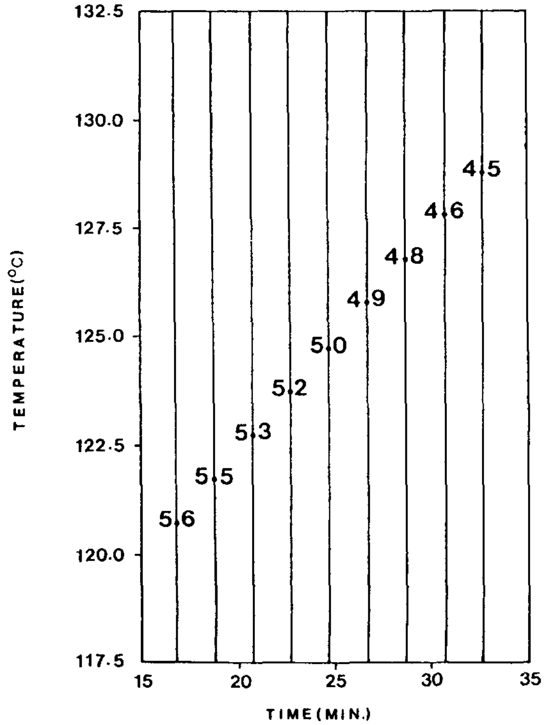

Fig. 5. Contour plotting lines for sensory evaluated color as a function of sterilization temperature and time at the center point (=0) of sterilization method (response surface equation; $Y5=5.045-0.354\ X2-0.003\ X2^2$, $R^2=0.254$)

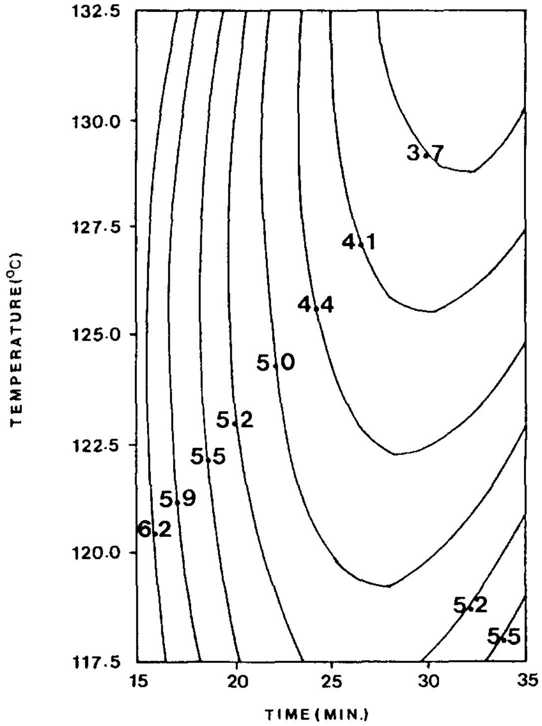

Fig. 6. Contour plotting lines for sensory evaluated viscosity as a function of sterilization temperature and time at the center point (=0) of sterilization method. (response surface equation; $Y6=4.329-0.338\ X1-0.540\ X2+0.094\ X1^2-0.237\ X1\cdot X2+0.283\ X2^2$, $R^2=0.840$)

viscosity were fitted well with the experimental results, and panel scores were increased with decreasing the sterilization time regardless of the sterilization temperature when the sterilization time was below about 22 min. In the case of the sensory evaluated color, however, the statistical values did not correspond to the experimental values as shown in Fig. 5 ($R^2=0.254$). This means that the sensory scores for color on the product had no linearity with the sterility differing from other sensory terms(taste and viscosity).

F_0 values

Considering coefficients for multiple regression in Table 2, the effect of raising the sterilization temperature by 5℃ on the sterility was much higher than that of extending the sterilization time by 5 min. For instance, as the sterilization temperature was raised from 120℃ to 125℃, the sterilization time could be shortened from 33 min to 20 min. having similar sterility as shown in Table 1. The fact that a given increase in temperature causes a much larger increase in the rate of destruction of microorganisms than in the rate of destruction of nutrients or quality factors has been proved by considerable research efforts.[1-4,6,11] This situation is exploited to the advantage in high-temperature and short-time process. Experimentally, the destruction rate of spores of *Clostridium botulinum*, which is commonly used as a standard microorganisms for commercial sterilizations(also for this study), at 131℃ is tenfold(z=10℃) greater than at the standard temperature of 121℃, whereas the destruction rate of nutrients and quality factors would be only two to threefold greater than at that. The results from the present study fairly corresponded to the previous researches. Fig. 7 show the contour plot of F_0 values.

By superimposing the critical contour plotting lines for each quality response, acceptable range of sterilization condition was determined. The critical values of interested ranges were selected as follows: above about 80% for niacin retention; below 0 for color dif-

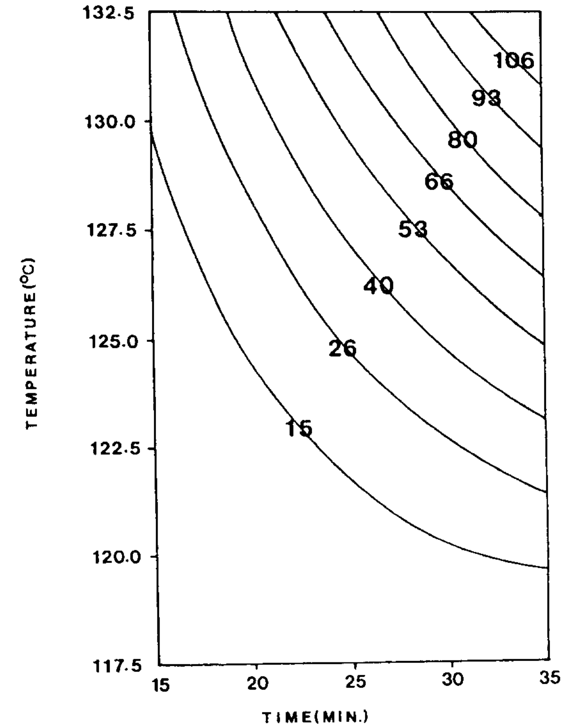

Fig. 7. Contour plotting lines for F_0 value as a function of sterilization temperature and time at the center point (=0) of sterilization method. (response surface equation; $Y7=28.412+23.957\ X1+12.925\ X2+3.435\ X1^2+8.885\ X1 \cdot X2+0.508\ X2^2$, $R^2=0.981$)

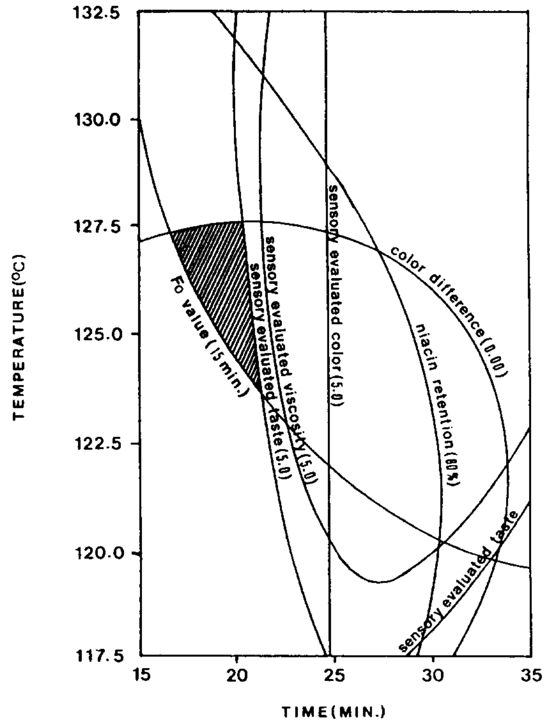

Fig. 8. The acceptable(striped) range obtained by superimposing the contour plots of evaluated product qualities of retort pouched curry sauce as a function of sterilization temperature and time. Critical contour plot for pH is not shown, as all pH range(5.22−5.58) was assumed reasonable. Values in parenthesis indicate critical values(values for the reference condition) for each term.

ference; above 5.00 for sensory evaluated terms; above about 15 min for F_0 value. All of these critical values were for the reference condition. As shown in Fig. 8, the optimum sterilization temperature and time ranges for producing products with better quality than product from the reference condition were 123.5-127.5℃ and 17.0-21.5 min, respectively. In these ranges, however, since the process above $F_0=15.0$ min. is excessive, more specific range(temperature-time sets) of an optimum sterilization process for the retort pouched curry sauce investigated in this study is formed following by the contour plotting line of $F_0=15$ min.

In view of economic aspect, the reduction of 30-40 % of utility cost for the production cost is predicted, when the retort pouched curry sauce is produced in the optimum process conditions(123.5℃, 21.5 min to 127.5℃, 17 min having 15 min F_0 value) established in this study, comparing with the reference condition (120℃, 33 min).

Consequently, the optimized sterilization conditions for the retort pouched curry sauce would make it possible not only to improve nutritional and quality attributes, but also to reduce the processing cost.

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레토르트 카레 소스 생산을 위한 최적살균 조건의 설정

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레토르트 카레 소스를 생산하기 위한 최적 살균 조건을 산출하기 위하여, $3^2 \times 2^1$ 요인 실험을 통하여 살균 온도, 시간 및 방법을 변화시켜 18개 조건에 대한 살균 공정이 행하여졌다. 살균 전후의 제품에 대한 품질 평가 항목은 비타민(niacin) retention, pH, 색차 및 관능 검사(맛, 색상, 점도)등 이었다. 각 살균 조건에 대한 F_0 값도 측정되었다. 살균 후 제품의 품질은 주로 살균 온도 및 시간에 의해 영향을 받았으며, 살균 방법에 의해서는 거의 영향을 받지 않았다. 품질을 저하시키는 영향에 있어 살균 시간의 연장이 살균 온도의 상승보다 큰 영향을 나타내었는데, 이는 고온 단시간 살균의 장점을 제시해준 결과라 하겠다. 반응 표면 분석에 의해 추정된 최적 살균 범위는 123.5℃, 21.5분-127.5℃, 17분 부근이었다. 이 범위에서 측정된 냉점에서의 F_0 값은 15분 정도였다.