

## Optimization of the Processing Conditions for Heated Garlic Juice by Response Surface Methodology

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### Abstract

This study was designed to determine the optimum conditions of heating temperature, heating time and dilution rates for producing heated garlic juice by using central composite design of response surface methodology. Garlic was heated using a high temperature and pressure treatment apparatus. Total soluble solid contents ranged from 4.4 at 130°C 3 hr and 6 fold dilution to 5.89 at 115°C for 2 hr and 8 fold dilution. The highest total acidity was 0.55% at 120°C for 3 hr and 2 fold dilution. The pH ranged from 4.01 at 130°C, 3 hr and 6 fold dilution to 5.85 at 120°C, 1 hr and 6 fold dilution. From the results of statistical analysis on the sensory evaluation the predicted optimum processing conditions for best color, taste, flavor and overall acceptance were 119.41°C, 3.11 hr, 5.85 fold dilution, 118.23°C, 3.57 hr, 3.15 fold dilution, 120.54°C, 3.47 hr, 5.01 fold dilution and 119.24°C, 3.18 hr, 5.66 fold dilution, respectively. The application of response surface methodology for preparing heated garlic juice processing showed a good correlation with high significance.

**Key words:** heated garlic juice, response surface methodology, viscosity, sensory evaluation

### INTRODUCTION

Garlic (*Allium sativum* L) has been used universally as a food, spice, and traditional medicine. Bioactive components of garlic including several sulfur-containing compounds such as alliin, allicin and diallyl disulfide are partially responsible for some effects of garlic (1). These components are known to possess antibacterial, antifungal, antiparasitic, antiviral, antioxidant, as well as antithrombotic, vasodilatory and anticancer activities (2-5). During the crushing of garlic, allicin is produced through the interaction of alliin (S-allyl-L-cysteine sulf-oxide) with the pyridoxal phosphate-containing enzyme, allinase (6).

Recent studies have shown that thermally processed foods, especially fruits and vegetables, have higher biological activities due to various chemical changes during heat treatment. Woo et al. (7) reported that the total polyphenol content of heated onions increased significantly with increased heating temperature and time. The polyphenol and flavonoid contents and antioxidant activity increase with heat treatment in plants such as, ginseng (8), licorice (9), garlic (10), pear (11), Shiitake mushroom (12), and citrus peel (13). The boiling of garlic

cloves significantly decreased its ability to inhibit cyclo-oxygenase activity and thromboxane B2 synthesis. In addition, microwave heating of garlic cloves for 60 s reduces its anticancer properties (14). Interestingly when microwave heating was applied 10 min after garlic crushing, the anticancer properties were preserved indicating that allinase activation is necessary to generate anticancer compounds which are thermostable (15). In a similar way, the hydroxyl radical scavenging properties of garlic were essentially preserved when garlic extracts were heated at 100°C by 20, 40, or 60 min (16).

There are few processed foods available using garlic. Production techniques for garlic-processed foods such as bread, wheat-gluten, jam, soybean paste, pills, vinegar, bean curd, and tea have been developed but are rarely incorporated into commercial products. Considering garlic's various effects on physiological activity, there is a gross lack of garlic-processed foods that can be consumed without trouble (17,18).

Therefore, in this study, in order to prepare the most favorable garlic beverage, heated garlic juices prepared with varying levels of heat treatment temperature, heat treatment time, and dilutions were prepared to observe their physicochemical properties, with the objective of

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developing a garlic drink with a high level of preference as determined by sensory evaluation.

## MATERIALS AND METHODS

### Sample and sample treatment

Garlic (*Allium sativum* L.) was purchased from the Chungbuk Agriculture and Marine Products Market in June 2006, cloves hull removed, and then stored at  $-20^{\circ}\text{C}$ . The garlic samples (100 g) were placed into a sample bottles (200 mL) and sealed tightly. Sample bottles were placed into the instrument (JISCO, Seoul, Korea) and heat treatment was performed using high-pressure steam generated by the temperature and pressure controlling apparatus (JISCO, Seoul, Korea) (7). The garlic was heated under the conditions described in Table 1. The heated samples were juiced and centrifuged (Hanil, Union5KR, Seoul, Korea) at 3,000 rpm for 10 min, and then the supernatants filtered through Whatman filter paper No. 2. Raw garlic subjected to the same process as heated garlic except for heating.

### Experimental design for response surface methodology (RSM)

RSM was applied to determine the optimum conditions for producing heated garlic juice. As shown in Table 1, the experimental design for processing conditions was made by control composite design. Independent variables such as heating temperature (X1:  $110\sim 130^{\circ}\text{C}$ ), heating time (X2:  $1\sim 5$  hr) and dilution rate (X3:  $2\sim 10$  times) were assigned as numbers (-2, -1, 0, 1, 2), and 16 different intervals were set for processing experiment. The dependent variables (Yn) such as total soluble solid

(Y1), total acidity (Y2), pH (Y3), viscosity (Y4), sensory color (Y5), sensory taste (Y6), sensory odor (Y7) and sensory overall acceptance (Y8) affected by the independent variables were determined three times, and their average values were used for the regression analysis. SAS software (SAS Institute, Cary, NC, USA) was used for analysis of variance (ANOVA), Duncan's multiple range tests (at  $p < 0.05$ ) and regression equation to describe the response surface.

### Physicochemical analysis

The total soluble solids were measured using an Atago hand refractometer (model N-1E, Kyoto, Japan) at  $20 \pm 0.5^{\circ}\text{C}$ . The refractometer prism was cleaned with distilled water after each analysis. Total acidity was measured with 0.1 N sodium hydroxide to an endpoint of pH 8.2 and expressed as percentage of lactic acid (18). The pH of heated garlic juice was measured using a digital pH meter (model 320; Thermo Orion, Beverly, MA, USA). The pH meter was calibrated with commercial buffer solutions at pH 7.0 and 4.0. The viscosity of the heated garlic juice was measured using a viscometer (Cannon Instrument Co., USA), spindle #4 at 10 rpm,  $25^{\circ}\text{C}$  and only the 10th round readings were recorded.

### Sensory evaluation

The sensory evaluation of the 16 heated garlic juice samples was conducted with 20 student judges randomly selected from the Department of Food Science and Technology at Chungbuk National University, Korea. Heated garlic juices were presented in glasses with 3 digit numbers. The judges scored each attribute on a

**Table 1.** Experimental data for total soluble solids, total acidity, pH, viscosity of heat treated garlic juices by central composite design for response surface analysis

Independent variables			Dependent variables			
Heating temp ( $^{\circ}\text{C}$ )	Heating time (hr)	Dilution rate (times)	Total soluble solids	Total acidity (%)	pH	Viscosity (cP)
Control			$6.72 \pm 0.03$	$0.12 \pm 0.01$	$5.74 \pm 0.03$	$1.04 \pm 0.02$
110 (-2)	3 (0)	6 (0)	$5.73 \pm 0.05$	$0.16 \pm 0.17$	$5.71 \pm 0.01$	$1.18 \pm 0.03$
115 (-1)	2 (-1)	4 (-1)	$5.71 \pm 0.07$	$0.22 \pm 0.17$	$5.71 \pm 0.08$	$1.07 \pm 0.03$
115 (-1)	2 (-1)	8 (1)	$5.89 \pm 0.02$	$0.19 \pm 0.17$	$5.89 \pm 0.02$	$0.95 \pm 0.04$
115 (-1)	4 (1)	4 (-1)	$5.19 \pm 0.05$	$0.27 \pm 0.01$	$5.20 \pm 0.06$	$1.05 \pm 0.01$
115 (-1)	4 (1)	8 (1)	$5.19 \pm 0.07$	$0.18 \pm 0.01$	$5.19 \pm 0.07$	$1.09 \pm 0.01$
120 (0)	1 (-2)	6 (0)	$5.85 \pm 0.04$	$0.19 \pm 0.01$	$5.85 \pm 0.04$	$1.02 \pm 0.02$
120 (0)	3 (0)	2 (-2)	$4.85 \pm 0.02$	$0.55 \pm 0.06$	$4.85 \pm 0.03$	$1.18 \pm 0.02$
120 (0)	3 (0)	6 (0)	$5.15 \pm 0.01$	$0.28 \pm 0.07$	$5.15 \pm 0.01$	$1.04 \pm 0.02$
120 (0)	3 (0)	6 (0)	$5.15 \pm 0.01$	$0.28 \pm 0.07$	$5.15 \pm 0.01$	$1.04 \pm 0.02$
120 (0)	3 (0)	10 (2)	$4.92 \pm 0.04$	$0.16 \pm 0.07$	$4.93 \pm 0.05$	$0.97 \pm 0.02$
120 (0)	5 (2)	6 (0)	$4.45 \pm 0.08$	$0.33 \pm 0.01$	$4.45 \pm 0.09$	$1.02 \pm 0.01$
125 (1)	2 (-1)	4 (-1)	$4.87 \pm 0.04$	$0.34 \pm 0.07$	$4.87 \pm 0.05$	$1.04 \pm 0.01$
125 (1)	2 (-1)	8 (1)	$5.04 \pm 0.04$	$0.19 \pm 0.06$	$5.04 \pm 0.05$	$1.01 \pm 0.01$
125 (1)	4 (1)	4 (-1)	$4.20 \pm 0.01$	$0.44 \pm 0.04^{\text{h}}$	$4.21 \pm 0.02^{\text{b}}$	$1.03 \pm 0.01$
125 (1)	4 (1)	8 (1)	$4.17 \pm 0.05$	$0.36 \pm 0.01^{\text{g}}$	$4.18 \pm 0.01^{\text{b}}$	$1.09 \pm 0.05$
130 (2)	3 (0)	6 (0)	$4.00 \pm 0.07$	$0.36 \pm 0.01^{\text{g}}$	$4.01 \pm 0.08^{\text{a}}$	$1.05 \pm 0.05$

five-point hedonic scale (1: dislike extremely, 5: like extremely) for color, taste, flavor and overall acceptance, respectively.

## RESULTS AND DISCUSSION

### Total soluble solid

Table 1 shows the total soluble solid content of heated garlic juice prepared using the various experimental conditions. The total soluble solid content ranged from 4.00 to 5.89 according to treatment conditions. The highest total soluble solid content of 5.89 was observed at 115°C of heating temperature, 2 hr of heating time, and 8 fold dilution ratio. In contrast, the lowest total soluble solid content of 4.0 was observed at 130°C of heating temperature, 3 hr of heating time, and 6 fold dilution ratio. Although it showed that as heat-treatment temperature and time increase, the total solid content tends to gradually decrease, total solid content increased as heat-treatment time increased at low temperature and heat-treatment temperature increased within short time. On the contrary, total solid content tends to decrease as heat-treatment time increased at high temperature. This is considered to be due to an eventual decrease in macromolecule saccharides such as fructan at high temperature which have been degraded and are gradually broken down into smaller saccharides due to heat treatment.

### Total acidity

The total acidity of heated garlic juice prepared under various experimental conditions is shown in Table 1. The total acidity of raw garlic juice is as low as 0.12% but depending on heat treatment conditions, it ranged from

0.16% to 0.55%. The highest total acidity of 0.55% was observed at 120°C heating temperature, heating time of 3 hr, and 2 fold dilution ratio. In contrast, the lowest total acidity of 0.16% was observed with heating at 110°C for 3 hr and a 6 fold dilution ratio. As heating time and heating temperature increased, the total acidity tended to increase. This is considered to be due to increases in the total acid level by the conversion of fructose and glucose in garlic into organic acid by heat treatment. Choi et al. (19) reported that the total acidity of heated pear juice at 100°C for 30min increased. Akinyele et al. (20) observed also an increase of the total acidity after pasteurization of orange juice but Zarate and Ortega (21) did not find variations in total acidity of PEF-treated apple juice when compared with an untreated sample. In this experiment, heating temperature and time as well as dilution ratio significantly affected the total acidity ( $p < 0.001$ , Table 3).

### pH

Changes in the pH of heated garlic juice under various experimental conditions are shown in Table 1. The pH of raw garlic juice was  $5.74 \pm 0.03$ . pH under various experimental conditions was between  $4.01 \pm 0.08$  and  $5.89 \pm 0.02$  showing that it was slightly acidic. The highest pH of 5.89 was observed with heating at 115°C for 2 hr and an 8 fold dilution ratio. In contrast, the lowest pH of 4.01 was observed at 130°C for 3 hr and a 6 fold dilution ratio. As heating temperature and time increased, the pH tended to decrease. These results are likely the result of increases in titratable acidity with increases in heating temperature and heating time. Choi et al. (22) obtained similar results, that is when pear

**Table 2.** Experimental data for sensory properties of heat treated garlic juices by central composite design for response surface analysis

Independent variables			Dependent variables (sensory score)			
Heating temp (°C)	Heating time (hr)	Dilution rate (times)	Color	Taste	Odor	Overall acceptance
110 (-2)	3 (0)	6 (0)	$1.97 \pm 0.29$	$2.08 \pm 0.36$	$2.28 \pm 0.25$	$1.89 \pm 0.19$
115 (-1)	2 (-1)	4 (-1)	$2.28 \pm 0.25$	$1.97 \pm 0.29$	$2.22 \pm 0.69$	$2.00 \pm 0.00$
115 (-1)	2 (-1)	8 (1)	$1.86 \pm 0.34$	$1.86 \pm 0.34$	$2.28 \pm 0.25$	$1.89 \pm 0.19$
115 (-1)	4 (1)	4 (-1)	$4.03 \pm 0.29$	$3.92 \pm 0.36$	$4.06 \pm 0.59$	$4.39 \pm 0.67$
115 (-1)	4 (1)	8 (1)	$2.92 \pm 0.36$	$2.72 \pm 0.54$	$2.25 \pm 0.46$	$2.81 \pm 0.17$
120 (0)	1 (-2)	6 (0)	$1.81 \pm 0.17$	$2.58 \pm 0.22$	$3.20 \pm 0.83$	$2.69 \pm 0.34$
120 (0)	3 (0)	2 (-2)	$2.58 \pm 0.22$	$3.39 \pm 0.35$	$3.67 \pm 0.58$	$3.17 \pm 0.29$
120 (0)	3 (0)	6 (0)	$4.17 \pm 0.44$	$3.56 \pm 0.38$	$4.03 \pm 0.29$	$3.61 \pm 0.15$
120 (0)	3 (0)	6 (0)	$4.17 \pm 0.44$	$3.56 \pm 0.38$	$4.03 \pm 0.29$	$3.50 \pm 0.17$
120 (0)	3 (0)	10 (2)	$2.92 \pm 0.36$	$3.31 \pm 0.67$	$2.42 \pm 0.22$	$2.81 \pm 0.17$
120 (0)	5 (2)	6 (0)	$2.25 \pm 0.55$	$2.78 \pm 0.84$	$3.39 \pm 0.35$	$3.08 \pm 0.14$
125 (1)	2 (-1)	4 (-1)	$3.03 \pm 0.29$	$3.42 \pm 0.22$	$3.42 \pm 0.22$	$3.22 \pm 0.38$
125 (1)	2 (-1)	8 (1)	$3.42 \pm 0.22$	$2.08 \pm 0.14$	$1.69 \pm 0.05$	$2.08 \pm 0.14$
125 (1)	4 (1)	4 (-1)	$1.81 \pm 0.17$	$2.31 \pm 0.34$	$3.81 \pm 0.17$	$2.31 \pm 0.34$
125 (1)	4 (1)	8 (1)	$1.97 \pm 0.29$	$2.11 \pm 0.19$	$3.31 \pm 0.67$	$2.11 \pm 0.19$
130 (2)	3 (0)	6 (0)	$1.81 \pm 0.17$	$1.81 \pm 0.17^e$	$2.39 \pm 0.35^c$	$1.92 \pm 0.14$

**Table 3.** Regression coefficients of the second order polynomials for total soluble solids, total acidity, pH, viscosity, sensory properties with heat treatment conditions

Parameter <sup>1)</sup>	df	Estimate							
		Total soluble solid	Total acidity	pH	Viscosity	Color	Taste	Odor	Overall acceptance
Intercept	1	5.600***	0.272***	5.16***	1.0440***	4.143***	3.368***	3.891***	-3.775***
X <sub>1</sub>	1	-0.128	0.054***	-0.45***	-0.0161*	-0.072	-0.068	0.110	-0.068
X <sub>2</sub>	1	-0.038	0.036***	-0.35***	0.0081	0.064	0.1274	0.264**	0.085
X <sub>3</sub>	1	-2.667**	-0.007***	0.03***	-0.0319***	-0.014	-0.1896*	-0.402***	-0.131*
X <sub>1</sub> × X <sub>1</sub>	1	-0.083	-0.005	-0.07***	0.0142*	-0.562***	-0.391***	-0.425***	-0.491***
X <sub>2</sub> × X <sub>1</sub>	1	-0.082	0.028**	-0.04***	-0.0004	-0.679***	-0.479***	0.020	-0.554***
X <sub>2</sub> × X <sub>2</sub>	1	-0.004	-0.005	0.00	-0.0121	-0.529***	-0.208**	-0.183*	-0.366***
X <sub>3</sub> × X <sub>1</sub>	1	0.097	-0.013	-0.00	0.0113	0.254***	-0.029	-0.054	0.087
X <sub>3</sub> × X <sub>2</sub>	1	-0.005	0.001	-0.05***	0.0321**	-0.1208	-0.004	-0.079	-0.037
X <sub>3</sub> × X <sub>3</sub>	1	1.027***	0.018**	-0.07***	0.0088	0.350***	-0.041	-0.245	-0.254***
R-Square	—	0.9571	0.9052	0.9939	0.5944	0.8596	0.6310	0.6036	0.8299

<sup>1)</sup>X<sub>1</sub>: Temperature (°C), X<sub>2</sub>: Time (hrs), X<sub>3</sub>: Dilution rate (times).

\*p<0.05, \*\*p<0.01 and \*\*\*p<0.001.

juices were processed using heat and pressure, it was observed that pH of the treated juice decreased more than in untreated juice. Heating temperature and heating time as well as dilution ratio significantly affected the pH, and among them, heating temperature and heating time were found to have the most significant effects (p<0.001, Table 3).

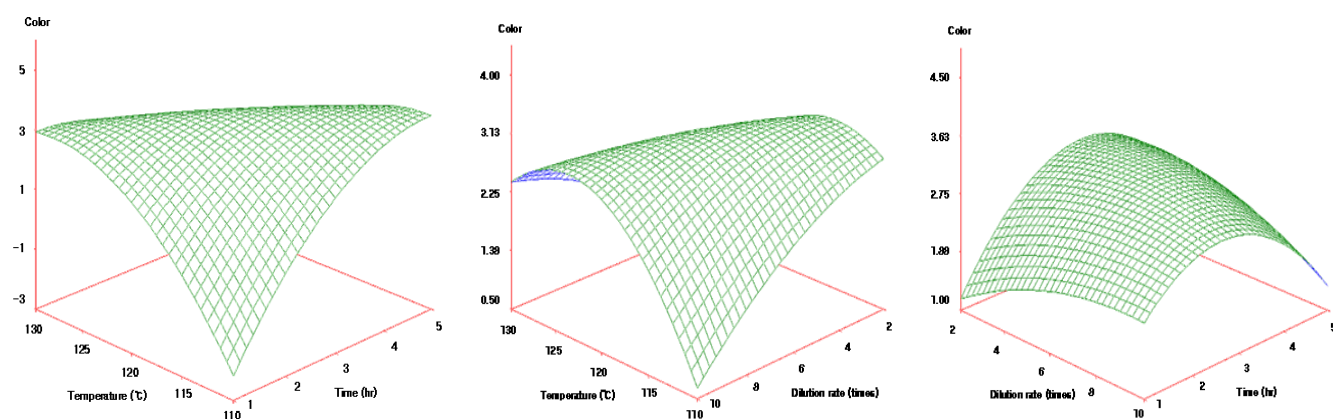
### Viscosity

The viscosities of heated garlic juice under various experimental conditions are shown in Table 1. Viscosity under various experimental conditions was similar to that of water from 0.98 to 1.18 cP, without significant differences. The highest viscosity of 1.18 cp was observed with heating at 110°C for 3 hr and a 6 fold dilution ratio. It was found to have been effected more by dilution ratio than by heat treatment temperature or time. As heating time increased viscosity was increased, and as dilution ratio increased viscosity was decreased. This decrease in viscosity is believed to be the result of in-

creases in reducing sugar as temperature increases. In the Kuo (23) report, it was shown that the viscosity of tomato juice increased linearly as pressure level was elevated from 100 to 500 MPa at various temperatures.

### Sensory evaluation

Table 2 shows the sensory evaluations of color, taste, odor and overall acceptance for the heated garlic juice prepared under various experimental conditions. The preference for the color, taste, odor and overall acceptance of heated garlic juice increased as the heating temperature and heating time increased. However, the preference of the four factors decreased with heating above 120°C. This result occurred because with increases in temperature and time, the color gets darker the taste sour and bitter and stronger. However, preference showed a decreasing trend after 130°C, and as in the result of color, it is possibly because acidity increased as well as a sour taste with increases in heating temperature and time, and the burned taste resulting from long term heat



**Fig. 1.** Effect of temperature, time, and dilution rate on the color of the heated garlic juice.

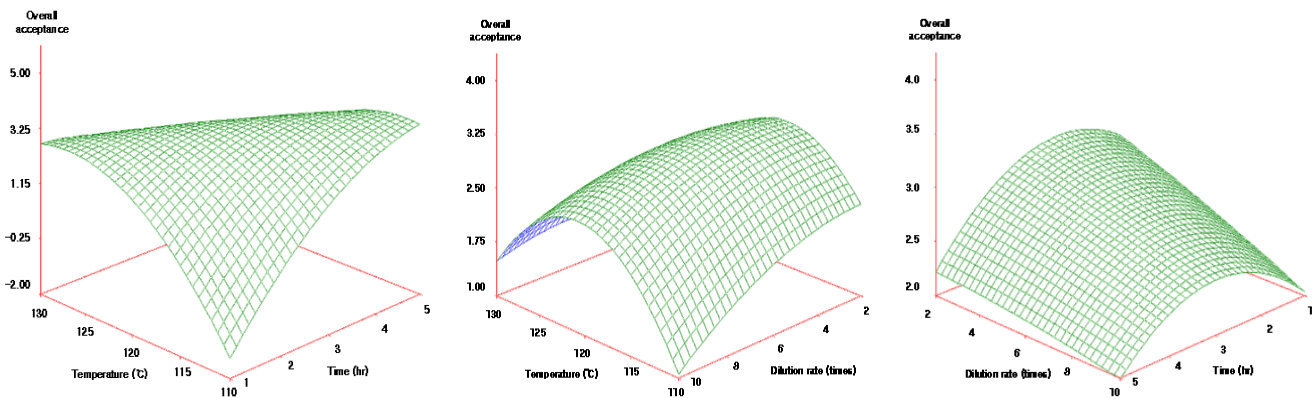


Fig. 2. Effect of temperature, time, and dilution rate on the overall sensory acceptance of the heated garlic juice.

Table 4. Comparison between predicted and observed values for sensory qualities of the heated garlic juice

Responses	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	Predicted value (A)	Actual value (B)
Color	119.41	3.11	5.85	4.16	3.60
Taste	118.23	3.57	3.15	3.66	3.70
Odor	120.54	3.47	5.01	4.23	4.00
Overall acceptance	119.24	3.18	5.66	3.82	3.70

treatment which in turn decreases its likeability as a beverage. Heating temperature and time as well as dilution ratio all affected color (Fig. 1). Among them, Heat treatment temperature and time were found to have the most significant effect ( $p < 0.001$ ).  $R^2$  for the sensory color was 0.8596. As shown in Fig. 2 and Table 3, heating temperature and time as well as dilution ratio all showed significant effects on the overall acceptance, and among them, heat treatment temperature and time were found to have the most significant effect ( $p < 0.05$ ).  $R^2$  for the overall acceptance was 0.8299.

#### Predicted and observed values for sensory qualities of the heated garlic juice

Table 4 shows the comparison between predicted and observed values for sensory qualities of the heated garlic beverage. The optimal conditions observed for heat treated garlic beverage, taking its physicochemical properties into account, were 3 hr treatment time, 120°C temperature and 6 fold dilution. The optimal conditions for best color, taste, odor and overall acceptance were 119.41°C, 3.11 hr, 5.85 fold dilution; 118.23°C, 3.57 hr, 3.15 fold dilution; 120.54°C, 3.47 hr, 5.01 fold dilution; and 119.24°C, 3.18 hr, 5.66 fold dilution, respectively. In the group that was treated for longer than 3 hours at 120°C, likeability decreased due to the bitter and sour tastes. As a result, temperature of 120°C with a duration of 3 hours and 6 fold dilution were the most favorable conditions for optimizing likeability and physicochemical properties. However, since the unfavorable taste of the

garlic remained, further treatments such as decompression deodorization or adding sweeteners or flavor are needed to improve the taste.

#### ACKNOWLEDGMENTS

This study was supported in part by Research & Development Project on Area-based Development in Agriculture, Republic of Korea. The authors acknowledge a graduate fellowship provided by the Ministry of Education through the Brain Korea 21 Program.

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(Received September 25, 2008; Accepted December 3, 2008)