Optimization of microwave heating on orange sacs products

Jun Wang¹², Guijie Li¹², Yujiao Cheng¹², Linhua Huang¹², Rongrong Sun¹², and Houjiu Wu¹²,*

¹National Citrus Engineering Research Center, Chongqing 410125, China
²Citrus research institute, Southwest University, Chongqing 400712, China

* Corresponding author: wuhoujiu@cric.cn

Abstract. Various applications of microwave have been put into effect in the field of food processing. The microwave assisted thermal sterilization (MATS) was of great significance because of its less time consuming and higher efficient. In this paper, response surface methodology was used to optimize the process. The sterilization of orange sacs was optimized using response surface methodology (RSM). The optimal processing parameters of MATS on orange sacs are as follow: microwave power, 6 kW, microwave sterilization time, 350 s, amount of orange sacs, 4500 g. Sterilized under this condition by MATS, orange sacs only maintained 3.7 CFU lethal magnitude which was consistent with the standards requirement. The key factor in the response surface using microwave sterilization and the interaction between them were also determined and discussed which could make it evident that MATS should be an idea candidate in the sterilization of some fruit or vegetable products as ready to eat. From these results, the best conditions of microwave sterilization are power 6kw, microwave sterilization time 350s and material quantity 4500g.

1. Introduction

Orange juice is sweet and sour, rich in flavor, and rich in vitamin C, carotene and other nutrients, which is deeply loved by consumers [1-2]. At present, the key to the popular orange juice beverage in the market is the suspension of juice cells. Generally, juice cells are required to be full, uniform in size, bright in color and rich in vitamin C. However, in the process of industrial production, storage and transportation, juice cells are easy to be polluted by microorganisms, resulting in spoilage [3-4]. Therefore, it is particularly important to find a bactericidal technology that can control the growth and reproduction of microorganisms, and reduce the spoilage and deterioration of juice cell quality.

At present, orange juice often uses thermal sterilization technology (pasteurization and high temperature instantaneous sterilization) [5-6]. Pasteurization temperature is too low, so the sterilization effect is not ideal; high temperature instantaneous sterilization can achieve better sterilization effect, but it will lead to excessive loss of nutritional quality of fruit juice. In recent years, there are more and more ways different from traditional sterilization, such as microwave sterilization, ultra-high pressure sterilization, pulse electric field sterilization, UV sterilization and so on [7-9]. Microwave technology can reduce the number of microorganisms in fruit juice, but also maintain the nutrition, flavor and safety quality of the product. It has achieved good results in apple juice, grapefruit juice, blueberry juice and other fruit juice sterilization [10-14].

In this study, microwave sterilization was used to treat juice cells in bags, and the best way of sterilization was selected. At the same time, the nutritional quality of navel orange and summer orange
juice cells after microwave sterilization was evaluated in order to provide scientific guidance for the application of microwave sterilization technology in juice cell sterilization.

The process optimization of microwave heating orange sacs products had not been studied. This research is based on the laboratory experiments, the factory research is needed in the future.

2. Methods

2.1. Preparation of juice cells of navel orange and sweet orange
Fruit washing → Enzyme treatment, peeling, whole fruit ball rinsing → Juice cell separation with juice cell disperser → Removal of damaged capsule, seeds and other sundries → Juice cell filtration with 20 mesh sieve of JBT pulp tester → Juice cell and fresh fruit juice 6:4 mixing, packaging and sealing with high temperature resistant cooking bags, 200 ± 5g per bag.

2.2 Single factor test design
For navel orange juice cells, the microwave power was 6 kW, the sterilization time was 150 s, and the material quantity is 3500 g. The effects of microwave power (3, 6, 9 kW), sterilization time (50, 100, 150, 200, 250, 350 s), and material quantity (1500, 2500, 3500, 4500, 5500 g) on the germicidal efficacy of navel orange juice cells were investigated.

2.3 Response surface test optimization of microwave sterilization process
According to the design principle of box Behnken test and the results of single factor test, three factors, namely microwave power, sterilization time and material quantity, were taken as independent variables, and the order of magnitude y (Formula 1) with the total number of colonies reduced was taken as the response value to design the test. See Table 1 for test factors and level design. Formula 1 was $Y = -\log(N/N_0)$, in the formula: Y was the order of magnitude of the reduction of the total number of colonies; N was the total number of bacteria in 1 mL juice after microwave treatment; N0 was the total number of bacteria in 1 mL juice of the control group.

| Factor               | Code | Coding level |
|----------------------|------|--------------|
| Power (kW)           | $X_1$| 3 6 9        |
| Time (s)             | $X_2$| 150 250 350  |
| Sacs weight (g)      | $X_3$| 2500 3500 4500 |

2.4 Microbiological determination
Take the sterilized sample into 5, 10, 20 times test tube for dilution, take 1 mL sample to 3m test piece, cover the surface film and press it gently with finger pressure plate. Three parallel experiments were conducted for each treatment. The total number of colonies was cultured in 37°C constant temperature incubator for 48 h, and the control group was used as the control group. Take out the test group and the control group, count and record, and finally calculate the sterilization rate. Sterilization rate (%) = (number of control group - number of experiment group)/number of control group ×100.

2.5 Determination of soluble solid content (SSC), total acid (TA), vitamin C (Vc)
SSC was determined by hand-held saccharometer, and TA was determined by acid-base titration, and Vc was determined by 2, 6- two chlorophenol indophenol titration.

2.6 Hardness measurement
The hardness of orange juice cell was measured by texture analyzer. The parameters were: TPA measurement mode, P/0.5 down pressure probe. The speed before measurement was 5 mm/s, and the
speed after measurement was 1 mm/s, meanwhile the speed after measurement was 5 mm/s, the compression rate was 30%, and the initiating force was 5 g.

2.7 Color difference measurement
The color of juice cells was measured by color i5 colorimeter. The juice cells of each variety were measured 10 times, and the average value was taken. Chroma measurement was mainly based on CIE L*, a*, b* color standards, respectively, to determine L*, a*, b* values of juice cells.

2.8 Statistical analysis of data
Using the software of origin 8.0 and SPSS13.0, the one-way ANOVA and Duncan multiple comparison were carried out, and the significance level was 0.05.

3. Results and discussion

3.1 Microwave sterilization of navel orange juice sacs

3.1.1 Influence of microwave power on sterilization rate
The fixed sterilization time was 150 s, the material quantity was 3500g, the range of sterilization power was 3, 4.5, 6, 7.5 and 9 kW, and the better power was selected with the sterilization rate as the index. It could be seen from Fig. 1 that with the increase of microwave power, the number of colonies in the juice cell showed a downward trend, and the sterilization rate has increased significantly. When the microwave power was 6kW, the sterilization rate reached 97.50%, which was significantly different from the microwave power of 3.0 kW, 4.5 kW, 9 kW treatment group (P<0.01), but not significantly different from the 7.5 kW treatment group (P>0.05). In addition, there was no significant difference between the 7.5 kW treatment group and 9 kW treatment group (P>0.05). Considering the comprehensive energy consumption, it was suitable to choose the microwave power of 6 kW.

![Fig. 1. Effects of microwave power on sterilization rate.](image)

3.1.2 Influence of microwave time on sterilization rate
Microwave sterilization equipment determines the processing time through the rotation speed of the conveyor belt, and the rotation speed is related to the processing time. Fixed sterilization power 6 kW, sacs weight 3500 g, microwave time range of 50, 100, 150, 250 and 350 s, with sterilization rate as the index to choose the better time. It could be seen from Fig. 2 that with the increase of microwave time, the number of colonies in the juice cell showed a downward trend, and the sterilization rate showed an upward trend. When the microwave time was 50, 100, 150, 250 and 350 seconds, the sterilization rate was 96.5%, 97.0%, 98.22%, 99.5% and 99.6% respectively. The difference of sterilization time between 250 s treatment group and 50s, 100s and 150 s treatment group was very significant (P<0.01), but not between 350 s treatment group and 250 s treatment group (P>0.05). According to the comprehensive sterilization efficiency and energy consumption ratio, 250 s was the best microwave treatment time.
3.1.3 Influence of sacs weight on sterilization rate

In microwave sterilization, the sacs weight determines the microwave absorption efficiency. Fix the sterilization power 6 kW, time 250 s, and set the range of sacs weight 1500, 2500, 3500, 4500 and 5500 g; meanwhile select the better sacs weight with sterilization rate as the index. As shown in Fig. 3, with the increase of sacs weight, the sterilization rate showed an upward trend. When the sacs weight was 1500, 2500, 3500, 4500 and 5500 g, and the sterilization rate was 99.96%, 97.94%, 99.47%, 98.18% and 97.33% respectively. In addition to 1500 g treatment group, the difference between 2500 g treatment group and each treatment group was very significant (P<0.01). Considering working efficiency, energy consumption and other factors, 3500 g was the better sacs weight.

3.2 Model establishment and significance test

Using the software of design expert 7.1.6 to fit the test data of Table 2, we can see that the quadratic multiple regression model equation of the reduction of the total number of colonies of orange juice after microwave treatment to the independent variable microwave power, temperature and sterilization time was $Y=3.59+0.37x1+0.35x2+0.16x30.023x1x2+0.020x2x3-0.45x1^2-0.16x2^2-0.22x3^2$. Table 3 was the variance analysis of the model, and table 4 was the significance test of the model coefficient.

It could be seen from Table 3 that $F=37.32>F_{0.01}(9,4)=14.66$, $P<0.0001$, indicating that the model was extremely significant, and the difference between different treatments is extremely significant. $F=6.3<F_{0.05}(9,3)=8.81$, missing item $P=0.0537>0.05$, the difference was not significant, which showed that the residual error is caused by random error. The correction decision coefficient of the model $R^2_{Adj}=0.9533$, which shows that 95.33% of the response value can be explained by the model, only 4.67% of the variation can not be explained, further indicating that the model fitting is good, the
test error is small, and the model can be used to analyze and predict the order of magnitude of the reduction of the total bacterial count of the juice cells after microwave treatment.

It could be seen from the significance test of regression equation coefficient in Table 4 that the first term X1 (P<0.0001), X2 (P<0.0001), X3 (P<0.0001), the second term X12 (P<0.0001), X32 (P=0.0037) of model equation are extremely significant; the second term X22 (P=0.0145) is significant, and the interaction terms are not significant.

Table 2. Factors, levels, and results of response surface tests.

| No. | Coding level | Response value |
|-----|--------------|----------------|
| 1   | 1            | 2.92           |
| 2   | 1            | 3.70           |
| 3   | -1           | 3.07           |
| 4   | 0            | 3.55           |
| 5   | 0            | 3.64           |
| 6   | 1            | 3.02           |
| 7   | 0            | 3.02           |
| 8   | 0            | 3.36           |
| 9   | 0            | 3.6            |
| 10  | -1           | 2.6            |
| 11  | 1            | 3.64           |
| 12  | 1            | 3.64           |
| 13  | 0            | 3.62           |
| 14  | 0            | 2.84           |
| 15  | 0            | 2.2            |
| 16  | 0            | 3.51           |
| 17  | -1           | 2.42           |

Table 3. Analysis of variance of the developed regression model.

| Variation source | Sum of squares | Freedom | Mean square | F value | P value | Saliency |
|------------------|----------------|---------|-------------|---------|---------|----------|
| Model            | 3.59           | 9       | 0.40        | 37.32   | < 0.0001 | **       |
| Residual         | 0.075          | 7       | 0.011       | /       | /       | /        |
| Missing item     | 0.062          | 3       | 0.021       | 6.30    | 0.0537  | /        |
| Pure error       | 0.013          | 4       | 0.00327     | /       | /       | /        |
| Sum              | 3.67           | 16      | /           | /       | /       | /        |
| R²=0.9796        | R² Adjusted=0.9533 | /     | /           | /       | /       | /        |

Table 4. Significance test of regression coefficients of the developed regression model.

| Coefficient term | Regression coefficient | Freedom | Standard error | Lower confidence limit | Upper confidence limit | P value |
|------------------|------------------------|---------|----------------|------------------------|------------------------|---------|
| Constant term    | 3.59                   | 1       | 0.046          | 3.48                   | 3.70                   | /       |
| X₁               | 0.37                   | 1       | 0.037          | 0.29                   | 0.46                   | < 0.0001|
| X₂               | 0.35                   | 1       | 0.037          | 0.26                   | 0.43                   | < 0.0001|
| X₃               | 0.16                   | 1       | 0.037          | 0.069                  | 0.24                   | 0.0038  |
| X₁X₂             | -0.023                 | 1       | 0.052          | -0.14                  | 0.100                  | 0.6766  |
| X₁X₃             | 0.11                   | 1       | 0.052          | -0.012                 | 0.23                   | 0.0710  |
| X₂X₃             | 0.020                  | 1       | 0.052          | -0.10                  | 0.14                   | 0.7105  |
| X₁²              | -0.45                  | 1       | 0.050          | -0.57                  | -0.33                  | < 0.0001|
| X₂²              | -0.16                  | 1       | 0.050          | -0.28                  | -0.044                 | 0.0145  |
| X₃²              | -0.22                  | 1       | 0.050          | -0.33                  | -0.096                 | 0.0037  |
In order to judge the contribution rate of the primary term, interactive term and secondary term of each factor to the sterilization effect, it is suitable to compare the contribution rate of each factor. The size of each factor is shown in Table 5. It can be seen that the leading effect of the equation is the microwave power, the primary time effect and the secondary power effect. The effect of the three test factors is $X_1 > X_2 > X_3$.

**Table 5. Contribution rates of various factors.**

| Factor   | Sum of squares | Freedom | Contribution rate (%) |
|----------|----------------|---------|-----------------------|
| $X_1$    | 1.120          | 1       | 31.517                |
| $X_2$    | 0.960          | 1       | 27.015                |
| $X_3$    | 0.190          | 1       | 5.347                 |
| $X_1X_2$ | 0.002          | 1       | 0.057                 |
| $X_1X_3$ | 0.048          | 1       | 1.351                 |
| $X_2X_3$ | 0.002          | 1       | 0.045                 |
| $X_1^2$  | 0.860          | 1       | 24.201                |
| $X_2^2$  | 0.110          | 1       | 3.095                 |
| $X_3^2$  | 0.200          | 1       | 5.628                 |
| Missing item | 0.062      | 3       | 0.017                 |
| Pure error | 0.013       | 4       | 0.004                 |

3.3 Contour and response surface analysis of microorganism response value of navel orange sacs treated by microwave

The response surface and contour map of the model equation were shown in Fig. 4, 5 and 6. The microwave sterilization conditions of juice cells were optimized by reducing about 3.5 orders of magnitude. As shown in Fig. 4, when the amount of material was 3500 g, the interaction effect of microwave power and microwave time on the number of colony death was significant. In the range of microwave power 4.59–9 kW and microwave sterilization time 207.59–350 s, the total number of colonies was 3.50. And in a certain range, with the increase of microwave power, the death order of the total number of colonies in the juice cell increased sharply, and the increasing trend was more significant than that caused by the extension of microwave sterilization time. When the microwave power was lower than 7.36 kW, and the number of colony death decreases with the decrease of microwave sterilization time.

Fig. 4. Response surface plot and its contour plot showing the effects of microwave power, treatment time and their mutual interactions on totalviable count reduction of Orange juice sacs after sterilization.

Fig. 5 showed that when the microwave time was 250 s, the interaction effect of microwave power and orange sacs on the death level of the total number of colonies was significant. In the range of microwave power 5.33-9 kW and orange sacs weight 2961.93-4500 g, the death level of the total number of colonies could reach 3.50 or above. When the microwave power was at 7.09 kW, the amount of material was in the range of 2961.93-3442.61 g, and the death order of the total number of colonies decreases with the increase of the amount of orange sacs.
As shown in Fig. 6, when the microwave power is 6 kW, within the microwave sterilization time of 221.82-350 s and the orange sacs of 2707.81-4500 g, the death level of the total number of colonies was 3.50. When the orange sacs weight was fixed in the range of 2708.81-4500 g, with the increase of microwave sterilization time, the total number of colonies would be increased. When microwave sterilization time was fixed at 221.82-350 s, and the amount of orange sacs increased from 38779.26 g to 38779.26 g, which would lead to the death of the total number of colonies, and then gradually decreased.

Fig. 5. Response surface plot and its contour plot showing the effects of microwave power, weight quantity and their mutual interactions on total viable count reduction of Orange juice sacs after sterilization.

Fig. 6. Response surface plot and its contour plot showing the effects of microwave power, treatment time and their mutual interactions on total viable count reduction of Orange juice sacs after sterilization.

3.4 Model validation
The optimal conditions of microwave sterilization of orange juice cells were obtained by the software design expert 7.1.6. When the microwave power was 6 kW, the time of microwave sterilization was 350 s, and the amount of materials was 4500 g, the death level of the total number of colonies reached 3.73. In order to verify the validity of the model, 5 groups of model validation experiments were conducted with response value of 3.73, and the results were shown in Table 6. The results showed that the relative error between the measured value and the predicted value was less than 2%, which proves that the key factor of optimizing the technological parameters of microwave sterilization of orange sacs by response surface was feasible. By using design expert and response surface method (RSM), the second multiple mathematical model of microwave sterilization of orange sacs was established, which was proved to be reasonable and reliable. At the same time, the key factors and interaction of shadow microwave sterilization were discussed by using the response surface and its contour lines of the model, and the process parameters of killing 3.77 order of magnitude colonies were optimized. They are microwave power 8 kW, microwave sterilization time 324 s and orange sacs 3200 g.
Table 6. Validity verification of the developed regression model.

| Experiment group | Factor | Response value | Relative error (%) |
|------------------|--------|----------------|-------------------|
| | $x_1$/kw | $x_2$/s | $x_3$/g | Measured | Forecast |
| 1 | 6 | 350 | 4500 | 3.71 | 3.73 | 0.53% |
| 2 | 6 | 350 | 4500 | 3.77 | 3.73 | 1.33% |
| 3 | 6 | 350 | 4500 | 3.75 | 3.73 | 0.53% |
| 4 | 6 | 350 | 4500 | 3.76 | 3.73 | 0.80% |
| 5 | 6 | 350 | 4500 | 3.74 | 3.73 | 0.37% |

3.5 Comparison of nutritional indexes of navel orange sacs and summer orange juice cells treated by the best microwave processing parameters

As shown in Table 7, under the best technological conditions, microwave sterilization could reduce the nutritional quality of juice cells. There was no significant difference in the effect on SSC and TA ($P>0.05$); the loss rate of Vc was the most serious, which was 16.4-18%, which was mainly related to the degradation of Vc caused by the thermal effect of microwave sterilization. The increase of hardness index was related to citrus varieties, for example, there was significant difference before and after sterilization ($P<0.05$). There was no significant difference before and after sterilization ($P>0.05$). Microwave sterilization also had a certain effect on the color of juice cells. The values of $L^*$ and $B^*$ were increased, $a^*$ is slightly decreased, and $L^*$ of summer orange is larger, with better brightness; $a^*$ of navel orange is larger, with better red, the difference of color is mainly related to the carotene contained in the fruit.

Table 7. Comparison of nutritional indexes of navel orange and summer orange by microwave treatment.

| Treatment | Navel orange | Summer orange |
|-----------|--------------|---------------|
| | Before sterilization | After sterilization | Before sterilization | After sterilization |
| Nutritional indicator | | | | |
| SSC (°Brix) | 12.8±0.1 | 12.6±0.2 | 10.2±0.2 | 10.0±0.2 |
| TA (mg/100 mL) | 7.6±0.1 | 7.4±0.1 | 15.5±0.2 | 15.4±0.1 |
| Vc (mg/100 mL) | 42.0±2.0 | 35.1±2.0 | 40.6±2.1 | 33.3±1.5 |
| Hardness (g) | 113±3.0 | 115±3.0 | 115±2.0 | 125±2.10 |
| Color difference ($L^*$) | 40.2±0.1 | 42.3±0.3 | 45.5±0.4 | 48.7±0.4 |
| Color difference ($a^*$) | 3.2±0.3 | 2.6±0.1 | 2.5±0.2 | 2.0±0.2 |
| Color difference ($b^*$) | 25.2±0.7 | 26.1±0.3 | 21.2±0.3 | 22.0±0.1 |

4. Conclusion

The best conditions of microwave sterilization are power 6kw, microwave sterilization time 350s and material quantity 4500g. Compared with thermal sterilization, microwave sterilization is safe and efficient. It can be completed in 350s and meet the requirements of relevant health indicators. It not only consumes less energy than thermal sterilization, but also has less impact on color. What's more, the industrialization of microwave continuous treatment related equipment is mature, which has the basic conditions to promote the application to the industry, and has the potential to replace the traditional thermal sterilization of the factory. The nutritional indexes of different varieties (navel
orange and summer orange) changed after microwave sterilization. Compared with the nutritional indexes of navel orange and summer orange, it was found that the content of soluble solids of navel orange was higher, the color a* was larger, and the acidity was suitable. Compared with summer orange, navel orange is more suitable for juice processin

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