Advance Journal of Food Science and Technology 12(12): 679-687, 2016
DOI:10.19026/ajfst.12.3329
ISSN: 2042-4868; e-ISSN: 2042-4876

Submitted: January 29, 2016 Accepted: May 5, 2016 Published: December 25, 2016

**Research Article**

# Optimization for Brewing Technology of *Jujube* Brandy Using Response Surface Methodology

Yanan Xia, Lijuan Hou, Yanli Ma and Jie Wang
College of Food Science and Technology, Agricultural University of Hebei, Baoding 071000, China

**Abstract:** In order to obtain a proper brewing method of *jujube* brandy, one-factor experiment and response surface methodology were applied to get the maximum alcohol content. Using single-yeast GH and fermentate at 28°C for 20d was suggested by one-factor test. The use of a central composite design and the response surface methodology to determine the best conditions allows the optimum combination of analytical variables (yeast strains, fermentation temperature and time) to be identified: single-yeast GH, fermentation temperature of 18°C, fermentation time of 24d and the alcohol content was 38.7%vol, almost accords with the predicted data. The optimized process improved the mellow flavor of *jujube* brandy, which has great practical values.

**Keywords:** Alcohol, flavor compounds, *jujube* brandy, one-factor tests, response surface method

## PRACTICAL APPLICATIONS

*Jujube* output increases rapidly in China, Hebei is a major produce place of *jujube*, but the development of *jujube* brandy in trade market is restricted severely because of lacking mature production technology. The present study provides a proper brewing method for *jujube* brandy. The results indicated that the fermentation temperature and time have more significant effect on quality of *jujube* brandy than yeast strains. The new fermentation process was feasible for brewing *jujube* brandy with higher alcohol content and richer flavor compounds, which would be helpful to brew other brandy products.

## INTRODUCTION

*Jujube* brandy, a unique brandy product in China, has a long history. *Jujube* brandy is produced by solid fermentation, distillation and aging using Chinese *jujube* as raw material. However, since mature production technology is lacking, development of *jujube* brandy in trade market is restricted severely, it cannot be produced as a standardized commodity.

*Jujube* is one of the characteristic fruit in China. The total cultivating area of *jujube* in China has reached 3200000 hectares by 2012, with annual output of 4.683 million tons. Hebei is a major produce place of *jujube*, but the development of processing technology and high value-added products need to be improved. Studies have shown that *jujube*s are rich in sugar and contain similar components as grapes, which means *jujube* are proper to produce brandy (Claus and Berglund, 2005; Li *et al*., 2007).

Fermentation conditions are the decisive factor of quality and flavor of liquor (Jackson, 2002). The main factors influencing the liquor aroma components include yeast strains, fermentation temperature and time (Rapp, 1998). In western countries, brandy is produced with grape juice or hide trimmings and different kinds of yeasts (Jiming and Puchao, 2004; Jijun *et al*., 2005). Britain liquor brewster think that the best temperature for brewing fruit wine is between 22-25°C, because low fermentation temperature could reduce the generation of higher alcohols (Huafeng *et al*., 2003). But for the French and German winemaker, 15-18°C is considered the best temperature for fermentation for a long time (Qianwen and Zhengjun, 2000). Daqu and solid-state fermentation are characteristic of Chinese traditional liquor production techniques (Zheng *et al*., 2011; Berradre *et al*., 2009; Zhang *et al*., 2013) and have recently been used in the brewing of fruit wine, bringing unique flavors and improving the quality of production (Chang *et al*., 2014; Fan and Qian, 2005). Most white wines in China have long fermentation time at the temperature of 25-30°C, maybe as long as 3 months (Fan and Qian, 2006; Zhu *et al*., 2007; Luo *et al*., 2008; Fan *et al*., 2011).

In this study, yeast strains, fermentation temperature and time were selected for one-factor experiment, then the response surface analysis test was

**Corresponding Author:** Jie Wang, College of Food Science and Technology, Agricultural University of Hebei, Baoding 071000, China, Tel.: +86-13131262819

performed to get the optimal fermentation parameters, which would obtain higher quality *jujube* brandy.

## MATERIALS AND METHODS

**Samples:**

***Jujube***: Dried Ziziphus*jujube* (Hebei, Fuping).

**Brewing process of *jujube* brandy:**

- Add equal water to shredded *jujube*, soak 5-6 h.
- Boil, add 1/6 rice hull after cooling.
- Take 1.5% yeast or Jiuqu in 100 mL of 2% glucose water, 40°C water baths for 30 min. Inoculate activated yeast or Jiuqu.
- Solid-state fermentate, then distill, store.

**Alcohol test:** Alcohol content is tested with alcohol meter. All of the analyses were performed three times.

**SPME-GC-MS parameters:** *Jujube* brandy was diluted to 10% alcohol content by distilled water. 1 g NaCl was added to 7.5 mL of sample solution in a 20 mL sealed glass vial. Flavor compounds were exacted at 40°C for 40 min with 50/30μm DVB/CAR/PDMS fiber, then used to GC-MS analysis.

Flavor compounds of *jujube* brandy were detected by GC-MS (Agilent 5975 Mass Spectrometer coupled to an Agilent 7890A Gas Chromatograph, DB-WAX column, 60 m×0.25 mm ID and 0.25 μm film thickness, USA). The injector temperature was 250°C, EI source was 230°C, MS Quad was 150 °C and transfer line was 250°C. The initial temperature was 50 °C for 3 min, which was increased to 80°C at a rate of 3 °C/min. The temperature was further raised to 230°C at 5°C/min and maintained at 230°C for 6 min. The carrier gas had a flow rate of 1.0 mL/min. Samples were injected using the splitless mode. A mass range of 50-550 m/z was recorded at one scan per second.

Table 1: Independent variables and their levels used in the response surface design

| Level | $X_1$ (Yeast strains) | $X_2$ (Temperature/°C) | $X_3$ (Time/d) |
|---|---|---|---|
| -1 | Single-yeast | 18 | 8 |
| 0 | Mixd-yeast | 24 | 16 |
| 1 | Jiuqu | 30 | 24 |

**Qualitative and quantitative analysis:** Flavor compounds were identified by Nist 2005 library of GC-MS. The contents of flavor compounds were quantified using an internal standard (3-octanol, 99%, Sigma-Aldrich).

$$m_i = (f^*A_i)/(A_s/m_s),\ f = (A_s/m_s)/(A_r/m_r)$$

$m_i$, $m_s$, $m_r$ represent contents of determinand, internal standard, contrast, $A_i$, $A_s$, $A_r$ represent peak area or peak height of determinand, internal standard, contrast, f represent correction factor.

**Experimental:** Six kinds of yeast strains (single-PH, PZ, GH, SX, mixed-GS, HGS, Anqi yeast company, China), 5 kinds of Jiuqu (N, J, Q, AQ and ZJ, Anqi yeast company, China), fermentation temperature (15, 18, 24, 28, 32°C), fermentation time (6, 10, 14, 20, 24, 28d) was performed as one-factor test.

**Box-Behnken design:** Based on one-factor test, a Box-Behnken Design (BBD) with three independent factors ($X_1$, yeast strains; $X_2$, fermentation temperature; $X_3$, fermentation time) set at three variation levels was implemented (Table 1). And +1, 0, -1 encoded factors represent variables (Ni and Zeng, 2010). The alcohol content of *jujube* brandy was selected as the responses for the combination of the independent variables (Table 2).

## RESULTS AND DISCUSSION

**One-factor test results:**

**Yeast strains:** Besides ZJ Jiuqu, alcohol of *jujube* brandy maintain between 33 to 36% vol. *Jujube* brandy

Table 2: Variable levels and responses of flavor content based on yeast, fermentation temperature and time

| Run | Yeast strains ($X_1$) | Temperature ($X_2$/°C) | Time ($X_3$/d) | Observed ($Y_0$/%vol) | Predicted (Y/%vol) |
|---|---|---|---|---|---|
| 1 | 3 | 18 | 16 | 33.4 | 33.45 |
| 2 | 2 | 24 | 16 | 34.4 | 35.74 |
| 3 | 2 | 24 | 16 | 36.6 | 35.74 |
| 4 | 2 | 30 | 24 | 36.6 | 36.69 |
| 5 | 2 | 24 | 16 | 36.2 | 35.74 |
| 6 | 2 | 18 | 24 | 38.0 | 38.21 |
| 7 | 1 | 24 | 24 | 38.8 | 38.76 |
| 8 | 2 | 24 | 16 | 35.3 | 35.74 |
| 9 | 3 | 24 | 24 | 37.2 | 36.94 |
| 10 | 2 | 24 | 16 | 36.2 | 35.74 |
| 11 | 3 | 30 | 16 | 34.6 | 34.77 |
| 12 | 3 | 24 | 8 | 36.0 | 36.04 |
| 13 | 1 | 24 | 8 | 35.5 | 35.76 |
| 14 | 1 | 30 | 16 | 33.2 | 33.15 |
| 15 | 2 | 18 | 8 | 35.9 | 35.81 |
| 16 | 2 | 30 | 8 | 35.4 | 35.19 |
| 17 | 1 | 18 | 16 | 36.8 | 36.63 |

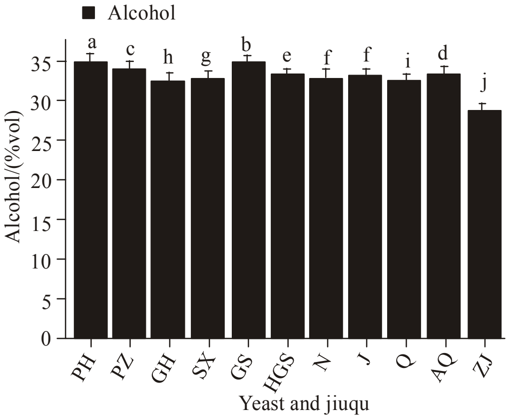


Fig. 1: Influence of yeast and Jiuqu on the alcohol of *jujube* brandy

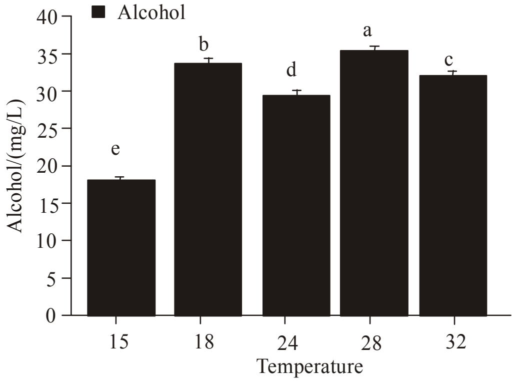


Fig. 2: Influence of fermentation temperature on the alcohol of *jujube* brandy

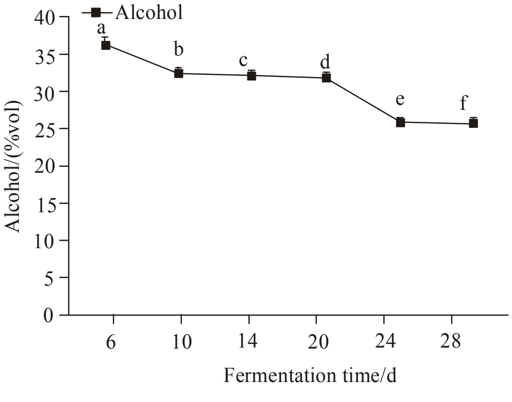


Fig. 3: Influence of fermentation time on the alcohol of *jujube* brandy

fermented with single-yeast PH and mixed-yeast GHSX have higher alcohol than others (Fig. 1). Therefore, single-yeast GH, PH and mixed-yeast GHSX are proper yeast strains for brewing *jujube* brandy.

**Fermentation temperature:** Significant difference of alcohol appeared with different fermentation temperatures ($p<0.05$). *Jujube* brandy got the highest alcohol at 28°C, then at 18°C, the least at 15°C (Fig. 2). Therefore, the proper temperature for brewing *jujube* brandy is 28°C.

**Fermentation time:** Significant difference of alcohol also appeared with different fermentation time ($p<0.05$). *Jujube* brandy got the highest alcohol at 6d, then decreased gradually, which means *jujube* brandy got fully fermentation during 6d, then went on flavor generation reaction (Fig. 3). Therefore, although alcohol fermentation finish at 6d, for obtaining high-quality-flavor *jujube* brandy, 20d should be chosen to be the proper fermentation time.

**Box-Behnken result:**

**Statistical analysis and model building:** Seventeen tests were complemented as Box-Behnken designing (Table 2). Regression and variance analysis was carried out to determine the coefficient of determination, lack of fit and the significance of the linear, interaction effects and quadratic of the independent variables on the response (Table 3).

F-test and p-value were used to determine the significance of each coefficient (Table 3). The p-value represents the significance of the corresponding coefficients in terms of alcohol content, with a smaller p-value indicating more significant impact of the corresponding coefficient. The results of regression coefficient analysis showed that the variable with the largest effect was the quadratic term of fermentation time ($X_3^2$), followed by liner term of fermentation time ($X_3$), which were extremely significant ($p<0.01$). Also, the quadratic term of fermentation time ($X_2^2$) and the interaction effects of yeast strains and fermentation temperature ($X_1X_2$) were significant ($p<0.05$). However, the interaction effects of yeast strains and fermentation time ($X_1X_3$), fermentation temperature and time ($X_2X_3$), the quadratic term of fermentation temperature ($X_1^2$), liner term of yeast strains ($X_1$) were not significant ($p>0.05$).

Design Expert was applied to make regression fitting analysis, the quadratic model was obtained as follows:

$$Y=33.835-2.4575X1+0.67875X2-0.41188X3+0.2X1X2-0.065625X1X3-4.68750E\text{-}003X2X3-0.42X1\hat{}2-0.022778X2\hat{}2+0.024297X3\hat{}2$$

where, Y is the predicted response (alcohol content of *jujube* brandy) and X1, X2, X3 are coded values of yeast strains, fermentation temperature and fermentation time, respectively.

From F-test, the low value of CV (1.96) indicates that the experiments are precise and reliable (Prakash Maran *et al*., 2013). The determination coefficient ($R^2$)

implies that the sample variation of 90.03% for the alcohol content of *jujube* brandy is attributed to the independent variables. Meanwhile, the high $R^2$ (0.9003), adj-$R^2$ (0.7720) and pre$R^2$ (0.7143) clearly demonstrated that the experiment and the theoretical values predicted by polynomial model had a very close agreement. From the analysis, the F-value of 7.02 and p-value<0.01 indicates the response surface quadratic model was significant. Furthermore, results of the ANOVA indicated that the lack of fit of 0.9359 was insignificant.

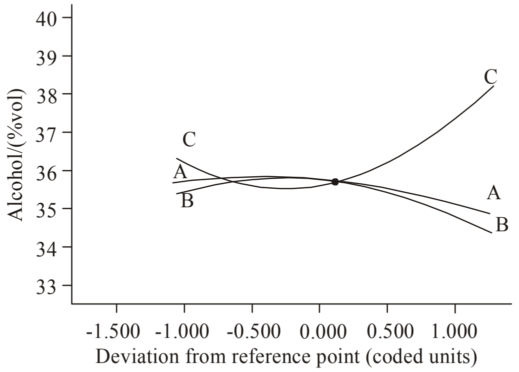


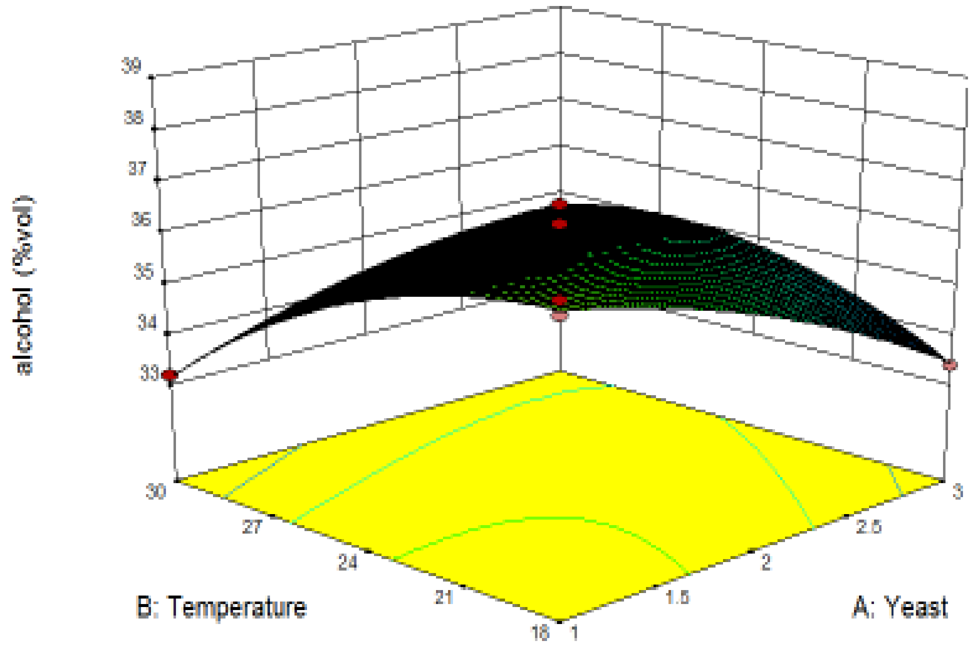
Fig. 4: Perturbation plot showing the effect of process variables

**Analysis of response surface:**

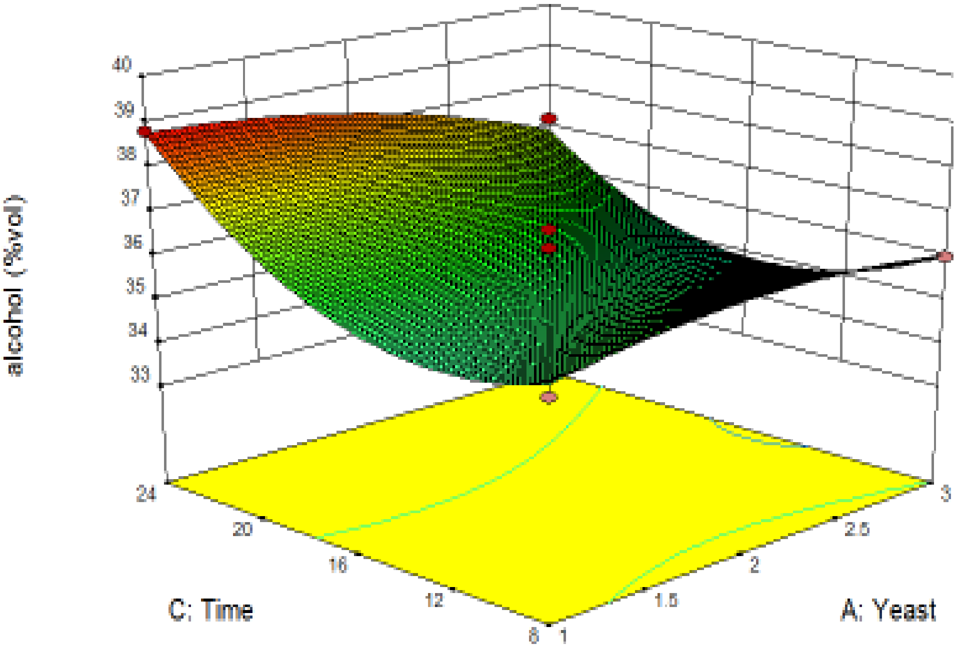
**Perturbationplot:** Perturbation plot could be used to find the most effective factors by the steep slope or

Table 3: Analysis of Variance (ANOVA) for response surface quadratic model for flavor content of *jujube* brandy and independent variables ($X_1$, $X_2$, $X_3$)

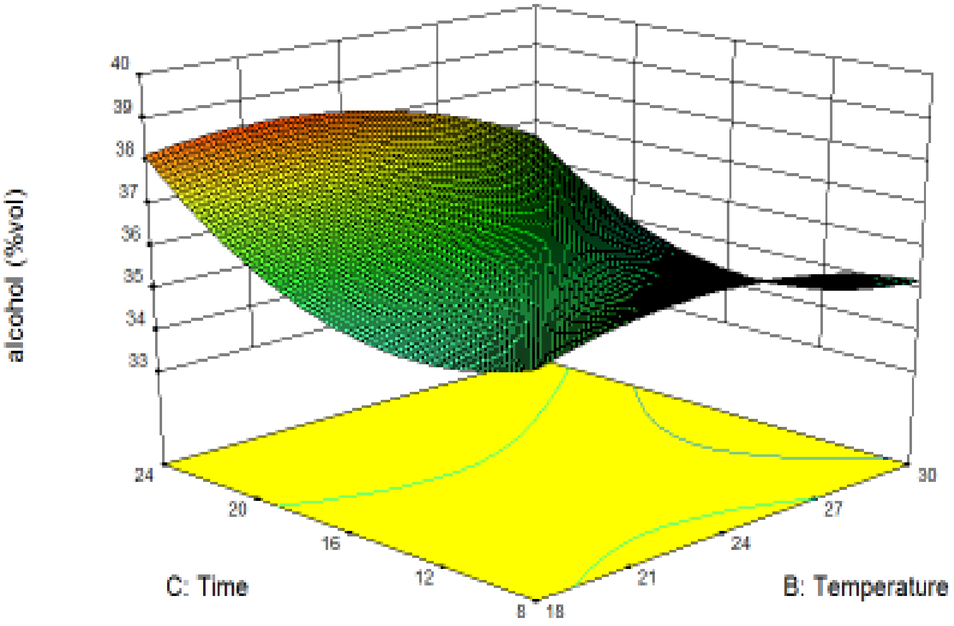
| Factor | Coefficient estimate | Sum of squares | df | Standard error | F-value | p-value |
|---|---|---|---|---|---|---|
| Model | | 31.27 | 9 | 3.47 | 7.020 | 0.0088 |
| A-Yeast | -0.39 | 1.20 | 1 | 0.25 | 1.200 | 2.430 |
| B-Temperature | -0.54 | 2.31 | 1 | 0.25 | 2.310 | 4.670 |
| C-Time | 0.98 | 7.61 | 1 | 0.25 | 7.610 | 15.37 |
| AB | 1.20 | 5.76 | 1 | 0.35 | 5.760 | 11.64 |
| AC | -0.52 | 1.10 | 1 | 0.35 | 1.100 | 2.230 |
| BC | -0.22 | 0.20 | 1 | 0.35 | 0.200 | 0.410 |
| A^2 | -0.42 | 0.74 | 1 | 0.34 | 0.740 | 1.500 |
| B^2 | -0.82 | 2.83 | 1 | 0.34 | 2.830 | 5.720 |
| C^2 | 1.55 | 10.18 | 1 | 0.34 | 10.18 | 20.57 |
| Residual | | 3.46 | 7 | 0.49 | | |
| Lack of fit | | 0.31 | 3 | 0.10 | 0.130 | 0.9359 |
| Pure error | | 3.15 | 4 | 0.79 | | |
| Cor total | | 34.74 | 16 | | | |
| SD | | 0.70 | | $R^2$ | 0.9003 | |
| Mean | | 35.89 | | $R^{Adj2}$ | 0.7720 | |
| C.V. % | | 1.96 | | Pred R-Squared | 0.7143 | |
| PRESS | | 9.93 | | Adeq Precision | 10.402 | |



(a)



(b)

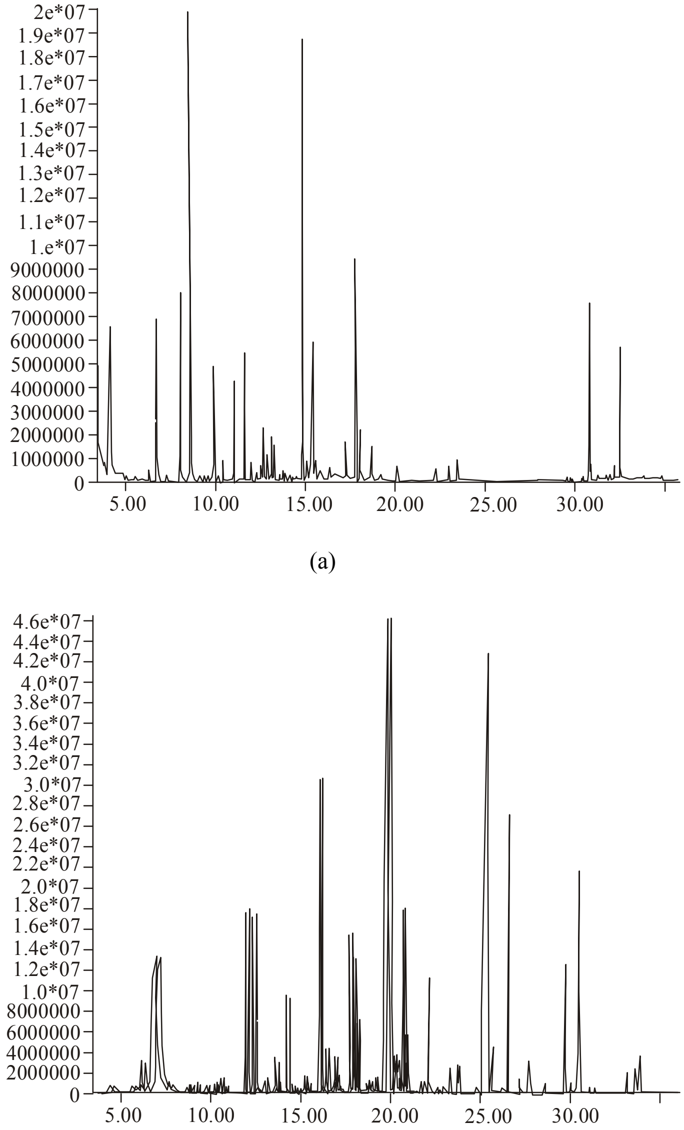


(c)

Fig. 5: Surface plots for flavor content of *jujube* brandy; (a): figure plot to show yeast strains and temperature; (b): figure plot to show yeast strains and time; (c): figure plot to show temperature and time

curvature. Arelatively flat line means in sensitive to change in that particular factor. The response (Y) was plotted against the deviation from the reference point by changing only one factor over its entire range while holding all other factors constant (Actual Factors: A-yeast = 2.02703, B-temperature = 24, C-time = 16, Fig. 4). The relationship between the responses and the experimental variables can be clarified graphically by plotting three-dimensional response surface plots (Fig. 5a to 5c). Fermentation temperature and time have great influence on the alcohol content compared with yeast strains (Gupta and Ako, 2005).

**Validation of the model:** The aim of optimization was to find out the conditions which give the maximum alcohol content of *jujube* brandy. The optimum brewing



(a)

(b)

Fig. 6: Total ion chromatogram of volatile components in *jujube* brandy; (a): normal brewing method; (b): optimized brewing method

conditions and the maximum alcohol content were obtained desirability function approach was single-yeast GH, fermentation temperature of 18°C, fermentation time of 24d and the maximum alcohol content of *jujube* brandy was 39.905%vol with a desirability value of 0.399. Triplicate duplicate tests were performed under the optimized conditions with the mean values of 38.70±0.02%vol, which was consistent with the expected value of 39.905%vol, demonstrating that the optimized conditions agree well with the real experiments.

**Quality of *jujube* brandy:** Under the optimum fermentation conditions, the concentration of alcohol, total acid and esters in the final product were 38.7%vol, 0.55 g/L (calculated by the content of acetic acid) and 2.35 g/L, respectively. The product had a typical characteristic of brandy. Harmful by-products of methanol were 0.034 g/100 mL.

**Flavor compounds of *jujube* brandy:** Flavor compound of *jujube* brandy with optimized and normal brewing method have been compared (Fig. 6). The GC-MS results demonstrated that there is a large difference between optimized and normal brewing method (Table 4). It determined that amount and content of flavor compounds in *jujube* brandy brewed by optimized process were higher than that of normal process, especially the esters. Such results indicated that the optimized process improved the mellow flavor of *jujube* brandy.

Table 4: Flavor compound of *jujube* brandy with optimized and normal brewing method

| Time/min | Flavor compounds | Mol.wt. | Optimized | Normol |
|---|---|---|---|---|
| Esters | | | | |
| 8.75 | Butanoic acid, ethyl ester | 116.084 | 2.857 | - |
| 9.03 | Butanoic acid, 2-methyl-, ethyl ester | 130.099 | 3.545 | 0.4230 |
| 9.31 | Butanoic acid, 3-methyl-, ethyl ester | 130.099 | 1.716 | 0.2350 |
| 10.31 | 1-Butanol, 3-methyl-, acetate | 130.099 | 3.185 | - |
| 10.54 | Pentanoic acid, ethyl ester | 130.099 | 2.918 | 0.6190 |
| 12.47 | Hexanoic acid, ethyl ester | 144.115 | 64.235 | 12.905 |
| 13.28 | 3-Hydroxymandelic acid, ethyl ester, di-TMS | 340.153 | - | 0.3230 |
| 14.31 | Heptanoic acid, ethyl ester | 158.131 | 26.597 | 2.9030 |
| 14.58 | Phthalic acid, ethyl tetradecyl ester | 390.277 | - | 0.1940 |
| 14.60 | Ethyl 2-hexenoate | 142.099 | 2.393 | 0.3390 |
| 15.33 | Octanoic acid, methyl ester | 158.131 | 1.038 | 6.2660 |
| 15.37 | 3-Heptenoic acid, ethyl ester, (E)- | 156.115 | 0.633 | - |
| 16.13 | Octanoic acid, ethyl ester | 172.146 | 158.427 | - |
| 16.51 | Isopentylhexanoate | 186.162 | 13.931 | - |
| 16.97 | 7-Octenoic acid, ethyl ester | 170.131 | 11.764 | 0.7250 |
| 17.07 | 3-Octenoic acid, ethyl ester | 170.131 | 6.014 | 0.2940 |
| 17.28 | Pentanoic acid, 4-methyl-, methyl ester | 130.099 | - | 0.2350 |
| 17.80 | Nonanoic acid, ethyl ester | 186.162 | 70.447 | 1.5910 |
| 18.68 | 3-Nonenoic acid, ethyl ester | 184.146 | 1.293 | - |
| 18.73 | Decanoic acid, ethyl ester | 200.178 | 2.457 | - |
| 18.86 | Decanoic acid, methyl ester | 186.162 | 2.576 | - |
| 18.87 | 10-Undecenoic acid, ethyl ester | 4574612 | 0.261877 | - |
| 18.90 | Decanoic acid, methyl ester | 186.162 | 4.045 | 0.2880 |
| 19.93 | Decanoic acid, ethyl ester | 200.178 | 903.618 | 26.004 |
| 20.20 | Octanoic acid, 3-methylbutyl ester | 214.193 | 12.457 | - |
| 20.36 | Ethyl trans-4-decenoate | 198.162 | 15.349 | 0.4390 |
| 20.53 | Butanedioic acid, diethyl ester | 174.089 | 6.63 | - |
| 20.73 | Benzoic acid, ethyl ester | 150.068 | 88.742 | 8.1310 |
| 20.86 | Ethyl 9-decenoate | 198.162 | 22.222 | 0.7600 |
| 21.57 | Decanoic acid, propyl ester | 214.193 | 1.93 | - |
| 22.03 | Undecanoic acid, ethyl ester | 214.193 | 52.626 | 0.4750 |
| 22.38 | n-Capric acid isobutyl ester | 228.209 | 4.244 | - |
| 22.66 | Ethyl trans-2-decenoate | 198.162 | 2.028 | - |
| 23.60 | Benzeneacetic acid, ethyl ester | 164.084 | 13.69 | - |
| 24.55 | Acetic acid, 2-phenylethyl ester | 164.084 | 0.93 | - |
| 24.66 | Benzoic acid, 2-hydroxy-, ethyl ester | 166.063 | 4.07 | - |
| 25.31 | Dodecanoic acid, ethyl ester | 228.209 | 926.025 | 18.382 |
| 25.61 | Pentadecanoic acid, 3-methylbutyl ester | 242.225 | 16.9 | - |
| 26.46 | Benzenepropanoic acid, ethyl ester | 178.099 | 145.549 | 2.2840 |
| 27.14 | 1-Butanol, 3-methyl-, benzoate | 192.115 | 2.973 | - |
| 27.52 | Ethyl tridecanoate | 242.225 | 7.009 | - |
| 28.51 | Ethyl 9-hexadecenoate | 282.256 | 3.257 | - |
| 28.85 | Methyl tetradecanoate | 242.225 | 0.849 | - |
| 28.91 | Benzenepropanoic acid, 2-methylpropyl ester | 206.131 | 0.904 | - |
| 29.62 | Tetradecanoic acid, ethyl ester | 256.24 | 55.654 | 1.6200 |
| 29.92 | Isoamyllaurate | 270.256 | 5.08 | - |
| 30.57 | (E)-9-Octadecenoic acid ethyl ester | 310.287 | 0.988 | 2.5400 |
| 30.94 | 3-Phenylpropionic acid, 3-methylbutyl ester | 220.146 | 2.389 | - |
| 31.22 | 2-Propenoic acid, 3-phenyl-, ethyl ester, (E)- | 176.084 | 1.292 | - |
| 31.32 | Pentadecanoic acid, ethyl ester | 270.256 | 0.954 | - |
| 33.03 | Hexadecanoic acid, ethyl ester | 284.272 | 11.083 | 0.326 |
| 33.50 | Ethyl 9-hexadecenoate | 282.256 | 17.363 | 0.883 |
| 33.75 | E-11-Hexadecenoic acid, ethyl ester | 282.256 | 25.158 | - |
| Alcohols | | | | |
| 9.87 | 1-Propanol, 2-methyl- | 74.073 | 2.491 | - |
| 10.50 | 1-Hexanol | 102.104 | - | 0.782 |
| 12.08 | 1-Octen-3-ol | 128.12 | - | 0.813 |
| 12.03 | 1-Butanol, 3-methyl- | 88.089 | 83.681 | |
| 12.17 | Heptanol | 116.12 | - | 0.266 |
| 13.77 | 1-Octanol | 130.136 | - | 0.188 |
| 13.96 | Fluoren-9-ol, 3,6-dimethoxy-9-(2-phenylethynyl)- | 342.126 | - | 0.421 |
| 15.28 | 3-Octanol | 130.136 | 4.08 | 4.080 |
| 20.05 | 1-Nonanol | 144.151 | 3.692 | - |
| 21.29 | 2-Tridecanol | 200.214 | 0.855 | - |
| 27.00 | Phenylethyl Alcohol | 122.073 | 6.717 | - |
| 28.86 | 1,2,3,4-Butanetetrol, [S-(R*,R*)]- | 122.058 | - | 0.565 |
| Acids | | | | |
| 12.38 | Acetic acid | 60.021 | - | 0.469 |
| 15.63 | Hexanoic acid, 2-methyl- | 130.099 | - | 0.577 |
| 18.16 | Hexanoic acid | 116.084 | - | 3.449 |
| 20.21 | Heptanoic acid | 130.099 | - | 1.428 |

Table 4: Continue

| | | | | |
|---|---|---|---|---|
| 23.09 | Octanoic Acid | 144.115 | - | 2.189 |
| 26.15 | Nonanoic acid | 158.131 | - | 0.494 |
| 26.55 | 2-Octenoic acid, (E)- | 142.099 | - | 0.250 |
| 28.12 | n-Decanoic acid | 172.146 | - | 12.018 |
| 29.59 | Undecanoic acid | 186.162 | - | 0.345 |
| 30.29 | Benzenecarboxylic acid | 122.037 | - | 1.089 |
| 30.83 | Dodecanoic acid | 200.178 | - | 8.232 |
| 34.09 | Z-11-Tetradecenoic acid | 226.193 | - | 0.303 |
| Aldehydes and ketones | | | | |
| 5.59 | 3,6-Bis-dimethylaminomethyl-2,7-dihydroxy-fluoren-9-one | 326.163 | - | 0.222 |
| 6.74 | Butanal, 3-methyl- | 86.073 | 11.148 | - |
| 8.61 | 3-Heptanone, 5-methyl- | 128.12 | - | 0.148 |
| 8.96 | 2-Butenal | 70.042 | 1.854 | - |
| 9.64 | Hexanal | 100.089 | 1.075 | 0.563 |
| 11.00 | 2-Nonanone | 142.136 | - | 0.298 |
| 13.61 | Octanal | 128.12 | 2.776 | - |
| 14.19 | Benz[e]azulene-3,8-dione, 5-[(acetyloxy)methyl] 3a,4,6a,7,9,10,10a,10b-octahydro-3a,10a-dihydroxy-2,10-dimethyl-, (3a.alpha.,6a.alpha.,10.beta.,10a.beta.,10b.beta.)-(+)- | 348.157 | - | 0.295 |
| 14.50 | 5-Hepten-2-one, 6-methyl- | 126.104 | 0.464 | 0.353 |
| 15.49 | Nonanal | 142.136 | 3.604 | - |
| 16.23 | 2-Tridecenal, (E)- | 196.183 | 4.29 | - |
| 16.84 | Furfural | 96.021 | 3.794 | - |
| 17.27 | Decanal | 156.151 | 2.175 | - |
| 18.01 | Benzaldehyde | 106.042 | 58.846 | 0.190 |
| 19.01 | 2-Undecanone | 170.167 | 0.88 | - |
| 19.04 | 2-Undecanone | 170.167 | 0.825 | - |
| 20.27 | Benzeneacetaldehyde | 120.058 | 23.358 | - |
| 23.21 | 2H-1-Benzopyran-2-one, 3,4-dihydro- | 148.052 | 13.651 | - |
| 23.70 | 2(3H)-Benzofuranone, 3-methyl- | 148.052 | 19.222 | - |
| 24.78 | 2-Buten-1-one, 1-(2,6,6-trimethyl-1,3-cyclohexadien-1-yl)-,(E)- | 190.136 | 1.437 | - |
| 25.51 | 5,9-Undecadien-2-one, 6,10-dimethyl-, (E)- | 194.167 | 4.953 | - |
| 27.99 | 1-Hexanone, 1-phenyl- | 176.12 | 0.748 | - |
| 28.28 | 2(1H)-Naphthalenone, octahydro-4a,7,7-trimethyl-, cis- | 194.167 | 0.687 | - |
| Hydrocarbons | | | | |
| 9.40 | Butane, 1,1-diethoxy-3-methyl- | 160.146 | 2.631 | 37.629 |
| 9.47 | 3,5-Diisopropoxy-1,1,1,7,7,7-hexamethyl-3,5-bis (trimethylsiloxy) tetrasiloxane | 546.217 | - | 0.253 |
| 12.72 | 3-Isopropoxy-1,1,1,7,7,7-hexamethyl-3,5,5 tris (trimethylsiloxy) tetrasiloxane | 576.21 | - | 0.441 |
| 13.10 | Styrene | 104.063 | 5.464 | - |
| 13.18 | 1H-Trindene, 2,3,4,5,6,7,8,9-octahydro-1,1,4,4,9,9-hexamethyl- | 282.235 | - | 2.343 |
| 13.44 | Decane, 3,7-dimethyl- | 170.203 | 1.239 | - |
| 13.49 | Dodecane, 2,6,11-trimethyl- | 212.25 | 1.326 | - |
| 14.36 | Cyclopentane, 1-ethyl-2-methyl-, cis- | 112.125 | 0.561 | - |
| 15.24 | Silane, [[4-[1,2-bis[(trimethylsilyl)oxy]ethyl]-1,2-phenylene]bis(oxy)]bis[trimethyl- | 458.216 | - | 0.673 |
| 15.89 | Bicyclo[4.2.0]octa-1,3,5-triene, 7-(2-propenyl)- | 144.094 | - | 0.929 |
| 16.38 | Benzene, 1,2,4,5-tetramethyl- | 134.11 | 1.181 | - |
| 17.32 | Benzene, 1-ethyl-2,3-dimethyl- | 134.11 | 0.785 | - |
| 21.64 | Benzene, (2,2-diethoxyethyl)- | 194.131 | 3.671 | - |
| 22.85 | Naphthalene | 128.063 | 4.465 | - |
| 26.00 | Naphthalene, 2-methyl- | 142.078 | 4.779 | - |
| 26.86 | Naphthalene, 1-methyl- | 142.078 | 1.41 | - |
| 27.61 | Benzeneacetaldehyde, .alpha.-ethylidene- | 146.073 | 13.618 | - |
| 29.17 | Naphthalene, 2,6-dimethyl- | 156.094 | 0.924 | - |
| 30.46 | Benzene, 1-isocyano-2-methyl- | 117.058 | - | 0.135 |
| 32.75 | Naphthalene, 1,6-dimethyl-4-(1-methylethyl)- | 198.141 | 0.627 | - |
| Others | | | | |
| 7.32 | Pyrrolidine | 71.073 | - | 0.473 |
| 7.44 | 2-Chloro-4-(4-methoxyphenyl)-6-(4-nitrophenyl)pyrimidine | 341.057 | - | 0.321 |
| 9.28 | 1,2,4-(4H)-Triazole, 3-(1-benzoylamino)ethyl-4-propyl- | 258.148 | - | 0.476 |
| 12.97 | N-[4-Methoxy-3-methoxycarbonyl)benzoyloxy]succininide | 307.069 | - | 1.169 |
| 13.84 | 1,2-Epoxy-3,4-dihydroxycyclohexano[a]pyrene, | 530.267 | - | 0.407 |
| 16.50 | trans-4-(2-(5-Nitro-2-furyl)vinyl)-2-quinolinamine | 281.08 | - | 0.165 |
| 16.61 | Naphthalene | 128.063 | - | 0.286 |
| 16.75 | Oxime-,methoxy-phenyl-_ | 151.063 | 6.244 | 0.453 |
| 16.88 | Acetamide | 59.037 | - | 0.079 |
| 21.08 | Levoglucosenone | 126.032 | - | 0.342 |
| 22.15 | Benzenepropanenitrile, .beta.-oxo- | 145.053 | 1.264 | 0.185 |
| 27.30 | .alpha.-Calacorene | 200.157 | 1.714 | |
| 29.90 | 1,4:3,6-Dianhydro-.alpha.-d-glucopyranose | 144.042 | - | 0.363 |
| 32.96 | 2-Furaldehyde dimethyl hydrazone | 138.079 | - | 0.321 |
| 33.75 | 1,4-Benzenediol, 2,3,5-trimethyl- | 152.084 | - | 0.314 |
| 34.46 | Ferrocene | 186.013 | - | 0.423 |

## CONCLUSION

In this present study, the brewing conditions of *jujube* brandy were optimized with a three factor three level Box-Behnken response surface design coupled with desirability function methodology. The results showed that, fermentation temperature and time had significant effect on the alcohol content of *jujube* brandy and a high correlated quadratic polynomial mathematical model was developed. The optimal conditions were determined to be: single-yeast GH, fermentation temperature of 18 °C, fermentation time of 24d. Under the optimal conditions, the experimental values (38.70±0.02%vol) agreed with the predicted values (39.905%vol). The optimized process improved the mellow flavor of *jujube* brandy, which has great practical values.

## ACKNOWLEDGMENT

This research was supported by the National Natural Science Foundation of China:

- The Research of Methanol and Fuel Oil Formation Mechanism and Control Measures in Traditional Chinese *Jujube* Brandy (Founding No.31171725).
- Study on the flavor character and its formation mechanism of Chinese date brandy (Founding No. 31371815).
- China Scholarship Council (201508130082).

## REFERENCES

Berradre, M., M. Mejias, J. Ferrer, C. Chandler, G. Paez, Z. Marmol, E. Ramones and V. Fernandez, 2009. Solid state fermentation of the wastes generated in the wine-making industry. Rev. Fac. Agron., 26(3): 398-422.

Chang, M., J. Lian, R. Liu, Q. Jin and X. Wang, 2014. Production of yellow wine from *Camellia oleifera* meal pretreated by mixed cultured solid-state fermentation. Int. J. Food Sci. Tech., 49(7): 1715-1721.

Claus, M.J. and K.A. Berglund, 2005. Fruit brandy production by batch column distillation with reflux. J. Food Process Eng., 28(1): 53-67.

Fan, W. and M.C. Qian, 2005. Headspace solid phase microextraction and gas chromatography-olfactometry dilution analysis of young and aged Chinese “Yanghe Daqu” liquors. J. Agr. Food Chem., 53(20): 7931-7938.

Fan, W. and M.C. Qian, 2006. Characterization of aroma compounds of Chinese ‘Wuliangye’ and ‘Jiannanchun’ liquors by aroma extraction dilution analysis. J. Agr. Food Chem., 54(7): 2695-2704.

Fan, W., H. Shen and Y. Xu, 2011. Quantification of volatile compounds in Chinese soy sauce aroma type liquor by stir bar sorptive extraction and gas chromatography-mass spectrometry. J. Sci. Food Agr., 97(1): 1187-1198.

Gupta, B.S. and J.E. Ako, 2005. Application of guar gum as a flocculant aid in food processing and potable water treatment. Eur. Food Res. Technol., 221(6): 746-751.

Huafeng, Y., Z. Xinan and C. Yong, 2003. Research on artificial aging of fresh wine with high strength electromagnetic field. Liquor Making, 3: 40-42.

Jackson, R.S., 2002. Wine Tasting: A Professional Handbook. Elsevier Academic Press, California.

Jijun, W., X. Gengsheng and L. Xueming, 2005. Research on new technology of fruit wine. Liquor Making, 32(2): 76.

Jiming, L. and H. Puchao, 2004. Analysis on flavor components ofwild wine. J. Fruit Sci., 21(1): 11-16.

Li, J.W., L.P. Fan, S.D. Ding and X.L. Ding, 2007. Nutritional composition of five cultivars of Chinese *jujube*. Food Chem., 103(2): 454-460.

Luo, T., W. Fan and Y. Xu, 2008. Characterization of volatile and semi-volatile compounds in Chinese rice wines by headspace solid phase microextraction followed by gas chromatography-mass spectrometry. J. I. Brewing, 114(2): 172-179.

Ni, M.L. and Q.X. Zeng, 2010. Study on the extraction technology of oat polyphenol by response surface methodology [J]. Sci. Technol. Food Ind., 4: 77.

Prakash Maran, J., V. Mekala and S. Manikandan, 2013. Modeling and optimization of ultrasound-assisted extraction of polysaccharide from *Cucurbita moschata*. Carbohyd. Polym., 92(2): 2018-2026.

Qianwen, L. and H. Zhengjun, 2000. Infrared artificial aging of Xueshanrhodiola wine. Liquor Making, l: 88-89.

Rapp, A., 1998. Volatile flavour of wine: Correlation between instrumental analysis and sensory perception. Nahrung, 42(6): 351-363.

Zhang, R., Q. Wu and Y. Xu, 2013. Aroma characteristics of Moutai-flavour liquor produced with *Bacillus licheniformis* by solid-state fermentation. Lett. Appl. Microbiol., 57(1): 11-18.

Zheng, X.W., M.R. Tabrizi, M.J.R. Nout and B.Z. Han, 2011. Daqu— A traditional Chinese liquor fermentation starter. J. I. Brewing, 117(1): 82-90.

Zhu, S., X. Lu, K. Ji, K. Guo, Y. Li, C. Wu and G. Xu, 2007. Characterization of flavor compounds in Chinese liquor Moutai by comprehensive two-dimensional gas chromatography/time-of-flight mass spectrometry. Anal. Chim. Acta, 597(2): 340-348.