Food Chemistry 150 (2014) 94–98

Contents lists available at ScienceDirect

Food Chemistry

journal homepage: www.elsevier.com/locate/foodchem

Analytical Methods

# Modelling and optimisation of enzymatic extrusion pretreatment of broken rice for rice wine manufacture

Hongyan Li [a,b], Benxi Wei [a,b], Chunsen Wu [b], Bao Zhang [a,b], Xueming Xu [b,c], Zhengyu Jin [a,b,c,*], Yaoqi Tian [a,b,c,*]

[a] The State Key Laboratory of Food Science and Technology, Jiangnan University, 1800 Lihu Road, Wuxi 214122, China
[b] School of Food Science and Technology, Jiangnan University, 1800 Lihu Road, Wuxi 214122, China
[c] Synergetic Innovation Center of Food Safety and Nutrition, Jiangnan University, Wuxi 214122, China

ARTICLE INFO

*Article history:*
Received 28 March 2013
Received in revised form 11 October 2013
Accepted 24 October 2013
Available online 4 November 2013

*Keywords:*
Alcoholic degree
Broken rice
Enzymatic extrusion
Response surface methodology
Rice wine

ABSTRACT

The manufacture of Chinese rice wine involves an uneconomical, time-consuming, and environmentally unfriendly pretreatment process. In this study, the enzymatic extrusion of broken rice was applied to the brewing of rice wine. The response surface methodology was used to study the effects of the barrel temperature (BT), moisture content (MC), and amylase concentration (AC) on the alcohol yield. A second-order polynomial model had a good fit to the experimental data and the coefficient of determination ($R^2$) was 0.9879. According to the model, the optimal parameters required to obtain the highest alcoholic degree of 17.94% were: BT = 100.14 °C, MC = 43%, and AC = 1.45‰. Under these optimal conditions, the alcoholic degree actually reached 18.3%, which was close to the value predicted by the model. Enzymatic extrusion improved the yeast growth and alcohol yield during the fermentation process. The fermentation recovery and efficiency of processed rice wine were 38.07% and 94.66%, respectively.



## 1. Introduction

Chinese rice wine is produced by simultaneous saccharification and fermentation (SSF), and is one of the three most ancient types of wine in the world (Shen et al., 2012). In general, the production of Chinese rice wine is divided into two major stages: raw rice pretreatment and alcohol fermentation. However, the traditional pretreatment process is water-, energy-, and labour-consuming (Fig. 1). At present, the large amounts of wastewater generated cause severe environmental problems for the industry. Enzymatic extrusion is a promising technology for the processing of starch-based food, where cereal starch or flour can be hydrolysed into a variety of dextrins (Baks, Kappen, Janssen, & Boom, 2008). A previous study showed that a high yield of glucose syrup was obtained using enzymatic extrusion pretreatment (Govindasamy, Campanella, & Oates, 1995). Broken rice is a by-product of rice processing and recent studies have shown that broken rice is an efficient material for ethanol fermentation (Gohel & Duan, 2012b). Therefore, the enzymatic extrusion of broken rice was applied to the brewing of Chinese rice wine in the present study. The aim of this investigation was to optimise the extrusion parameters (barrel temperature (BT), moisture content (MC), and amylase concentration (AC)) to obtain the maximum alcohol yield. The SSF processes were also monitored and compared during novel and traditional rice wine processing.

## 2. Materials and methods

### 2.1. Materials

Broken rice (*Nuo-535*, China) was purchased locally (composition: protein = 10.2%, fat = 2.1%, and starch = 80.9%). The thermostable α-amylase (Termamyl 120L, Novozymes, Denmark) derived from *Bacillus licheniformis* was used. The enzyme had an optimum pH of 6–8 and an activity of 120 kilo novo units α-amylase (KNU)/g (1 KNU is defined as the amount of enzyme that hydrolyses 5.26 g starch/h). Wheat Qu and yeast were provided by Zhejiang Nverhong Shaoxing Wine Co. Ltd.

### 2.2. Enzymatic extrusion liquefaction

Enzymatic extrusion was performed according to our previous method using a laboratory scale twin-screw extruder (TSE 24 MC, Thermo Scientific, USA) with a length to diameter ratio of 40:1. The screw speed of the extruder was maintained at 100 rpm. The feed rate was kept stable at 1.5 kg/h (Li et al., 2012).

### 2.3. Chinese rice wine fermentation

The extrudate (200 g, dry basis (db)) was supplemented with 400 mL water, 0.2 g yeast, and 32 g wheat Qu in a flask (1 L), and

* Corresponding authors. Address: The State Key Laboratory of Food Science and Technology, Jiangnan University, 1800 Lihu Road, Wuxi 214122, China. Tel./fax: +86 510 85913299 (Z. Jin). Tel./fax: +86 510 85328571 (Y. Tian).
*E-mail addresses:* jinlab2008@yahoo.com (Z. Jin), yqtian@jiangnan.edu.cn (Y. Tian).

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http://dx.doi.org/10.1016/j.foodchem.2013.10.146

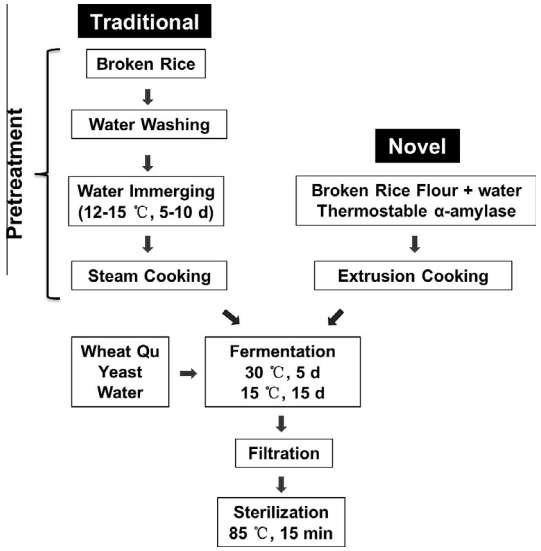


**Fig. 1.** The manufacture processes of enzymatic extrusion- and tradition-pretreated Chinese rice wine.

incubated for 20 days (30 °C, 5 days; 15 °C, 15 days). For traditional rice wine fermentation, the broken rice (200 g, db) was steeped in water for 15 days at 10 °C, steam-cooked for 30 min, and incubated in the same manner (Fig. 1). After fermentation, the fermentation broth was centrifuged at 5000*g* for 20 min and the supernatant was sterilized at 85 °C for 15 min. Finally, the rice wine was stored in a ceramic container. All of the experiments were performed in triplicate.

### 2.4. Determination of the alcoholic degree and yeast cell counts

Wine samples were taken at specific intervals. The alcoholic degree (v/v at 20 °C) and yeast cell counts were measured according to the procedures described by Gohel and Duan (2012a).

### 2.5. Rice wine recovery and fermentation efficiency

The alcohol recovery (%) and fermentation efficiency (%) were calculated as follows:

$$\text{Alcohol recovery}\ (\%) = \frac{\text{Total slurry (Kg)} \times \text{alcohol (\% v/v at 20 °C)} \times \theta}{\text{Total grain (Kg)}}, \quad (1)$$

$$\text{Fermentation efficiency}\ (\%) = \frac{\text{Total slurry (Kg)} \times \text{alcohol (\% v/v at 20 °C)} \times \theta \times 100}{\text{Total grain (Kg)} \times \text{\% starch} \times 0.5679}, \quad (2)$$

where $\theta$ is the mass concentration conversion coefficient and 0.5679 is the theoretical alcohol yield produced by 1 g of starch.

### 2.6. Experimental design and statistical analysis

A central composite design was used to optimise the factors BT, MC and AC to maximise ethanol yield of Chinese rice wine (Table 1). A second-order polynomial equation was used to describe the main effects in terms of linear, quadratic, and interactions:

$$Y = b_0 + \sum_{i=1}^{3} b_i X_i + \sum_{i=1}^{3} b_i X_i^2 + \sum_{i=1}^{2} \sum_{j=i+1}^{3} b_{ij} X_i X_j, \quad (3)$$

where $Y$ is the alcoholic degree (v/v at 20 °C), $X_i$ represents the independent variables, and $b_i$ represents the regression coefficients. The goodness of the fit of the second-order equation was evaluated based on the coefficients of determination $R^2$ and $R_{adj}^2$. Design Expert 8.0.5 (Stat-Ease Inc., Minneapolis, USA) was used to perform analysis of variance (ANOVA) and to generate graphical representations of the data.

**Table 1**
Experimental design and response of the three independent variables.

| Run | BT (°C) $X_1$ | MC (%) $X_2$ | AC (%) $X_3$ | Alcoholic degree (% v/v 20 °C), $Y$ | | |
|---|---|---|---|---|---|---|
| | | | | Experimental[a] | Predicted | Residual |
| 1 | 95 (−1) | 33 (−1) | 0 (−1) | 8.5 ± 0.23 | 8.6 | −0.1 |
| 2 | 105 (1) | 33 (−1) | 0 (−1) | 9.6 ± 0.08 | 9.6 | 0.0 |
| 3 | 95 (−1) | 43 (1) | 0 (−1) | 9.1 ± 0.03 | 9.1 | 0.0 |
| 4 | 105 (1) | 43 (1) | 0 (−1) | 9.8 ± 0.02 | 9.8 | 0.0 |
| 5 | 95 (−1) | 33 (−1) | 1.8 (1) | 14.2 ± 0.07 | 14.2 | 0.0 |
| 6 | 105 (1) | 33 (−1) | 1.8 (1) | 14.5 ± 0.05 | 14.4 | 0.1 |
| 7 | 95 (−1) | 43 (1) | 1.8 (1) | 16.2 ± 0.00 | 16.2 | 0.0 |
| 8 | 105 (1) | 43 (1) | 1.8 (1) | 16.3 ± 0.12 | 16.2 | 0.1 |
| 9 | 95 (−1) | 38 (0) | 0.9 (0) | 15 ± 0.11 | 14.9 | 0.1 |
| 10 | 105 (1) | 38 (0) | 0.9 (0) | 15.2 ± 0.03 | 15.4 | −0.2 |
| 11 | 100 (0) | 33 (−1) | 0.9 (0) | 15.4 ± 0.13 | 15.5 | −0.1 |
| 12 | 100 (0) | 43 (1) | 0.9 (0) | 16.6 ± 0.11 | 16.6 | 0.0 |
| 13 | 100 (0) | 38 (0) | 0 (−1) | 10.5 ± 0.08 | 10.4 | 0.1 |
| 14 | 100 (0) | 38 (0) | 1.8 (1) | 16.2 ± 0.08 | 16.4 | −0.2 |
| 15 | 100 (0) | 38 (0) | 0.9 (0) | 16.2 ± 0.02 | 16.1 | 0.1 |
| 16 | 100 (0) | 38 (0) | 0.9 (0) | 16.2 ± 0.11 | 16.1 | 0.1 |
| 17 | 100 (0) | 38 (0) | 0.9 (0) | 16 ± 0.28 | 16.1 | −0.1 |
| 18 | 100 (0) | 38 (0) | 0.9 (0) | 16.3 ± 0.31 | 16.1 | 0.2 |
| 19 | 100 (0) | 38 (0) | 0.9 (0) | 16 ± 0.08 | 16.1 | −0.1 |
| 20 | 100 (0) | 38 (0) | 0.9 (0) | 16.3 ± 0.24 | 16.1 | 0.2 |

[a] Data are expressed as means of three replicates (mean value ± standard deviation).

## 3. Results and discussion

### 3.1. Anova

Table 1 shows the design matrix for the factors in the experimental runs. ANOVA (Table 2) was used to check the adequacy of the proposed model and to identify the significant factors. The second-order polynomial model is shown in Eq. (4).

$$Y = 16.14 + 0.24X_1 + 0.58X_2 + 2.99X_3 - 0.17X_1X_3 + 0.38X_2X_3 - 1.01X_1^2 - 2.76X_3^2 \quad (4)$$

The *F*-value of 1.23 for the lack of fit (Table 2) suggests that it was not significant relative to the pure error, which indicated that the model correlated well with the experimental values. $R^2$ was used as a measure of the goodness of fit of the model and it was 0.9879. The coefficient of variation (1.02%) showed that the experiments were precise and reliable. The adequate precision ratio of 78.72 showed that the signal was adequate, which demonstrated that the model could be used to navigate the design space.

### 3.2. Adequacy of the model

The normality of the data, which was checked using a normal plot of the residuals and the difference between the observed values and the values predicted from the regression, showed that the experimental points were normally distributed around a straight line, so the assumption of normality was satisfied. The plot of the residuals versus the predicted values showed that the residuals were scattered randomly around zero with no outliers or unexpected errors, thereby indicating that all of the values were within the accepted range required to validate the model (plot not shown).