

# RSM-Modeling and Optimization of High Titer Functional Xylo-Oligosaccharides Production by Edible Gluconic Acid Catalysis

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## Research Article

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# Abstract

Xylo-oligosaccharides as a functional prebiotic have great value in food, feed fields. Previous studies have shown that organic acids catalyze the hydrolysis of xylan-rich sources for the production of xylo-oligosaccharides. In this study, gluconic acid of edible aldonic acid, generated xylo-oligosaccharides via hydrolysis of xylan from corncob. In order to maximize the efficiency of xylo-oligosaccharide production, a model was designed and optimal conditions were determined by Box-Behnken design-based response surface methodology. The developed process resulted in a maximum xylo-oligosaccharides yield of 57.73% using 4.6% gluconic acid at 167°C for 28 min, which was similar to the predicted value and fitted models of xylo-oligosaccharides production. The results showed that the reaction temperature was crucial to xylo-oligosaccharides production, and by-product yields (xylose and furfural) could be effectively controlled by both reaction temperature and time. In addition, 44.87 g/L XOS was achieved by decreasing the solid-liquid ratio. Overall, the described process may be a preferred option for future high concentration xylo-oligosaccharides production.

## 1. Introduction

Xylo-oligosaccharides (XOS), which contain two-to-seven xylose molecules with  $\beta$ -(1,4) linkages, are low molecular weight carbohydrate oligomers; non-cariogenic and have a low calorific value (Han et al. 2020). XOS, as non-digestible oligosaccharides (NDOs), are known to benefit the host by selectively stimulating the growth or activity of bacteria in the colon (Dong et al. 2020; Zhang et al. 2020; Wang et al. 2021). Appropriate intake of XOS can selectively increase the number of bifidobacteria in the intestinal tract without causing excessive proliferation of other bacteria, so as to prevent antibiotic related diarrhea, constipation, and senile diseases (Cao et al. 2020). In addition to relieving constipation of pregnant women without side effects, taking XOS can also significantly improve the gastrointestinal digestion and absorption function of infants (Qian et al. 2020; Isci et al. 2021). Hence, XOS are considered prebiotics, which are important ingredients of functional pharmaceutical and food fields (Zhang et al. 2019). Clinical trials have confirmed that XOS as nutraceuticals can improve the biological availability of calcium, lower the cholesterol level, exhibit antioxidant and anti-inflammatory properties, immunomodulatory, antidiabetic and anti-cancer activities (Samanta et al. 2015; Guo et al. 2020). Additionally, XOS do not exhibit toxic or other negative effects toward human health, and research has shown that in healthy men and elderly subjects it is strongly bifidogenic (Zhou and Xu 2019a). The demand for XOS is rapidly increasing in enlarged health conception and antibiotic-free era, these compounds have an important place in the global market (Hao et al. 2020; Zhao et al. 2020). Considering the tremendous health benefits XOS offer, further research is vital.

Current studies describe the production of XOS from xylan or xylan-rich sources either by chemical methods (acid and alkali), auto-hydrolysis, microwave irradiation, direct enzymatic hydrolysis of a susceptible substrate or a combination of chemical and enzymatic treatments (Chen et al. 2018; Lai et al. 2019). Among these, acidic hydrolysis of xylan is an effective and practical approach. In order to obtain high purity XOS, it is generated in two stages: alkaline extraction of xylan from lignocellulosic biomass

followed by enzymatic or acid hydrolysis of xylan(Zhang et al. 2018). Compared with enzymatic hydrolysis, the fragmentation of xylan by controlled acid hydrolysis is more easily and effectively accomplished, which also results in desired degree of polymerization (DP2-6)(Immerzeel et al. 2014). Thus, acidic hydrolysis is more practical for XOS preparation. Until now only acids have been used in XOS production, including mineral and organic acids(Zhang et al. 2014). Applications of mineral acids lead to larger inorganic waste stream and generation of larger amounts of byproducts such as xylose and furfural(Zhang et al. 2018; Wu et al. 2020). Compared with mineral acids, organic acids have some desirable characteristics(Zhang et al. 2017a), including low equipment corrosion, less degradation byproducts and high XOS yield(Zhou et al. 2019). Various organic acids, such as xylonic acid, acetic acid, and oxalic acid, have been developed to directly hydrolyze xylan to XOS products(Xin et al. 2019). Lin et al. used three kinds of material hydrolyzed by oxalic acid to produce XOS in 39.31%, 27.29%, and 30.32% yield. Zhou et al. found that to the hydrolysis sugarcane bagasse using xylonic acid can produce XOS in 44.5% yield.

Recently, it has been found that gluconic acid (GA), a green and edible organic acid(Zhou and Xu 2019b), can release H<sup>+</sup> to efficiently pre-hydrolyze corncob, directly producing XOS in 54.3% yield. However, the methods that employ organic acids assist in depolymerization of the hemicellulose present in lignocellulosic biomass turning it into XOS, producing low concentration(Chen et al. 2019; Wen et al. 2020). Moreover, liquid phase from acidic hydrolysis of lignocellulosic biomass contains not only xylan-degraded by-products but also cellulose and lignin such as formic acid, acetic acid HMF(Jiang et al. 2013), phenolic compounds and crude proteins(Neto et al. 2020). Hence, direct production of XOS from biomass is not feasible(Faryar et al. 2015; Zhang et al. 2017b). In light of the advantages of organic acid pre-hydrolysis of XOS production over enzymatic or chemical process, and considering the target of high concentration XOS, the present work studies its production conditions for GA-assisted hydrolysis of alkaline soluble xylan from corncob.

## **2. Materials And Methods**

### **2.1 raw materials**

Xylan extracted from corncob was obtained from Jiangsu province in China. The main components were determined according to National Renewable Energy Laboratory and contained 69.15% xylan, 2.98% glucan, and 10.36% arabinan. D-Gluconic acid solution was purchased from Shanghai Aladdin Biochemical Technology Co. Ltd (Shanghai, China).

### **2.2 Pre-experimental design**

The experiments were designed based on the principle of Box-Behnken Center combination test design. In order to optimize XOS yield, three factors were used as independent variables: temperature 130-170°C, retention time 5-75 min, and GA concentration 2.5%-20% (w/w). The independent variables and dependent variable values (XOS yeild) are listed in Table 1. According to the Box-Behnken design matrix, 18 sets of experiments were designed and conducted.

1 g xylan and 10 mL GA with different concentrations were mixed in a digestion tube and stirred. Then, the tube was placed into the multifunctional intelligent digestion instrument (GL-16, Greencarey Shandong), which was preheated to a set temperature and set to a specific time. When the reaction was completed, the digestion tube was immediately removed and allowed to cool naturally to room temperature. The solid and liquid were collected and separated by centrifugation. XOS content in the liquid was analyzed.

Table 1  
18 sets of experimental conditions conducted by Box-Behnken Center design, and corresponding XOS yields

Variable			Response
Reaction temperature (°C)	Retention time (min)	GA concentration (%)	XOS yield (%)
130	60	5	17.3
130	30	20	27.6
130	60	20	43.0
130	30	10	15.4
130	30	5	7.6
150	30	20	43.6
170	20	2.5	46.1
150	45	5	42.5
150	60	5	42.8
150	15	10	31.8
150	30	10	47.0
130	60	10	31.4
150	10	20	29.4
170	30	2.5	52.6
170	40	2.5	47.1
170	25	5	47.4
170	10	10	48.0
170	15	10	52.3

## 2.3 Methods of analysis

The content of xylose and furfural in the hydrolysate was analyzed using high performance liquid chromatograph (HPLC) (Agilent 1260, USA) which has an Aminex column (HPX-87H, Thermo, Shanghai, China). The mobile phase of HPLC was 0.005 M sulfuric acid at a flow rate of 0.6 mL/min and kept working at 50°C. XOS produced in pretreatment including xylobiose (X2), xylotriose (X3), xyloetraose (X4), xylopentaose (X5), and xylohexaose (X6) were analyzed by a high performance anion exchange chromatograph (HPAEC) (Dionex ICS-3000, USA) which was equipped with a CarboPac™ PA200 column (Xu et al. 2013). The mobile phase of HPAEC-PAD was 0.1 M sodium hydroxide and 0.5 M sodium acetate, and the flow rate was set at constant 0.3 mL/min.

The yields of furfural, xylose and XOS were calculated as following formula:

$$\text{Furfural yeild (\%)} = \frac{\text{The mass of furfural in hydrolyates (g)}}{\text{The mass of xylan weight (g)}} \times 100\%$$
$$\text{Xylose yeild (\%)} = \frac{\text{The mass of xylose in hydrolyates (g)}}{\text{The mass of xylan weight (g)}} \times 100\%$$
$$\text{XOS yeild (\%)} = \frac{\text{The mass of XOS in hydrolyates (g)}}{\text{The mass of xylan weight (g)}} \times 100\%$$

## 2.4 Statistical analysis

Use Design Expert (Version. 8), a statistical software, to perform regression analysis on experimental data and generate response surface plots (Xue et al. 2016). One-way analysis of variance (ANOVA) and Duncan's multiple range test ( $P < 0.05$ ) were used to determine the statistical significance of the data. The correspondence between independent variables and response values was calculated by the following equation:

$$Y = A_0 + \sum A_i X_i + \sum A_{ii} X_i^2 + \sum A_{ij} X_i X_j$$

where Y represents the predicted XOS yield,  $A_0$  is the constant term, X is the independent variables,  $A_i$  is the coefficient of the linear parameter,  $A_{ii}$  is the coefficient of the quadratic parameter and  $A_{ij}$  is the coefficient of the interaction parameter.

## 3. Results And Discussion

### 3.1 Production of XOS from xylan hydrolyzed by gluconic acid

Acid concentration, retention time and reaction temperature are important factors affecting the degradation of xylan, as well as the selectivity of XOS production. Therefore, these three factors needed

to be optimized by response surface methodology (RSM) to obtain the highest XOS yield. RSM is an effective statistical and analytical method, which requires only a minimum number of experiments to determine a more complete mathematical model with optimal conditions, and can be used to maximize the XOS yield. In this work, 1 g xylan was hydrolyzed with 2.5-20% GA at 130-170°C for 10-75 min (Table 1). During acidic hydrolysis, xylan was first degraded into saccharides with relatively high DP, which were further hydrolyzed into XOS, xylose and furfural. Thus, after hydrolysis of xylan by GA, these products and by-products were simultaneously analyzed in order to evaluate the effects of different conditions (Fig. 1a-c). The highest XOS yield was 52.6% when xylan was hydrolyzed with 2.5% GA at 170°C for 30 min.

As shown in Fig. 1a, XOS yield was only 15.4% under this condition: 130°C, 10% GA, 30 min, with the yield rapidly increasing to 46.9% at 150°C with 10% acid concentration after 30 min. Therefore, high temperature could accelerate the degradation of xylan to XOS under the same conditions. As known in Fig. 1, XOS with lower DP, such as X2 and X3, have higher XOS content at higher temperature, which may be caused by XOS degradation with higher DP into XOS with low DP at high temperature. Also, Fig. 1 presents that with increasing reaction temperature, retention time and acid concentration, XOS was further degraded into xylose and furfural. Just as the furfural content was 0.25g/L and xylose could reach 10.23g/L under the condition of 10% GA at 170°C for 20 min, which was much higher than the concentration of furfural and xylose obtained at 150°C with 10% GA for 15 min. Moreover, under constant temperature, high acid concentration and long retention time promoted high xylose and furfural yield. Fig. 1b shows that the concentration of xylose obtained with 20% GA for 20 min is 11.7 g/L, while that with 10% acid concentration was only 1.8 g/L for 15 min. Hence, xylan could be hydrolyzed efficiently to prepare XOS at high temperature, but xylose and furfural were also obtained. Thus, a kinetic study was conducted to gain greater insight into its degradation trend. Fig. 2 shows the degradation trend of xylan with 5% GA at 170 °C. In the first 10 min, the distribution of X2 to X6 was relatively average, which may be caused by the random action of GA on  $\beta$ -1,4 glycosidic bond resulting in cleavage. As the time increased, X5 and X6 decreased, while X2 and X3 gradually increased, which was due to the continuous depolymerization of X5 and X6 with high DP to form XOS with lower DP. In addition, X5 and X6 content in all hydrolysates were lower, while the contents of X2 and X3 with low polymerization degree were higher, which was also preferred in food application.

The trend of by-products over time is shown in Fig. 2. Prolongation of retention time, produces xylose and furfural reaching a concentration of 1.35 g/L and 16.42 g/L, respectively. The growth trend of by-products slowed down after 20 min of retention time, which decreased XOS yield after 20 min. Therefore, the acid hydrolysis conditions could be controlled both XOS and by-product yield. However, the design of optimal conditions was based on the reasonable model, which was verified.

## **3.2 Fitting model of XOS produced by hydrolysis of xylan**

As listed in Table 1, the acid concentration, retention time and reaction temperature are important factors affecting the production of XOS. When temperature was too high, the glycosidic bonds in XOS are further broken and degraded to xylose and furfural, while at too low temperature the reaction would take an

undesirable length of time, which was not economically viable. Therefore, a suitable model was required to determine optimal conditions. On the basis of the fit summary reports generated by the statistical analysis software, the response surface based on Box-Behnken design is an effective method that can effectively establish the mathematical model of acid hydrolysis and optimize the conditions. The regression equation fitted by Design-Expert 11.0 is as follows:

$$Y = -1148.35995 + 13.10173P_1 + 4.95866P_2 + 15.03257P_3 - 0.025442P_1P_2 - 0.084281P_1P_3 - 0.004338P_2P_3 - 0.035928P_1^2 - 0.012274P_2^2 - 0.091092P_3^2$$

where  $P_1$ ,  $P_2$ , and  $P_3$  are the reaction temperature, retention time, and concentration of GA, respectively. The model with the P-value of 0.0001 was shown by ANOVA analysis, which indicates that the fitting model was in good agreement with the actual experiment.  $R^2$ , the decisive coefficient, of 0.9598 indicates that the regression model was in good agreement with the model as well. Also, the value of adeq precision was 15.7234, hence the fitting model was reasonable. The P-value of  $P_1$ ,  $P_2$  and  $P_3$  were <0.0001, 0.0084 and 0.2006, respectively. P-value could reflect the order of importance of the factors affecting the hydrolysis of xylan by GA, which should be ranked as follows: reaction temperature > retention time > GA concentration, and the interaction of retention time and reaction temperature had the most important effect on the yield of XOS. Moreover, the 3D response surface generated by Design Expert 8 is shown in Fig. 3. According to Fig. 3a-c, the red area represents better conditions, in which the optimal conditions were 4.6% GA, 28 min and 167 °C. Under these conditions, the contents of X2, X3, X4, X5, and X6 were 11.59 g/L, 10.57 g/L, 9.52 g/L, 4.61 g/L, and 3.62 g/L, respectively, and the yield of XOS reached 57.7%. The predicted yield of XOS was 54.3%, which was comparable to the actual XOS yield, which also verifies that the model could well predict XOS yield. After fine-tuning the established model, the kinetic profile could be studied.

### 3.3 Improvement of XOS content by increasing solid-liquid ratio

High concentration XOS are easier to be collected and purified in industry, and the highest yield of XOS can be optimized based on response surface methodology. Therefore, the solid-liquid ratio was optimized to obtain higher concentration of XOS. Fig. 4 shows the composition distribution of different solid-liquid ratios under optimal conditions. The solid-liquid ratio of 1:7.5 effectively increased XOS concentration to 44.9 g/L, whereas the ratio of 1:7.5 improved the yield of XOS for subsequent industrial production.

However, when the solid-liquid ratio was 1:5, XOS concentration was only 38.42 g/L, which did not increase but decreased. This is due to the gluconic acid solution cannot fully mix with xylan, resulting in

incomplete hydrolyzation when coking and adhering to the inner wall of digestion tube at high temperature. Furthermore, when the ratio of solid to liquid was 1:10, the concentration of XOS decreased because XOS were diluted due to excessive liquid added. Therefore, for industrial production, solid-liquid ratio of 1:7.5 is recommended for pretreatment to obtain high concentration of XOS. Additionally, XOS content reached 5%, which could reduce the cost of subsequent purification. Overall, it was expected to produce high concentrations of XOS. 44.9 g/L XOS could be produced by GA under optimized conditions.

## **4. Conclusion**

As a functional prebiotic, XOS can selectively proliferate bifidobacteria in human intestinal tract to improve human intestinal condition. Additionally, XOS are also beneficial to human health in other aspects. Also, GA as an edible acid can be left unseparated during the subsequent refining of XOS. This work proposes an feasible and environmental- friendly method that can effectively degrade xylan to XOS, and RSM is employed to define the point wherein maximum production of XOS, which is achieved and proved experimentally. All results indicate that the yield of XOS depends on both acid concentration, time, and temperature, where 44.9 g/L XOS at a yield of 48.67% is produced with 4.6% GA at 167°C for 28 min and a solid-liquid ratio of 1:7.5. Therefore, GA pre-hydrolysis method provides guidance for solving future prospective of XOS, which depend on its economic scale-up production.

## **Declarations**

### **Declaration of competing interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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### **Ethics approval and consent to participate**

Not applicable

### **Consent for publication**

The authors have obtained consent of all co-authors.

### **Author's contributions**

YJG and XZ developed the idea for the study. YJG and JMG performed the research. YJG conducted the data analysis and prepared the manuscript. YX and XZ helped to revise the manuscript. All authors read

and approved the final manuscript.

### Availability of data and materials

No availability of data and materials.

### Funding

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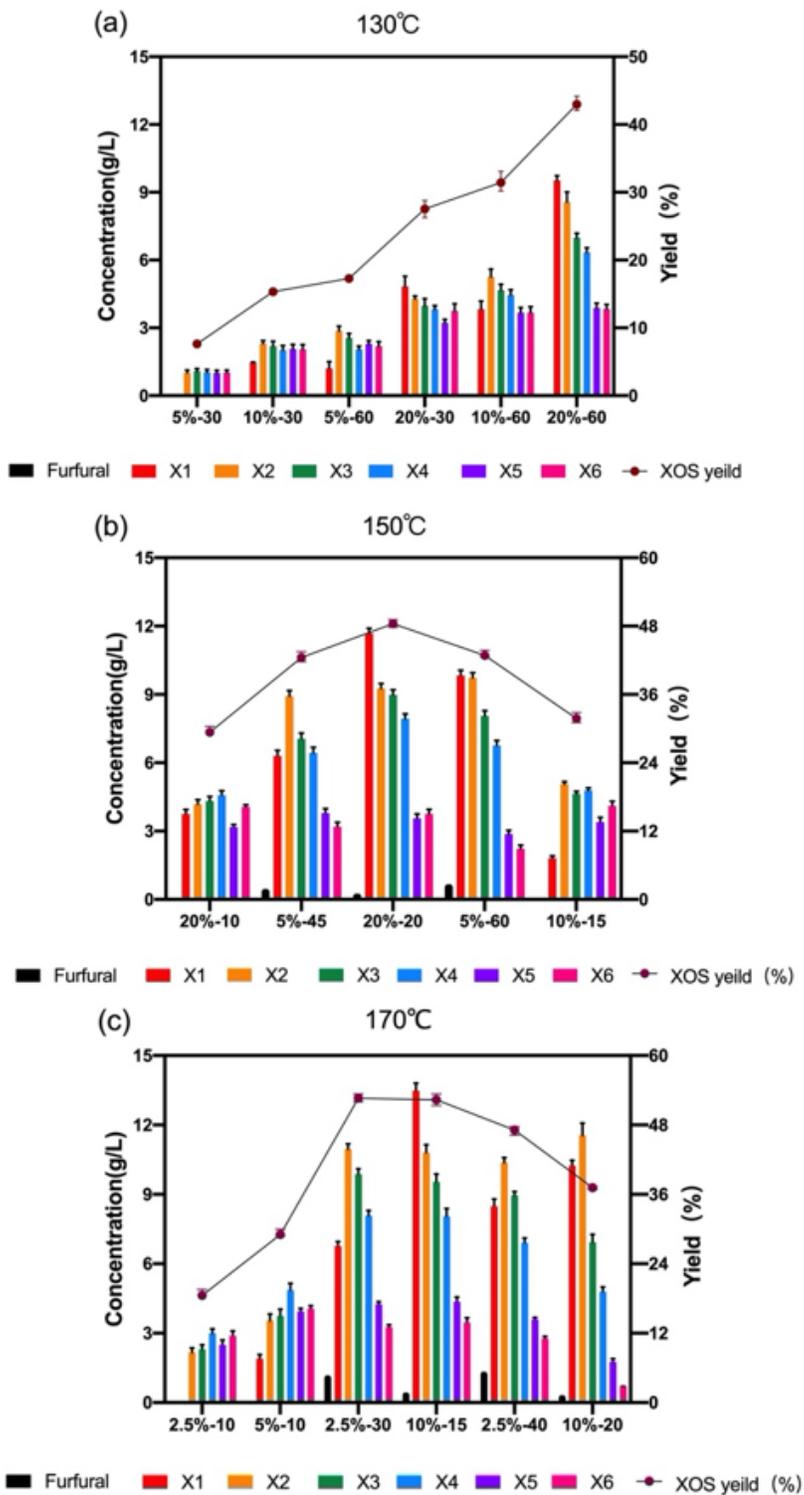
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## Figures



**Figure 1**

Concentration and yield of each degradation product in the hydrolysis solution under different conditions: (a) 130 °C; (b) 150 °C; and (c) 170 °C

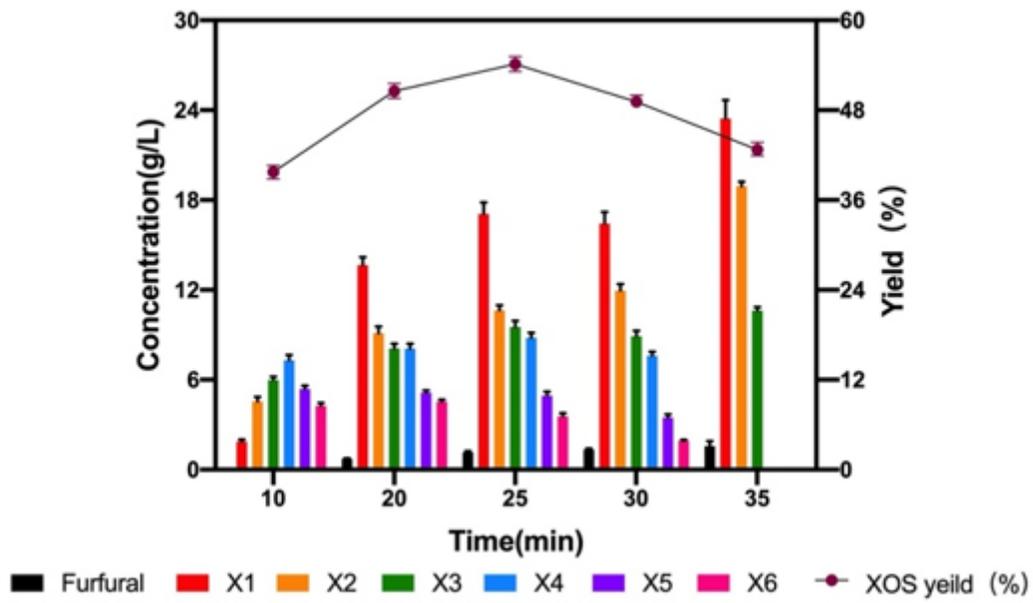
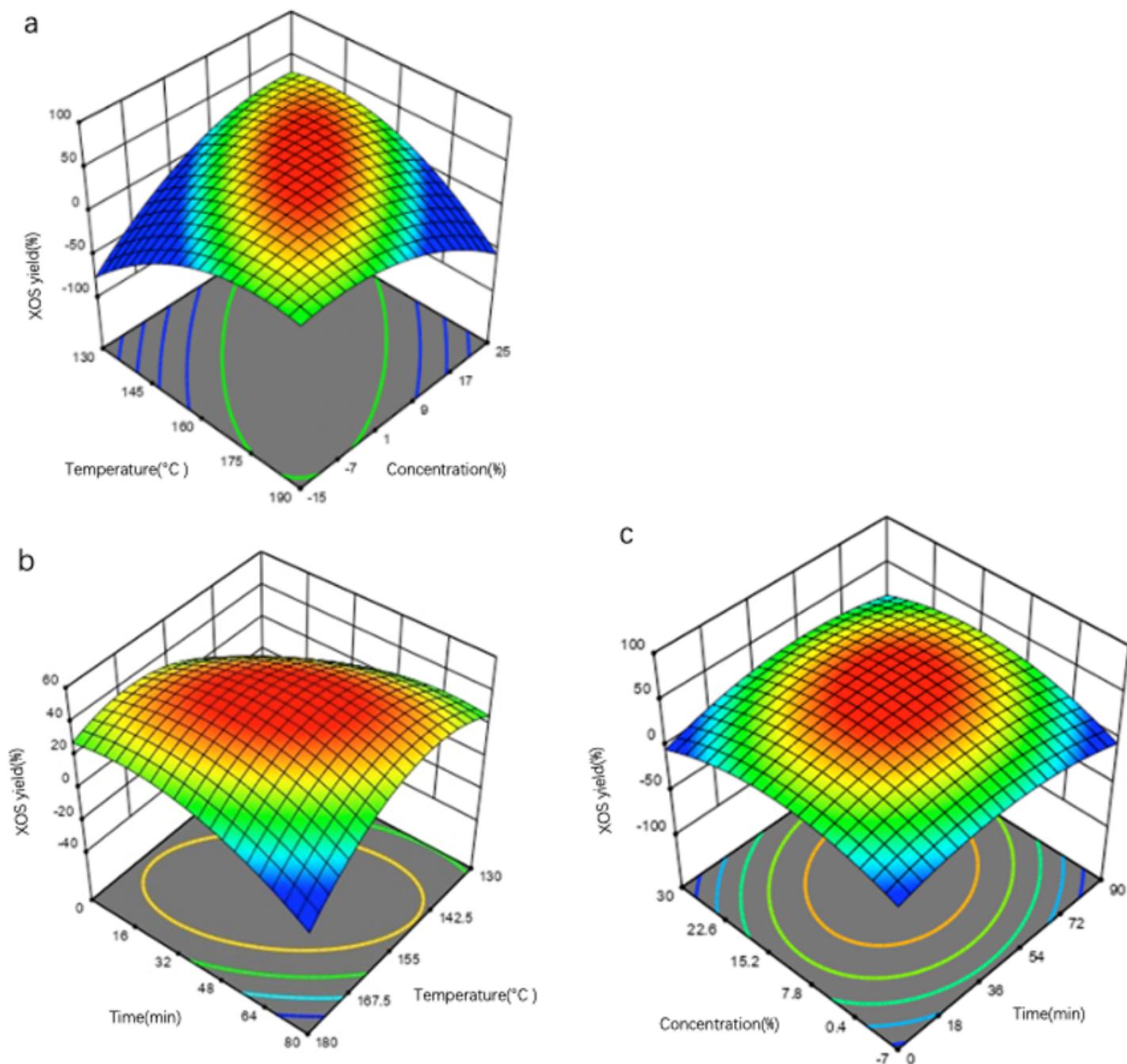


Figure 2

The concentration of furfural, X1-X6 with 5% GA at 170 °C at different times



**Figure 3**

Response surface models for the effect of independent variables on XOS yield: (a) reaction temperature (°C) and retention time (min); (b) reaction temperature (°C) and concentration of GA (%); and (c) retention time (min) and concentration of GA (%)

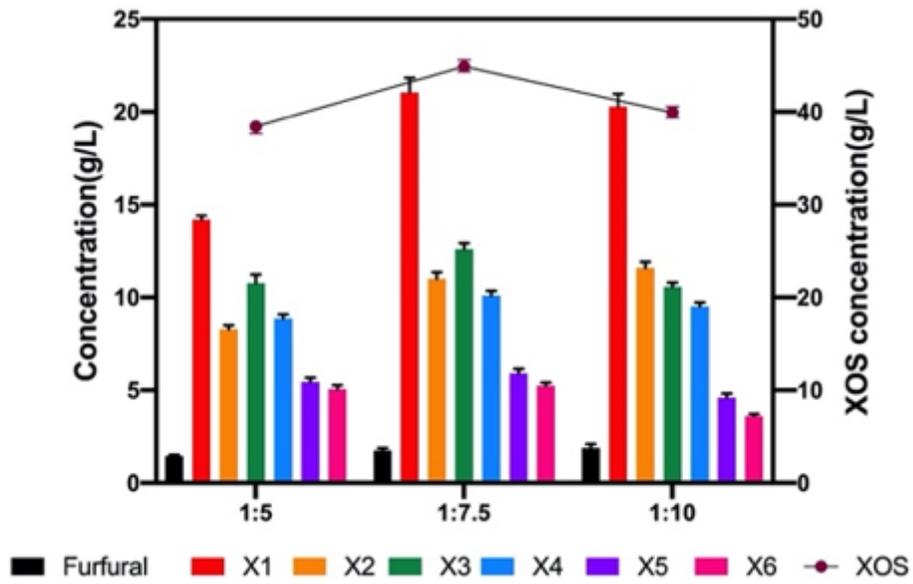


Figure 4

Composition distribution of different solid-liquid ratios under condition of 167°C,-28min-4.6%GA