Food Chemistry: X 23 (2024) 101517

Contents lists available at ScienceDirect

Food Chemistry: X

journal homepage: www.sciencedirect.com/journal/food-chemistry-x

Analysis of differences in physicochemical properties of different sorghum varieties and their influence on the selection of raw materials for winemaking

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ARTICLE INFO

Keywords:
Brewing industry
sorghum
Physicochemical properties
Starch characteristics
Maotai-flavour baijiu

ABSTRACT

Sorghum is one of the oldest crops in the world, an important grain crop in northern China, and a major raw material in the liquor-brewing industry. The physicochemical properties, cooking characteristics, and starch quality of sorghum seeds considerably affect the liquor-brewing process.To select suitable sorghums for liquor brewing and to determine the cooking characteristics and starch physicochemical properties of different sorghum varieties, 30 types of sorghum were used in this study, and their compositions were compared; six types of sorghum were further studied for their cooking quality and starch physicochemical and pasting characteristics. Gas chromatography time of flight mass spectrometry was used to analyse the cooking aroma of sorghum seeds. Additionally, scanning electron microscopy, a rapid visco analyser, and a differential calorimetric scanner were used to analyse the microstructure of sorghum starch, starch pasting characteristics, and thermodynamic properties, respectively. The results revealed that the water absorption and saccharification forces of glutinous sorghum were higher than those of japonica sorghum and that the aroma substances were significantly different. Glutinous sorghum starch had high crystallinity, freeze-thaw stability, and enthalpy, thus indicating its structural stability.

This study provides a theoretical basis for the selection of wine raw materials in the future.

1. Introduction

Sorghum (*Sorghum bicolor (L.) Moench*) is one of the oldest crops, widely grown in semi-arid regions of the world, and is the fifth largest cereal crop worldwide. (Mindaye, Mace, Godwin, & Jordan, 2016; Monkhan, Chen, & Somta, 2021;Rhodes et al., 2017). Sorghum has a 5000-year history of cultivation in China and is an important crop in the northeast, north, southwest, and other regions, mainly in Inner Mongolia, Jilin, Guizhou, Heilongjiang, Liaoning, Shanxi, and other provinces(Gui, Niu, & Hu, 2019;Zhou, 2019).Chinese Maotai-flavour baijiu is an alcoholic beverage made using sorghum as the main raw material, utilising a large song via the processes of cooking, saccharification, fermentation, distillation, storage, and blending(Su, Tzeng, & Shyu, 2010;Tang & Zhu, 2022). The sorghum variety has a direct impact on the quality of baijiu(Liu, Ao, & Wang, 2016). The different starch structures can be classified into glutinous sorghum and japonica sorghum; the proportion of straight-chain starch in glutinous sorghum is minimal, whereas that in japonica sorghum is higher(Feng, Han, & Yuan, 2015). The straight-chain starch content of sorghum directly affects its pasting and gelling characteristics, pasting temperature, and peak viscosity.

Maotai-flavour baijiu, also known as the national liquor, is the most typical of Moutai in Guizhou Province(Li, Cheng, & Wang, 2022). The raw materials used in brewing for the Moutai wine enterprises are mostly locally produced red-tasselled sorghum, which can retain the grain intact after nine cooking cycles, eight fermentation cycles, and seven wine extractions(Duan et al., 2022). This is due to the high branched-chain starch content in the red-tasselled sorghum grain and

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https://doi.org/10.1016/j.fochx.2024.101517
Received 25 January 2024; Received in revised form 27 April 2024; Accepted 27 May 2024

the dense grain cross-section, which is favourable for multiple rounds of cooking using the traditional process for soy sauce-flavoured wines (Chen et al., 2020;Yaoyi et al., 2022). At present, the research on brewing characteristics of different varieties of sorghum is relatively few, and the research on how to choose suitable brewing is also relatively few. Therefore, by studying the physicochemical properties, starch properties and cooking characteristics of different sorghum varieties in China, this study aims to provide a theoretical basis for the evaluation of the quality standards of wine sorghum, put forward requirements for the breeding of wine sorghum, and provide guidance for the material selection of Maotai liquor.

Scanning electron microscopy (SEM), X-ray diffraction (XRD), and differential scanning calorimetry (DSC) were used to investigate the differences in starch content among the sorghum varieties. The results of this study are expected to serve as a guide for the selection of raw materials for Maotai-flavour baijiu and improve the quality of the raw liquor.

2. Materials and methods

2.1. Materials

A total of 30 samples of different sorghum varieties were collected from different provinces of the country as well as from abroad; the names and origins of the samples are shown in Table 1. Sample No. 27 was the original grain used for brewing provided by a brewery in Guizhou, so it was named X Distillery, and the rest were purchased from an agricultural website(https://www.cnhnb.com/). The neatness of the varieties tested was not <98%.

2.2. Compositional analysis of sorghum

A total of 1000 grains were randomly collected from the sorghum samples and weighed to a thousand kernel weight. Sorghum moisture was determined using a moisture meter (Youke, Shanghai, China), ash was determined using the scorching method, fat was determined using the Soxhlet extraction method, protein was determined using a Kjeldahl nitrogen tester (Shanghai, China), starch was determined using a Total Starch (AA/AMG) Assay Kit (Megazyme, Wicklow, Ireland), branched-chain starch was determined using a Megazyme Amylose/Amylopectin Assay Kit (Megazyme, Wicklow, Ireland).

2.3. Characterization of Sorghum cooking

Whole-grain sorghum was weighed and placed into a beaker, soaked in boiling water, and steamed in a steamer for 1 h. The water absorption, resistance to cooking, iodine blue value, and saccharification power of sorghum were observed and determined during and after cooking.

2.3.1. Water absorption

The boiled sorghum was removed, left to stand for 15 min, filtered through a double gauze, and the filtrate was used as the liquid to be measured. The mass of the solids after filtration was denoted as M, and water absorption was calculated according to the following equation:

Water absorption = (M-40)/40 × 100%.

2.3.2. Steaming resistance

Using the high-temperature steaming method, 100 grains of sorghum were obtained from each treatment, and the samples were placed at room temperature for 12 h. After soaking, they were placed into a steamer for 60 min at high heat, and the seed coat cracks of the grains were counted after steaming.

2.3.3. Iodine blue value

The filtrate (5 mL) was added to 50 mL of water, and 5 mL of 0.5 moL/L HCl solution as well as 1 mL of 2 g/L iodine reagent were added; the volume was fixed to 100 mL and measured using a UV spectrophotometer with a wavelength set at 620 nm.

2.3.4. Saccharification power

Crushed sorghum (10 g) was weighed in a beaker, 200 mL of boiling distilled water was added and stirred well, and it was sealed in a boiling

Table 1
Physical properties and composition of different sorghum varieties.

No.	Varieties	Place of origin	1000/g	Hardness/kg	Ash /%	Hydration/%	Fat/%	Starch/%	Amylopectin/%	Protein/%
1	Jiniang4	Hebei,China	20.60	5.67	1.47	13.96	2.33	62.31	86.40	9.11
2	Jiniang6	Hebei,China	22.00	9.31	1.56	13.09	2.30	65.55	90.00	8.72
3	Hongyingzi	Guizhou,China	21.24	7.80	1.68	12.07	2.33	64.48	78.08	9.09
4	Jiaai60	Shanxi,China	36.60	7.08	1.33	13.04	2.43	66.44	79.60	9.20
5	Jingdu110	Hebei,China	24.00	5.53	1.52	13.01	2.36	65.20	77.48	9.01
6	Jingdu150	Hebei,China	29.34	7.81	1.45	11.59	2.36	66.71	76.82	9.65
7	Jiniang2	Hebei,China	11.66	6.89	1.63	12.42	2.35	61.06	87.76	8.79
8	Jingdu120	Hebei,China	14.66	5.74	1.52	13.03	2.28	62.44	78.15	9.21
9	Langnuohong19	Sichuan,China	18.00	5.57	1.45	11.28	2.32	65.22	85.37	8.53
10	Xiaonuoliang2	Sichuan,China	26.34	6.81	1.58	10.91	2.30	65.45	86.26	10.83
11	Niangaoliang	Jilin,China	25.00	5.52	1.52	15.70	2.30	62.66	80.00	8.37
12	Tiaozhou1	Shanxi,China	27.34	8.49	1.51	11.90	2.34	55.50	74.00	11.21
13	Dingxinnuo	Shanxi,China	22.66	8.67	1.58	12.70	2.31	65.70	95.28	8.89
14	Aigandatou	Jilin,China	23.32	5.04	1.49	12.48	2.38	65.14	87.12	9.66
15	HuangheGaoliang	Shanxi,China	26.34	8.10	1.53	12.67	2.33	63.93	78.00	8.87
16	Wanbeihong	Anhui,China	30.00	6.80	1.24	12.26	2.31	61.77	83.70	8.16
17	Jingdu4	Hebei,China	28.98	10.05	1.47	11.63	2.30	60.79	78.00	9.28
18	Hongnuo16	Hebei,China	20.00	6.37	1.46	13.94	2.33	62.16	83.61	9.01
19	Hongyingzi2	Guizhou,China	18.50	6.31	1.45	13.17	2.35	54.23	95.00	8.44
20	YunnanGaoliang	Yunnan,China	25.00	6.40	1.25	12.34	2.39	62.01	82.24	8.32
21	Jingdu5	Hebei,China	25.34	9.42	1.15	12.78	2.35	61.19	77.00	10.92
22	Lutongbai	Shandong,China	29.98	6.20	1.21	11.52	2.33	60.24	80.00	10.67
23	Jingdu100	Hebei,China	18.00	6.14	1.51	12.91	2.34	60.57	78.00	9.23
24	Jinza12	Shanxi,China	31.50	9.32	1.16	12.87	2.33	68.62	78.00	7.01
25	Dongbeihong	Dongbei,China	17.80	7.15	1.67	11.82	2.28	60.83	82.40	7.80
26	Shandnghong	Shandong,China	27.20	6.56	1.22	12.29	2.42	64.56	77.02	8.50
27	X Distillery	Guizhou,China	17.60	6.43	1.58	12.65	2.27	63.55	90.40	8.92
28	Dalishi	American	20.40	6.58	1.27	12.59	2.38	67.74	81.54	7.46
29	Maotaihong2	Guizhou,China	21.31	6.13	1.44	13.78	2.36	64.29	94.37	9.23
30	Liangnuo1	Hunan,China	23.46	6.79	1.42	11.37	2.38	64.17	93.97	10.40

water bath paste for 2 h. The sample was removed and cooled to room temperature, followed by addition of 0.1 g of saccharification enzyme and sealing in a stirred 60 °C water bath for saccharification. After 4 h, an appropriate amount of sorghum saccharification solution was added, the supernatant was centrifuged, and the reducing sugar content was determined to reflect the saccharification properties of the sorghum(Xia et al., 2022).

2.3.5. Detection of volatile substances in sorghum

The sorghum was crushed and 1 g of crushed sorghum was transferred to centrifuge tubes. 2-methylhexanoic acid (2 μL, 18.34 mg/mL, chromatographically pure) was added to each tube as the internal standard solution, 2 mL of saturated saline was added, and then 2 mL of the extractant solution (anhydrous ether:pentane = 1:1) was added. The mixture was thoroughly mixed for >3 min, and the upper layer of the organic phase was collected after the separation of the layers was allowed to stand. The organic phase was concentrated to 250 μL using a nitrogen blower(Liu et al., 2023).

Volatiles were detected using gas chromatography–time-of-flight mass spectrometry (GC/Q-TOF MS) (Agilent 8890–7250, America). The chromatographic column was two DB-WAXs (30 m × 0.25 mm × 0.25 μm, Agilent, America) in series. The injection volume was 1 μL, and the partition ratio was 20:1. The GC heating procedure was as follows: the target temperature was held at 35 °C for 1 min, the temperature was increased to 50 °C at 10 °C/min and held for 3 min, the temperature was increased to 100 °C at 3 °C/min and held for 3 min, and the temperature was increased to 235 °C at 3 °C/min and held for 20 min. The carrier gas flow rates were 1.0 mL/min and 1.2 mL/min. The mass spectra were performed in electron collision mode (70 eV), and the auxiliary heating temperature of the transfer line between the ion source and the DB-WAX column was 230 °C. The temperature of the transfer line was set at 1.5 °C. Detection was performed in scanning mode in the m/z 33–350 range.

2.4. Characterization of sorghum starch

2.4.1. Extraction of sorghum starch

Sorghum starch was extracted using wet milling. Peeling was performed prior to starch extraction. Sorghum samples (100 g) were weighed, washed, and soaked in 200 mL of 2.5 g/L NaOH solution for 24 h. After soaking, samples were washed repeatedly and then soaked in 60 g/L NaOH solution and a water bath at 55 °C for 10 min. The seed coat was scrubbed to remove it, the right amount of water was added, and it was polished using a grinder at high speed. After grinding, the seed coat was washed several times with water until the red precipitate on the surface disappeared and only the white starch precipitate remained. The washed white precipitate was dried in a 40 °C oven overnight and a 200-mesh sieve was used to obtain sorghum starch samples(Silva et al., 2019).

2.4.2. Scanning electron microscopy (SEM)

Scanning electron microscope (Quanta 250FEG, FEI, USA) was utilized to observe the starch granules of different varieties. An appropriate amount of dried sorghum starch was evenly spread on an aluminum sample stage and then gold sprayed to observe the starch morphology at 5000× magnification.

2.4.3. X-ray diffractometer (XRD)

An X-ray diffractometer (Rigaku Miniflex 600, Japan) was used to determine the crystal structure of sorghum starch grains. Measurement parameters(Bhat & Riar, 2019): the test target was a copper target, the scanning range was 5–45°, the scanning speed was 10°/min, the diffraction angle was 2θ, and the tube voltage and current were 45 Kv and 40 mA, respectively.

2.4.4. Determination of starch chain length distribution

Starch (10 mg) was dissolved in 5 mL water in a boiling water bath for 60 min. Sodium azide solution (10 μL 2%*w/v*), acetate buffer (50 μL, 0.6 M, pH 4.4), and isoamylase (10 μL, 1400 U) were added to the starch dispersion, and the mixture was incubated in a water bath at 37 °C for 24 h. The hydroxyl groups of the debranched glucans were reduced by treatment with 0.5% (*w/v*) of sodium borohydride under alkaline conditions for 20 h. The preparation about 600 μL was dried in vacuo at room temperature and allowed to dissolve in 30 μL of 1 M NaOH for 60 min. Then, the solution was diluted with 570 μL of distilled water.

The sample extracts were analyzed by high-performance anion-exchange chromatography (HPAEC) on a CarboPac PA-200 anion-exchange column (4.0 * 250 mm; Dionex) using a pulsed amperometric detector (PAD; Dionex ICS 5000 system). Technical support is provided by Sanshu Biotech. Co., LTD. Flow rate, 0.4 mL/min; injection volume, 5 μL; solvent system, 0.2 M NaOH: (0.2 M NaOH, 0.2 M NaAc); gradient program, 90:10 V/V at 0 min, 90:10 V/V at 10 min, 40:60 V/V at 30 min, 40:60 V/V at 50 min, 90:10 V/V at 50.1 min, 90:10 V/V at 60 min.

2.4.5. Determination of relative molecular weight of starch

Weigh exactly 5 mg of starch and dissolve it in 1.5 mL dimethyl sulfoxide (DMSO) solution containing 0.5% (w/w) LiBr, then stir in a boiling water bath to dissolve for 24 h. Samples were filtered with a 0.45 μm ptfe microporous membrane before testing. The relative molecular weight of starch was determined by gel permeation chromatography (GPC). The column temperature was maintained at 45 °C, and the mobile phase was chromatographically pure DMSO solution. The flow rate was 1 mL/min, and the sample size was 20 μL.

2.4.6. Transmittance of starch

Add distilled water into the starch sample to make the concentration of starch milk 1%, measure 25 mL of starch milk and pour it into a 50 mL beaker, then put it into a boiling water bath for 15 min with constant stirring, and finally cool it down to room temperature. The transmittance of starch paste was measured at 620 nm, and distilled water was used as blank control.

2.4.7. Freeze-thaw stability

Take 3.0 g of starch to prepare 6% starch solution. Take the appropriate amount of starch solution was transferred into a centrifuge tube, boiling water bath for 30 min, cooled to room temperature and placed in −18 °C for 12 h, thawed and centrifuged at 3000 ×g for 20 min, and freeze-thawed three times repeatedly(Ye et al., 2016).

2.4.8. Rapid viscosity analysis(RVA)

The parameters of starch paste characteristics were analyzed using a rapid viscometer (Perten, RVA4800, Sweden) by adding 3 g of starch to an aluminum crucible and mixing well with 25 mL of ultrapure water. Starting from 50 °C and keeping 1 min, the temperature was uniformly increased to 95 °C at 6 °C/min and kept for 5 min, and then cooled down to 50 °C at 6 °C/min (kept for 2 min), and the RVA curves were measured to obtain the paste temperature (pasting remperature, PR), peak viscosity (peak viscosity, PV), peak time (peak time, PT), thermal paste characteristic parameters, and the temperature of the starch paste (Jin, Kong, & Wang, 2019). Peak viscosity (PV), peak time (PT), tourgh viscosity (TV), final viscosity (FV), breakdown value (BD) and setback value (SB) were obtained.

2.4.9. Differential scanning calorimeter (DSC)

The thermal properties of starch were determined by differential scanning calorimeter (TA Q2000, DSC2500, USA). Weighing 3 mg of starch was placed in an aluminum crucible, 10 μL of deionized water was added and sealed with a matching base. The starch was left at room temperature for 4 h. The starch was heated with nitrogen gas at 10 °C/min from 30 °C to 120 °C, and a sealed blank aluminum crucible was used as a control(Juncai et al., 2023). Thermal characterization of starch was obtained.

3. Results and discussion

3.1. Analysis of quality components of different sorghum varieties

The thousand-grain weight, hardness, and component composition of 30 different sorghum varieties were determined. As shown in Table 1, the thousand-kernel weights ranged from 11.66 to 36.60 g among the 30 varieties, with large differences, the lowest being Jiniang2 and the highest being Jiaai60. The hardness of sorghum kernels of different varieties ranged from 5.67 to 10.05 kg, with the highest hardness being Jingdu4 and the lowest being Jiniang4. The ash content ranged from 1.15% to 1.68%, with the highest in Hongyingzi and the lowest in Jingdu5. The sorghum moisture content ranged from 10.91% to 15.70%. With the exception of Niangaoliang, the moisture content in the rest of the sorghum was less than 14%, which conformed to the raw-grain moisture requirement in the local standard for sorghum used for brewing Maotai-flavour baijiu in Guizhou Province (DB52/T 867–2014).

Lipid products in sorghum, obtained via oxidation or hydrolysis, are one of the main sources of flavour substances and flavour precursors in liquor. However, most lipid products have many off-flavours. Therefore, the lower the lipid content of the raw materials for brewing Maotai-flavour baijiu, the better. Thirty different sorghum varieties have lipid contents ranging from 2.27% to 2.43%, with the lowest being X Distillery and the highest being Jiaai60. Sorghum starch provides energy for the growth and metabolism of microorganisms during the fermentation process and is also a substrate for generating flavour precursor substances. The greater the proportion of branched starch to total starch, the more favourable it is to increase the yield. Branched starch has many branching structures, easily absorbs water and pasteurises, and has a good water-holding capacity, which is conducive to microbial

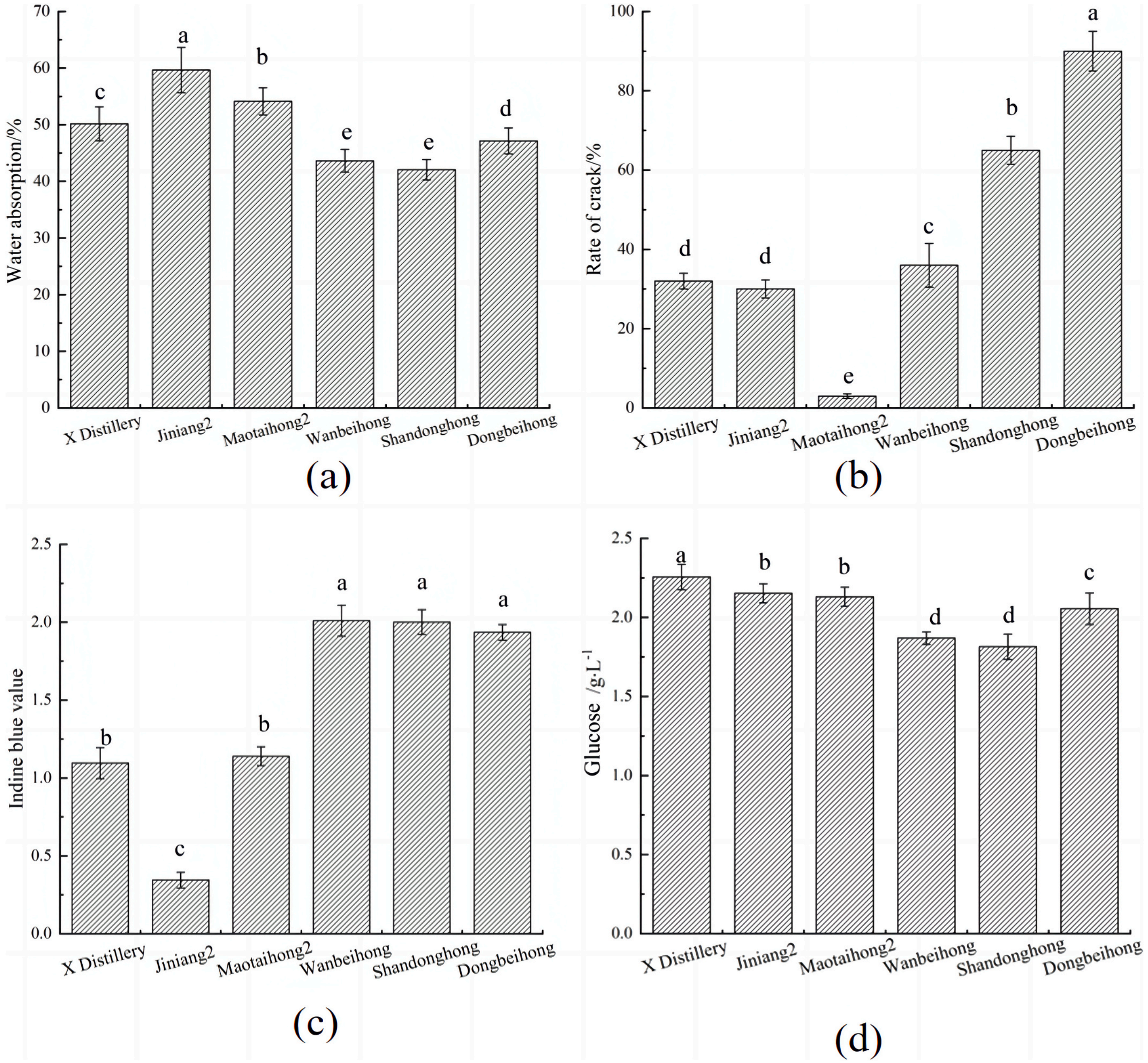

Fig. 1. Cooking characteristics of different sorghum varieties. (a) Water absorption; (b) splitting rate; (c) iodine blue value; (d) saccharification force. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

decomposition and utilisation. The content of branched starch in these 30 sorghums ranged from 74% to 95.28% of the total starch, with the highest being Dingxinnuo and the lowest being Tiaozhou1. The proteins in the sorghum provide a nitrogen source for microbial growth, and at the same time, the peptides and organic acids produced by metabolism are the flavour precursors of the Maotai-flavour baijiu; however, the protein content should not be too high, as it is prone to producing bad flavours. The measured protein content of sorghum ranged from 7.01% to 11.21%, with the highest being Tiaozhou1 and the lowest being Jinza12.

To further study the germplasm differences between brewing and non-brewing sorghum, combined with the composition and geographical distribution of different sorghum varieties, six varieties, X Distillery, Jiniang2, Maotaihong2, Dongbeihong, Shandonghong, and Wanbeihong, were selected for the next step of the study. According to the regulations on the seed composition of glutinous sorghum for brewing Maotai-flavour baijiu (DB52/T 867–2014) and glutinous sorghum for brewing small-curve clear-flavour baijiu (T/CQJX 1–2023), the three varieties X Distillery, Jiniang2, and Maotaihong2 met the varietal requirements for glutinous sorghum, and the three varieties X Distillery, Jiniang2 and Maotaihong2 met the varietal requirements for glutinous sorghum. Dongbeihong, Shandonghong, and Wanbeihong met the varietal requirements of japonica sorghum.

3.2. Analysis of Sorghum cooking characteristics

3.2.1. Analysis of sorghum cooking quality

In the brewing production of liquor, the water absorption of raw materials is a key factor in adjusting the cooking process of runny grains. The water absorption properties of different sorghum varieties are shown in Fig. 1a, and there are simultaneous differences in the water absorption capacity in which the water absorption rate of glutinous sorghum is significantly greater than that of japonica sorghum. This is because glutinous sorghum has a high branched-chain starch content, and the molecular composition of branched-chain starch has more branches, which makes it easy to absorb water for pasting and has good water-holding capacity, whereas straight-chain starch has poor water-holding capacity owing to the presence of cross-linking.

The cracking rate of sorghum during cooking reflects its cooking resistance, and the cracking rates of different sorghum varieties under the same cooking time are shown in Fig. 1b. The cracking rate of glutinous sorghum was significantly lower than that of japonica sorghum, indicating that glutinous sorghum is more resistant to cooking and more suitable for the brewing process of Maotai-flavour baijiu using multiple cooking cycles.

The iodine blue value indicates the degree of binding of straight-chain starch to iodine monomers. The iodine blue values of different sorghum varieties are shown in Fig. 1c. The iodine blue value of japonica sorghum was significantly greater than that of glutinous sorghum, indicating that glutinous sorghum contains less straight-chain starch and is easier to paste.

Starch is subjected to the action of glycolytic enzymes and further decomposed into small-molecule sugars. The saccharification power of different sorghum varieties is shown in Fig. 1d. The saccharification power of glutinous sorghum is significantly higher than that of japonica sorghum, which is due to the hydrolysis of starch by saccharolytic enzymes starting sequentially from the non-reducing end of starch, in which straight-chained starch contains only one non-reducing end, and branched-chained starch has more branches with multiple non-reducing ends; therefore, starch with a high branched-chained amylopectin content is more likely to be saccharified and utilized.

3.2.2. Analysis of sorghum cooking aroma

To study the aroma characteristics of different varieties of raw sorghum grain, the volatile components and relative contents of six different varieties of sorghum samples were analyzed and identified using liquid-liquid extraction coupled with GC/Q-TOF MS. 34 shared volatile substances were detected, including eight esters, nine acids, three ketones, four alcohols, and ten other furans and phenols. A heat map was drawn based on the volatile substance contents, as shown in Fig. 2a. With 34 shared aroma components as dependent variables and the different varieties as independent variables, effective differentiation of the six varieties of sorghum samples was achieved using OPLS-DA, as shown in Fig. 2b. The fit index (R^2x) for the independent variables in this analysis was 0.997, that for the dependent variables was 0.99, and a model prediction index greater than 0.5 indicated an acceptable model fit. After 200 replacement tests, Fig. 2c shows that the intersection of the Q^2 regression line with the vertical axis is less than 0, indicating that there is no overfitting of the model, the model is validated, and the results are considered to be useful for varietal identification analysis of sorghum aroma(Chaodi et al., 2022). Among the 34 aroma components, those with VIP values > 1 are ethyl acetate, acetic acid, butyl ester, sorbic acid, linoleic acid, 9,12-octadecadienoic acid, 2-butanone, acetoin, 3-hexanol, resorcinol dimethyl ether. A total of nine significant differences in the aroma substances were detected. The VIP value graph is shown in Fig. 2d.

3.3. Analysis of Sorghum starch properties

3.3.1. Microstructure of sorghum starch

Studies have shown that the microstructural analysis of sorghum seed starch is an important part of its physicochemical properties, and the internal structural morphology of seed starch granules influences the starch physicochemical properties. Fig. 3a shows the microstructure of starch granules of different sorghum varieties, from which it can be seen that most of the sorghum starch granules showed irregular polyhedral structure and some of the granules indicated depressions and honeycomb pores. The surface of Maotaihong2 starch granules was smoother than that of Shandonghong starch granules. The particle sizes of the six sorghum starch species ranged from 2 to 25 μm.

3.3.2. Determination of crystalline structure of sorghum starch

Crystallinity significantly affects the properties of starch granules, such as water absorption, swelling, pasting, and hydrolysis(Li, Dhital, Gilbert, & Gidley, 2020). As shown in Fig. 3b, the diffraction peaks of sorghum starch are mainly distributed between 10° and 50°. The left and right ends of the full peaks are connected as the back bottom, and the starting points of the left and right ends of the sharp peaks are represented by the dotted line, where the dotted line is the crystalline region and below the dotted line is the non-crystalline region. Origin was used to calculate the areas of the crystalline and non-crystalline regions, and the ratio of the two was the degree of crystallinity. From Fig. 3c, it can be seen that Jiniang2 has the highest crystallinity (23.94°), followed by Maotaihong2 (20.97°). X Distillery and Dongbeihong have similar crystallinity (19.25° and 19.92°), and Wanbeihong has a crystallinity of 14.4°. Shandonghong exhibited the lowest crystallinity (10.77°). The starch crystallinity of glutinous sorghum was significantly higher than that of japonica sorghum. During the brewing process of Maotai-flavour baijiu, the crystalline structure of starch granules is often destroyed by high-temperature cooking; starch granules with high crystallinity are not easily hydrolysed by enzymes and have a higher pasting temperature, which is suitable for the multiple pasting processes of Maotai-flavour baijiu(Zong, Wen, Mou, Wang, & Li, 2022).

3.3.3. Determination of freeze-thaw stability of sorghum starch

Sorghum starch paste maintains a certain water-holding capacity after cooling, and through many freeze-thaw cycles, the starch structure changes, the water-holding capacity decreases, and the water precipitation rate increases. The water-precipitation rates of the different sorghum starch varieties are shown in Fig. 3d. Jiniang2 and Maotaihong2 had the lowest water-precipitation rates, indicating that the structural stability of these starch varieties was better, whereas Shandonghong and

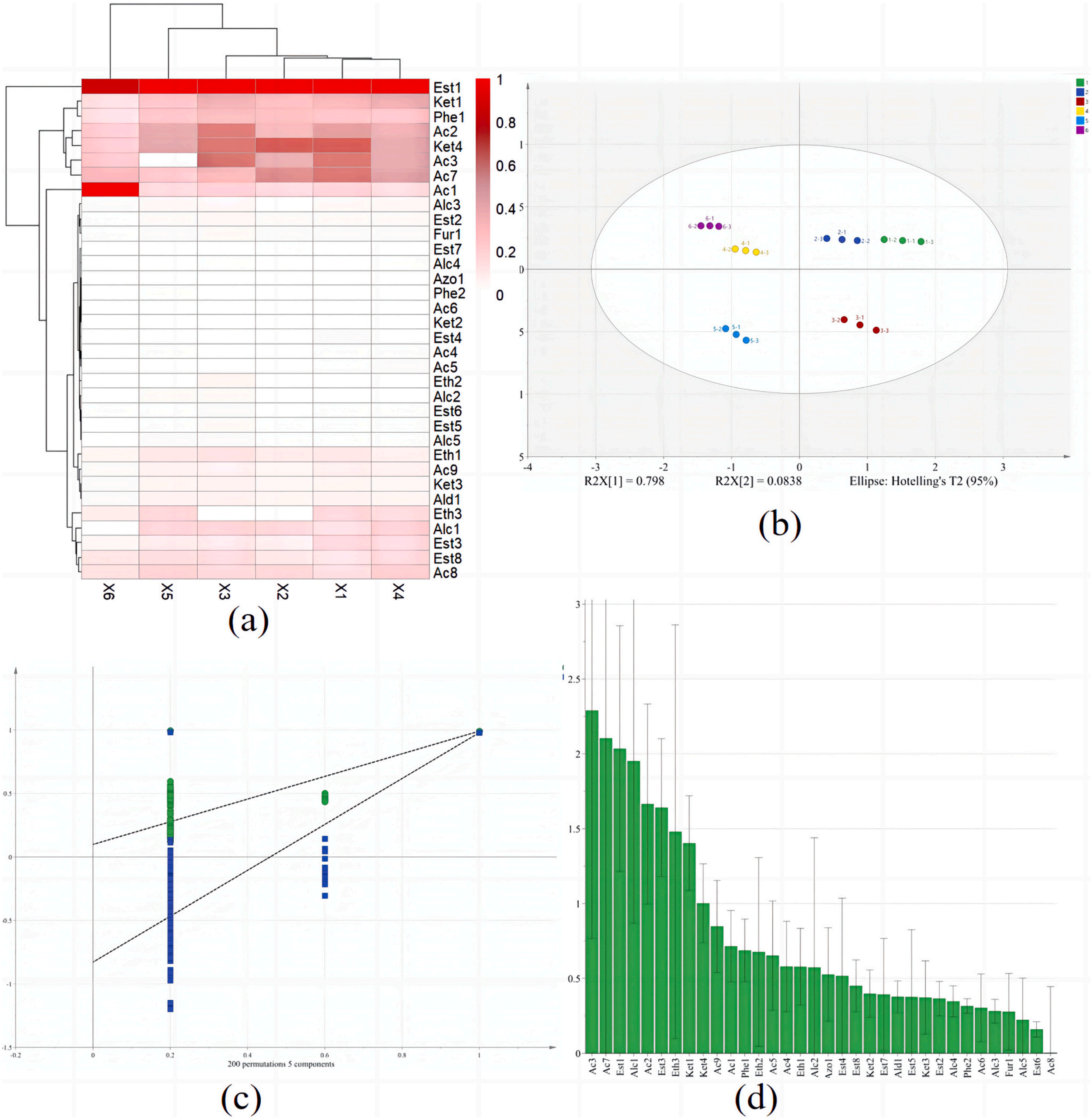

Fig. 2. Opls-da model check. (a) Thermograms of cooking volatiles of different sorghum varieties (b) PLS-DA scatter plots (c) model replacement test (d) VIP value plots.

Dongbeihong had the highest water-precipitation rates, indicating that the structural stability of these starch varieties was worse. The high-temperature and high-acid production environment of Maotai-flavour baijiu, as well as the complex brewing process, have high requirements for the structural stability of starch.

3.3.4. *Determination of light transmittance of sorghum starch*

Light transmittance is an index of the transparency of the pasted starch, reflecting the degradation process of the pasted starch(Li et al., 2020). The transmittance of sorghum starch is related to the degree of aging of the starch paste, and the starch transmittance of different sorghum varieties is shown in Fig. 3e. There was a significant difference in the transmittance of the different starch varieties. Glutinous sorghum starch paste was dispersed uniformly with low aging, and the light transmittance (54%–60%) was significantly higher than that of japonica sorghum starch. japonica sorghum has a high content of straight-chain starch; the starch granules swelled and did not collapse after pasting, and accompanied by starch aging, the formation of gel bundles made the starch paste turbid, and the light transmittance was low, making the light transmittance of japonica starch in the range of 24%–40%.

3.3.5. *Determination of starch chain length distribution*

The chain length distribution and average polymerization degree of 6 starch samples are shown in Table 2. The chain length distribution of starch is generally divided into four parts: chain A (DP 6–12), chain B1 (DP 13–24), chain B2 (DP 25–36) and chain B3 (DP $\geq$ 37). Hanashiro

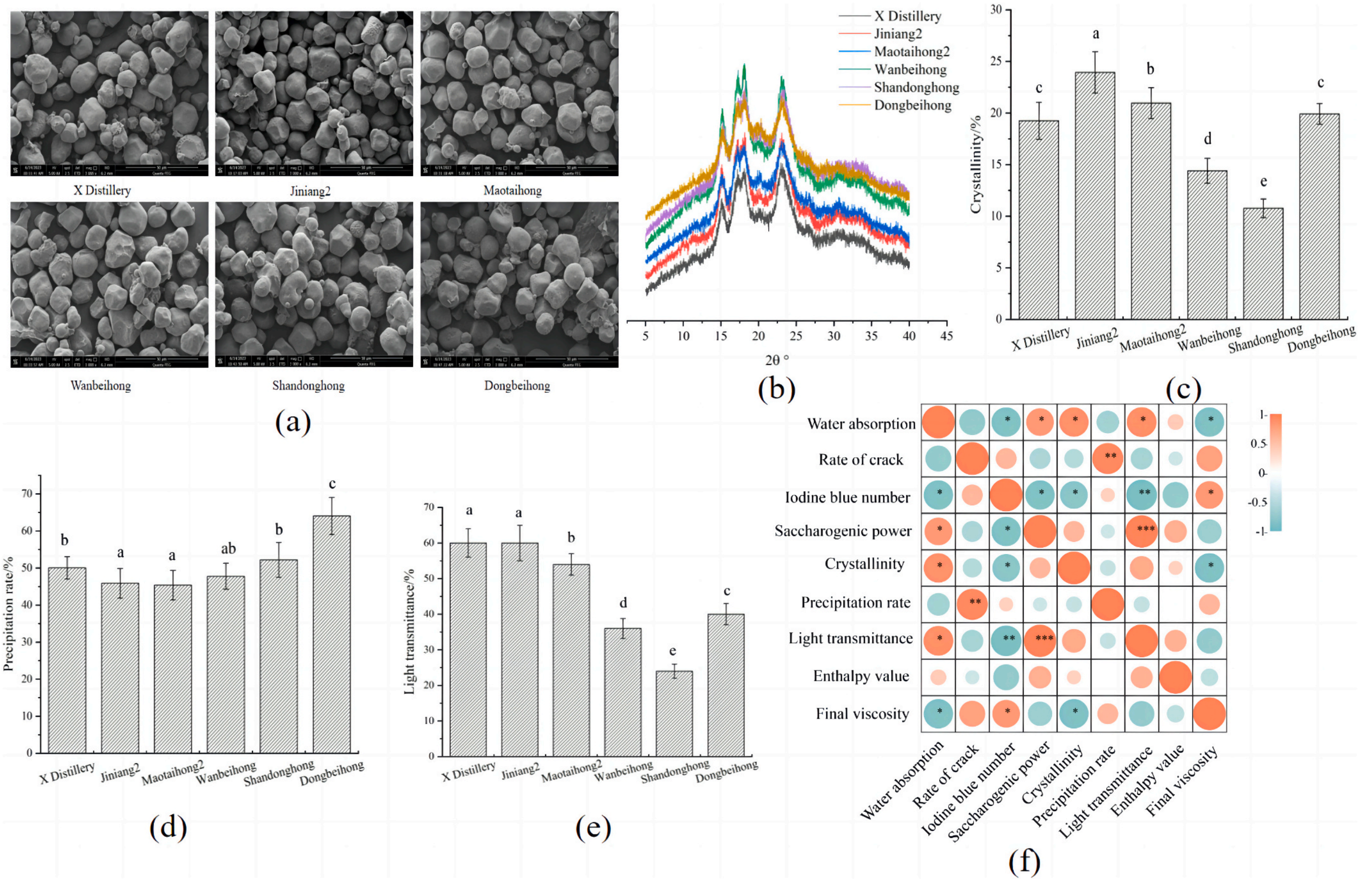

Fig. 3. Analysis of Sorghum Starch Properties. (a)Starch micromorphology of different sorghum varieties (b) X-ray diffractograms (c) Crystallinity (d)Freeze-thaw stability (e)Transmittance (f) Correlation analysis between starch cooking characteristics and starch pasting characteristics.

Table 2
Chain length distribution of different sorghum starches.

	Chain length distribution					degree of polymerization
	Afp(%)	Acltr(%)	B1(%)	B2(%)	B3(%)	
X Distillery	2.91 ± 0.03e	21.04 ± 0.09d	46.03 ± 0.17e	14.56 ± 0.06a	15.46 ± 0.11b	22.30 ± 0.12b
Jiniang2	2.84 ± 0.01f	20.91 ± 0.06e	45.97 ± 0.23f	14.57 ± 0.07a	15.72 ± 0.06a	22.42 ± 0.17a
Maotaihong2	3.47 ± 0.02b	21.49 ± 0.04b	46.29 ± 0.09d	14.24 ± 0.03b	15.22 ± 0.07c	21.80 ± 0.07e
Wanbeihong	3.29 ± 0.03c	21.27 ± 0.04c	46.87 ± 0.15c	14.12 ± 0.12c	14.45 ± 0.06e	21.81 ± 0.04d
Shandonghong	3 ± 0.05d	20.74 ± 0.07f	47.26 ± 0.24a	14.23 ± 0.08b	14.77 ± 0.07d	22.04 ± 0.08c
Dongbeihong	3.54 ± 0.05a	22.58 ± 0.01a	47.05 ± 0.18b	13.63 ± 0.11d	13.86 ± 0.04f	21.17 ± 0.05f

Note:Afp (DP 6–8); Acltr (DP 9–12); B1 (DP 13–24); B2 (DP 25–36); B3 (DP > 36).
Note:Values marked with different letters in the same column are significantly different ($p < 0.05$).

et al. believed that the short chains of starch (i.e., the A and B1 chains) could form a double helix(Hanashiro, Abe, & Hizukuri, 1996). Further studies suggest that the A chain contains Afp (DP 6–8) and Acltr chains (DP 9–12), where the Acltr chain can form a double helix while the Afp chain cannot(Fan Zhu, 2018). It can be seen from the table that the average short chain length of glutinous sorghum (chains A and B1) is smaller than that of non-glutinous sorghum, while the average long chain of non-glutinous sorghum is larger than that of non-glutinous sorghum. From the results of starch chain length distribution, it can be inferred that the double helix content of non-glutinous sorghum starch may be higher than that of glutinous sorghum starch. Glutinous sorghum starch has a higher content of long chain, and branching mainly occurs in long chain, while branching mainly occurs in short chain of non-glutinous sorghum. The average degree of polymerization showed that the average degree of polymerization of glutinous sorghum starch was higher than that of non-glutinous sorghum, and the degree of polymerization of starch molecules was closely related to its physicochemical properties. The higher the degree of polymerization, the greater the viscosity of starch, hydration ability is also stronger. At the same time, the gelation characteristics of starch during heating are also closely related to its polymerization degree. Starch with low degree of polymerization tends to gelate during heating, while starch with high degree of polymerization requires higher temperature and time to gelate.

3.3.6. *Determination of relative molecular weight of starch*

The weight average molecular weight (Mw) of starch molecules reflects the average size and distribution of all starch molecules in a starch sample, and is an important parameter to evaluate the molecular weight distribution of starch (Adam & Ghosh, 2001). The weight average molecular weight of starch samples is closely related to the particle size, composition and structure of starch particles, and there are some differences in the weight average molecular weight of starch samples from different sources. There is a certain relationship between weight average molecular weight and number average molecular weight (Mn), and there is a certain relationship between physicochemical properties, structure and function of starch. Mw and Mn of 6 starch samples are shown in Table 3. Mn and Mw values of glutinous sorghum are significantly higher than those of non-glutinous sorghum. Some studies have shown that starch with higher Mw has higher water absorption, gelling and viscosity, which can play a better stability in food processing, and is more suitable for wine making, especially for multi-round fermentation of sauce-flavour liquor. The starch sample with smaller Mn is easier to form uniform structure and taste, easy to digest, and more suitable for other food processing. The PDI of japonica sorghum is significantly higher than that of glutinous sorghum. The smaller the PDI is, the narrower and more concentrated the particle size distribution of nano-emulsion is, and the more compact the starch structure is. In practical applications, it is very important to choose the right variety according to different needs and uses.

3.3.7. *Thermal characterization of sorghum starch pasting*

Starch pasting refers to the process in which starch is heated under conditions of starch–water coexistence, resulting in the disappearance of the birefringence phenomenon in the starch granules, the absorption of water and swelling, the destruction of the crystal structure of the starch molecule and the unraveling of the double helix structure, and the precipitation of straight-chained starch(Takagi, Suzuki, Akdogan, & Kitamura, 2017;Zhiguang, Qi, Yinshuang, & Haixia, 2023). The results of the DSC of the different varieties of sorghum starch are shown in Table 4. The range of the onset pasting temperature of glutinous sorghum starch paste is 70.25–71.74 °C, and the peak pasting temperature is 76.072–76.897 °C. The paste termination temperature is 80.788–82.163 °C, and the enthalpy value is 12.578–14.788 J/g. The onset pasting temperature range of round-grained sorghum starch paste is 66.514–70.152 °C, the peak paste temperature is 70.263–75.153 °C, and the paste termination temperature is 74.578–14.788 J/g. The gelatinisation process is presented in Table 2. The peak pasting temperature was 70.263–75.153 °C, the pasting termination temperature was 74.107–80.128 °C, and the enthalpy value was 9.3379–13.415 J/g. The higher enthalpy of glutinous sorghum indicated a high degree of effective double-helix crystallisation and good structural stability. Northern japonica sorghum had a low degree of crystallisation and, therefore, a lower enthalpy of pasteurisation.

3.3.8. *Analysis of the pasting viscosity of sorghum starch*

Starch pasting is the irreversible swelling of starch molecules by heat and water absorption, accompanied by the destruction of crystalline zones, disappearance of birefringence, increase in viscosity due to swelling of starch granules, and transient decrease in viscosity due to disintegration of starch granules(Murillo, Padilla, & de la Rosa Millan, 2019;Waterschoot, Gomand, & Delcour, 2016). Starch aging is a phenomenon of precipitation, and the insolubility of starch paste when cooled or stored is known as starch aging or regrowth(Shujun, Mengge, Jinglin, Shuo, & Les, 2017).

As shown in Table 5, the peak viscosity of glutinous sorghum (3247–4076 cP) was higher than that of japonica sorghum (3088–3855 cP), indicating that the ability of starch to bind water and the amylase activity of glutinous sorghum were higher than those of japonica sorghum starch. The hot-paste viscosity is that of the sticky paste after the swelling starch grains rupture and collapse and no longer rub against each other, as is the viscosity of the starch paste when it is kept warm. Viscosity is one of the most important indicators of starch quality. The hot-paste viscosity of glutinous sorghum starch (1338–1645 cP) was higher than that of japonica sorghum (1289–1349 cP). The disintegration value is the difference between the peak viscosity and hot-paste viscosity and indicates the thermal stability of the starch paste. Starch is more easily utilized by microorganisms after disintegration, and the range of disintegration values is higher for glutinous sorghum (1909–2679 cP) than for japonica sorghum (1799–2507 cP). The final viscosity is the viscosity of the starch paste after aging due to temperature reduction, and reflects the regrowth characteristics of starch. Starch regrowth is defined as the turbidity of pasted starch while standing, producing an insoluble white precipitate. The final viscosity of japonica sorghum (2869–3485 cP) was significantly higher than that of glutinous sorghum (2064–2487 cP). The highest final viscosity was found in Wanbeihong sorghum (3485 cP) and the lowest in Maotaihong2 sorghum (2064 cP). The rebound value is the viscosity that increases during the cooling of the starch paste and reflects the degree of aging of the starch, that is, it is equal to the difference between the final viscosity and the hot-paste viscosity. Japonica sorghum starch had a high rebound value (1520–2137 cP), whereas the rebound value of the glutinous sorghum starch was significantly lower than that of japonica sorghum (518–1090 cP). The high disintegration and low rebound values of sorghum starch are important characteristics required for brewing high-quality Maotai-flavour baijiu. Glutinous sorghum has higher branched-chain amylose, and branched-chain amylose more easily absorbs water and pastes, so the time taken for glutinous sorghum starch to reach peak viscosity is less (4–4.27 min), while japonica sorghum takes more time (4.13–4.47 min). The pasting temperature of glutinous sorghum was slightly higher than that of japonica sorghum owing to the higher crystallinity of glutinous sorghum starch.

Table 3
Average molecular weight of different sorghum starches.

	Mn(g/mol)	Mw(g/mol)	PDI
X Distillery	1,093,322 ± 1180c	2,837,166 ± 177d	2.59 ± 0.003d
Jiniang2	4,018,175 ± 17294a	5,156,155 ± 1363c	1.28 ± 0.006f
Maotaihong2	3,504,680 ± 993b	7,760,612 ± 394a	2.21 ± 0.0007e
Wanbeihong	741,116 ± 343d	7,338,380 ± 399b	9.90 ± 0.004a
Shandonghong	576,781 ± 313f	2,349,650 ± 3883e	4.07 ± 0.008b
Dongbeihong	645,441 ± 607e	1,823,780 ± 331f	2.83 ± 0.003c

Note:Values marked with different letters in the same column are significantly different ($p < 0.05$).

Table 4
Results of Thermal Characterization of Pasting of Different Sorghum Starches.

Varieties	To/°C	Tp/°C	Tc/°C	ΔH/(J/g)
X Distillery	70.25 ± 0.35ab	76.897 ± 0.76a	82.163 ± 1.24a	14.788 ± 0.07a
Jiniang2	71.74 ± 0.49a	76.072 ± 0.74ab	81.481 ± 1.17a	14.307 ± 0.12a
Maotaihong2	71.379 ± 0.44a	76.094 ± 0.68ab	80.788 ± 1.06ab	12.579 ± 0.16bc
Wanbeihong	70.151 ± 0.37ab	75.153 ± 0.56b	79.473 ± 2.09c	9.3379 ± 0.05d
Shandonghong	70.02 ± 0.31b	75.008 ± 0.46b	80.128 ± 1.82b	13.415 ± 0.07b
Dongbeihong	66.514 ± 0.25c	70.263 ± 0.38c	74.107 ± 0.78d	10.066 ± 0.13d

To:onset pasting temperature.
Tp:peak pasting temperature.
Tc:paste termination temperature.
ΔH:enthalpy value.
Note:Values marked with different letters in the same column are significantly different ($p < 0.05$).

3.4. *Correlation analysis of sorghum cooking quality and starch pasting characteristics*

The correlation between the cooking quality and pasting characteristics of sorghum starch was further explored using Spearman correlation analysis. Fig. 3f shows that the water absorption of sorghum is significantly negatively correlated with the final viscosity of starch; the higher the viscosity, the weaker the ability of starch to combine with

Table 5
Results of paste viscosity analysis of different sorghum starches.

Varieties	Peak 1	Hot-paste viscosity	Breakdown	Final viscosity	Setback	Peak Time	Pasting Temp.
X Distillery	4076 ± 89.00a	1397 ± 56.53b	2679 ± 51.00a	2487 ± 34.00d	1090 ± 28.72d	4.27 ± 0.09b	79.95 ± 0.99a
Jiniang2	3993 ± 55.00a	1645 ± 24.58a	2348 ± 26.50c	2163 ± 29.50e	518 ± 18.36f	4.00 ± 0.08c	78.40 ± 0.50b
Maotaihong2	3247 ± 41.50d	1338 ± 38.50c	1909 ± 10.96e	2064 ± 18.50f	726 ± 27.02e	4.13 ± 0.01c	79.15 ± 0.31b
Wanbeihong	3855 ± 64.00b	1348 ± 23.00b	2507 ± 28.86b	3485 ± 34.00a	2137 ± 26.52a	4.13 ± 0.01c	79.20 ± 0.12b
Shandonghong	3088 ± 88.50e	1289 ± 57.02d	1799 ± 30.00f	3112 ± 21.00b	1823 ± 23.00b	4.27 ± 0.09b	79.95 ± 0.21a
Dongbeihong	3418 ± 50.50c	1349 ± 50.50b	2069 ± 41.00d	2869 ± 54.00c	1520 ± 19.20c	4.47 ± 0.11a	78.25 ± 0.16c

Note:Values marked with different letters in the same column are significantly different ($p < 0.05$).

water. The cracking rate was significantly positively correlated with the water precipitation rate and final viscosity of starch; the easier the starch precipitates water, the easier it absorbs water and swells, resulting in cracking and poor water-holding capacity. Sorghum saccharification power was significantly positively correlated with starch transmittance, whereas sorghum starch crystallinity, transmittance, and enthalpy of pasting were significantly negatively correlated with the final viscosity of starch pasting(Zhang, Li, Wu, Yang, & Ouyang, 2019). The final viscosity reflects the recombination of straight-chain starch and the degradation of branched-chain starch. The high branched-chain starch content of glutinous sorghum results in improved cooking quality.

4. Conclusions

The fractions of 30 different sorghum varieties were measured, analyzed. Three varieties of high-quality glutinous sorghum suitable for brewing were selected: X Distillery, Jiniang2, and Maotaihong2. To further investigate the varietal differences between glutinous sorghum and japonica sorghum, three japonica sorghum varieties were selected to further compare the cooking quality and starch characteristics of the different sorghum varieties. By comparison with japonica sorghum, it was found that glutinous sorghum was higher in the indices of water absorption and saccharification force, indicating that glutinous sorghum was easier to paste and be utilized by microorganisms. Its cracking rate was lower than that of japonica sorghum, indicating that the structure of glutinous sorghum was more dense, which was better suited for the complex process of Maotai-flavour baijiu, and there were also obvious differences in the aroma substances of japonica and glutinous sorghums. By comparing the physicochemical and pasting properties of japonica and glutinous sorghum, it was found that the crystallinity, freeze-thaw stability, and transmittance of glutinous sorghum were better than those of japonica sorghum. The DSC and RVA results showed that glutinous sorghum has a higher enthalpy and slightly higher pasting temperature than japonica sorghum, indicating that its crystallisation has a high degree of effective double-helix formation and good structural stability. Therefore, for the complex process of Maotai-flavour baijiu, the selection of raw materials is very important for improving quality and yield. In the brewing process, should be combined with the actual production, different raw material physical and chemical indicators, aroma and flavour slightly different, should be optimized and adjusted according to the raw material differences in order to high-quality production.

CRediT authorship contribution statement

Xin Shi: Writing – review & editing. **Chenming Fan:** Writing – original draft. **Chunmei Pan:** Funding acquisition. **Fangli Zhang:** Resources. **Xiaoge Hou:** Data curation. **Ming Hui:** Supervision.

Declaration of competing interest

All authors have no conflicts of interest to this work. We declare that we do not have any commercial or associative interest that represents a conflict of interest in connection with the work submitted.

Data availability

Data will be made available on request.

Acknowledgments

This work was funded by the research and application of microflora of yujiu, a sub-project of major science and technology projects of henan province, China (grant number 181100211400–8), R&D and Demonstration Application of Key Technologies for Ecological Brewing of Henan baijiu, China (grant number 231111112000).

Henan University of Technology (No. 31401184).

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