Original Article
Food Science and Technology

ISSN 0101-2061 (Print)
ISSN 1678-457X (Online)

DOI: https://doi.org/10.1590/fst.81622

Effects of carrot powder on properties of pre-gelatinized waxy rice starch

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Abstract

The physicochemical properties and structure changes of pregelatinized Waxy rice starch (PWRS) were studied by measuring the thermodynamic properties, crystal structure, texture properties, dynamic rheology, freeze-thaw stability, microstructure and sensory analysis of PWRS. The results showed that when carrot powder was added, the initial gelatinization temperature of PWRS increased, the crystallinity increased first and then decreased, the crystal structure presented no changes, the pores in the microstructure increased, the water holding capacity increased, the weak binding water content also increased, and the water separation rate decreased under different freezing-thawing cycles. Compared with pure glutinous rice starch, when the addition of carrot powder was controlled within 10%, the gel strength and solid properties of glutinous rice starch would be increased, and the elasticity and hardness could also be increased. When the additive amount was more than 10%, the starch gel tends to be liquid, the hardness decreased, and the gel structure was difficult to form. Therefore, adding carrot powder can improve the gel properties of glutinous rice starch, which is expected to provide a theoretical basis for delaying the aging of glutinous rice starch.

Keywords: pregelatinized waxy rice starch; carrot powder; gel properties; structure.

Practical Application: The results provided a theoretical basis for delaying the aging of Waxy rice starch.

1 Introduction

Starch is one of the most important commodities in the world, playing an important role in many fields, among which the composition and functional changes of glutinous rice starch have a great impact on its industrial application (Thitisaksakul et al., 2021). Gelatinization and rejuvenation of starch can form gels with a certain network structure, whose texture qualities such as hardness, elasticity and chewiness have an important impact on the food quality of related products (Xie et al., 2022; Wang et al., 2022). During the industrial production of glutinous rice flour by food enterprises, in order to achieve the purpose of sterilization and rapid drying, the high temperature treatment at 180 °C will lead to the pre-gelatinization of starch (Tian et al., 2022). This study aims to explore the aging law of PWRS and provide theoretical basis for product optimization of food enterprises. In recent years, fruit and vegetable powder has been widely studied and applied in cereal food because it is rich in dietary fiber, vitamins and other essential nutrients for human body (Tumwine et al., 2019). Cellulose, pectin and other substances in fruit and vegetable powder can affect the intermolecular arrangement of starch and change the characteristics and structure of starch gel (Correa & Ferrero, 2015). Therefore, in many researches fruit and vegetable powder is added in flour products to develop products with better properties (Zhang et al., 2022). When the ratio of Chinese yam powder to low-gluten flour was 1:5, the contents of fat and fast-digestible starch were significantly reduced, while the contents of flavonoids, resistant starch, slow-digestible starch and protein were significantly increased to meet the market demand (Yalindua et al., 2021; Kurek & Sokolova, 2020). That after adding carrot powder to wheat flour, the contents of dietary fiber, β-carotene, calcium, magnesium, sodium and other minerals in bread were significantly increased, and the nutrients in the product were more in line with the daily requirements of human body (Gustinovich, 2017; Hryshchenko et al., 2019). Many researchers have added carrot powder into various flaps to develop new products such as cookies, steamed buns and cakes with carrot flavor (Hudi et al., 2021; Mohtarami, 2019; Phebean et al., 2019). Carrot powder can affect the properties and structure of starch, and then affect the processing characteristics of its products and reflect the export sensation, shape and texture (Kumar et al., 2020). In this paper, carrot powder and PWRS were used as the research object to analyze the thermodynamic properties, crystal structure, starch gel texture, dynamic rheology, freeze-thaw stability, microstructure and water distribution of mixed carrot powder and PWRS with different proportions. This study intended to explore the effect of carrot powder on the aging of PWRS, and provide a theoretical basis for food enterprises to extend the shelf life of waxy rice food.

2 Materials and methods

2.1 Preparation of carrot powder

Fresh carrots were washed and sliced with a thickness of 0.3 cm, treated with 0.1% calcium chloride and 0.2% sodium bisulfite solution for 30 min, removed and dried in an oven at 75 °C until the moisture content was below 10%, ground through a 100-mesh screen, and stored in a sealed low temperature and out of light for later use.

Received 29 June, 2022
Accepted 23 Aug., 2022
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2.2 Preparation of PWRS

Glutinous rice was ground into slurry with water, pressed and filtered into powder, dried at 180 °C and ground to make glutinous rice flour. The glutinous rice flour was immersed in 0.2% NaOH solution in a constant temperature water bath at 40 °C for 90 min, with a solid-liquid ratio of 1:8. Then the suspension was centrifuged at 3000 R/min for 15 min with a centrifuge, the yellow substance in top part was discarded, and then the upper layer was cleaned with distilled water. The suspension was allowed to stand for 8-10 h each time, and the washing was repeated for 3 times. The lower yellow substance was scraped off to be precipitated and dried at 40 °C for 16 h, and ground to pass through 100 sieve. The product was stored in a sealed low temperature and avoid light for future use.

2.3 Preparation of test sample

Carrot powder was add in PWRS according to 0%, 5%, 10%, 15%, 20%, 25% of the total sample mass, mixed evenly, sealed and stored at low temperature in dark place for later use.

2.4 Gel preparation of powder mixing system

The mixed 3 g sample was dissolve in 25 mL distilled water, stirred evenly, and then soaked in 100 °C water bath for 30 min with constant stirring, cool naturally to room temperature. The mixing system was poured into aluminum box (inner diameter 25 mm, height 50 mm) and store at 4 °C for 24h to form stable starch gel, and store for later use.

2.5 Determination of thermodynamic properties

Three milligram mixed powder samples was added into 9 uL distilled water in aluminum crucible, stirred evenly, sealed the gland, and placed in dryer at room temperature for 24 h. A differential heat scanner DSC3 (Mettler Toledo Instruments Co., LTD., Shanghai, China) with the scanning temperature range as 20-100 °C, and the heating rate 5 °C/min. The blank crucible was used as the reference. The initial gelatinization temperature (To), peak gelatinization temperature (Tp), gelatinization termination temperature (Tc) and gelatinization enthalpy ((ΔH) of the samples were recorded.

2.6 Determination of crystal structure

The crystal characteristics of PWRS supplemented with different proportions of carrot powder were studied by X-ray diffractometer XD-3 (Putuo General Instrument Co., LTD., Shanghai, China). Lay the sample flat in the sample tank, press and remove the excess sample, and place it in the X-ray diffraction (XRD) sample table for testing. Diffraction test conditions: Cu Kα ray, tube voltage 36 kV, tube current 24 mA, scanning range 4-50 °C, scanning rate 2°/min, step size 0.02°. After testing, MDI JADE6 software was used to process the data and calculate the crystallinity.

2.7 Determination of texture characteristics

The PWRS gels with different proportions of carrot powder were placed on TA.XT Plus apparatus (SMS, UK) with TPA mode and P36R probe. The measured parameters were pre-test velocity 2 mm·s^{-1}, middle test velocity 1 mm·s^{-1}, and post-test velocity 2 mm·s^{-1}. Trigger force was 5 g. The compression ratio is 50% (Fonseca-Florido et al., 2017). Samples in each group were repeated 5 times and averaged.

2.8 Determination of dynamic rheology

We used a rotary rheometer HAAKE MARS (Thermo Mercer Scientific Co., LTD., Waltham, Massachusetts, USA) to place the powder mixing system gel on the rheometer induction plate. A 40 mm fixture was selected and the vibration measurement mode was set to 2 mm, and the balance was required for 2 min to eliminate the stress before the formal test. The test parameters were set as temperature maintained at 25 °C, strain set at 0.2%, and frequency scan range at 0.1-10 Hz. The elastic modulus (G'), viscous modulus (G") and tangent value of loss Angle (Tan δ) were recorded (Sonawane et al., 2020).

2.9 Determination of freeze-thaw stability

The samples with different proportions were prepared into 5% suspension, continuously stirred and gelatinized for 30 min under boiling water bath conditions. After cooling to room temperature, 30 g was weighed and placed in a 50 mL centrifuge tube (the mass of centrifuge tube was denoted as m_0, and the total mass of centrifuge tube and starch paste was denoted as m_1), and then placed in a -22 °C refrigerator for 20 h, and then removed in a 25 °C water bath for 3 h. The centrifuge was centrifuged at 5000 r·min^{-1} for 25 min in a TDL-50C centrifuge (Plater Electrical Equipment Co., LTD., Shanghai, China), the supernatant in the centrifuge tube was removed, and the total mass (m_2) of the centrifuge tube and sediment was weighed. The freeze-thaw cycle was repeated for 5 times, and the water separation rate was calculated according to the formula. The freeze-thaw stability was expressed as the water separation rate (Equation 1).

$$\textit{Drainage rate}(\%) = (m_1 - m_2) / (m_1 - m_0) \times 100\% \tag{1}$$

2.10 Sem observation

PWRS gels from different proportions of carrot powder ware put into JL-B10N-50C vacuum freeze dryer (Jin LAN Instrument Manufacturing Co., LTD., Shanghai, China) and freeze-dried at -50 °C for 48 h. The PWRS were cut into 0.3 mm long, 0.2 mm wide and 0.1 mm thick slices and fixed on the sample table with conductive double-sided adhesive. The plates were fixed with light pressure, and platinum was sprayed for 30 s. The plates were removed and placed into the scanning electron microscope sample chamber for observation and photography. The test conditions are 2000 times magnification and 20 kV acceleration voltage (Lu et al., 2020).

2.11 Determination of low field nuclear magnetism

The NMR instrument NM21-040H-I (Niumet Analytical Instruments Co., LTD., Suzhou, China) was to determine the water migration and distribution of the samples. 2 g gelatinized samples were weighed and transferred to 15 mm NMR tubes, cooled to room temperature, and stored at 4 °C for 12 h for NMR testing. The transverse relaxation time (T2) was measured by multi-pulse echo sequence (CPMG). The detection parameters are: instrument temperature 36 °C, main frequency SF = 20 MHz, offset frequency O1 = 899295.73 Hz, 90° pulse time P90 = 7.00 μs, 180° pulse time P180 = 15.00 μs, sampling number TD = 200250, Relaxation decay time TW = 300.00 ms, accumulation times NS = 64, echo time TE = 1.00 ms (Nawaz et al., 2021; Qu & Jin, 2022).

2.12 The sensory score

Ten panelists (5 males and 5 females, aged 24-30 years) were trained and asked to rate the sensory properties of PWRS gels supplemented with different proportions of carrot powder, such as colour, smell, taste, tissue morphology, elasticity and overall acceptability. We rated these characteristics on a 5-point scale, where 1 represented very poor and 5 represented very good for the selected attributes (Monga et al., 2022). All samples were evenly cut into 2 cm cubes before sensory scoring.

2.13 Data processing and statistical analysis

All tests were repeated three times, and SPSS (Statistical Program for Social Sciences) 19.0 software was used to analyze the significance of the data. All analyses were performed at 95% confidence interval ($P<0.05$), which was considered statistically significant. Drawing using Origin Pro 8.0 software.

3 Results

3.1 Analysis of thermodynamic properties of powder mixing system

Starch aging is mainly due to the disordered starch molecules reaggregate into ordered crystalline structure as the gelatinized starch paste decreases in storage temperature (Vamadevan et al., 2014). The gelatinization characteristics of such crystalline structure can be measured by differential scanning calorimeter (Figure 1). The gelatinization initiation, peak value, termination temperature and gelatinization enthalpy of samples with different proportions were different as listed in Table 1. There were significant differences in thermodynamic properties of different proportion powder mixing systems. With the increase of the amount of carrot powder, the initial gelatinization temperature showed a rising trend, indicating that carrot powder made the double helix structure more stable. The amylose content is negatively correlated with the peak value and termination temperature of starch gelatinization (Dhen et al., 2017), so the amylose content in the powder mixing system is gradually reduced to achieve the effect of delaying aging. There was a positive correlation between gelatinization enthalpy value and crystallinity, which reflected the energy required when the mixed powder crystal region was destroyed in the process of thermodynamic change. Gelatinization enthalpy value represented the endothermic peak area in the differential scanning calorimetry (DSC) spectrum, and the higher the gelatinization value was, the lower the gelatinization level was. Therefore, the addition of carrot powder had a significant effect on the gelatinization level of glutinous rice starch.

3.2 Determination of relative crystallinity

Crystallinity of a substance reflects the proportion of crystallites in a sample, and its crystal structure can be revealed by XRD (Santos et al., 2016). After adding different proportions of carrot powder to PWRS, they all had similar XRD spectra, and there were strong diffraction peaks at the diffraction Angle of 21.4°, indicating that carrot powder did not affect the crystal structure of PWRS. When the amount of carrot powder was increased from 0 to 25%, the relative crystallinity of each component was 25.42%, 25.87%, 26.43%, 26.19%, 25.97% and 25.45% (Figure 2). When the amount of carrot powder was 10%, the relative crystallinity of the gel reached the maximum. This change law is consistent with that of gelatinization enthalpy in thermodynamic analysis.

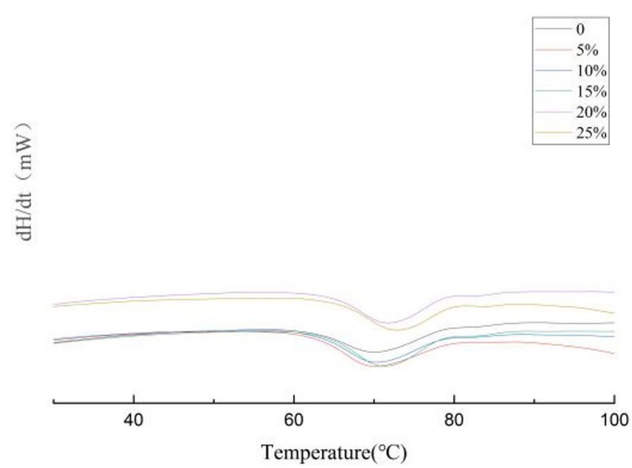

Figure 1. Pre-gelatinization with different proportions of carrot powder DSC pattern of Waxy rice starch.

Table 1. Thermodynamic properties of pre-gelatinized glutinous rice starch with different proportions of carrot powder.

Amount of carrot powder added	To (°C)	Tp (°C)	Tc (°C)	ΔHg (J/g)
0	63.87 ± 0.04^{e}	70.24 ± 0.05^{d}	78.17 ± 0.02^{c}	4.62 ± 0.04^{e}
5%	63.80 ± 0.02^{e}	69.63 ± 0.07^{e}	77.96 ± 0.02^{cd}	5.10 ± 0.01^{d}
10%	64.00 ± 0.02^{d}	70.30 ± 0.00^{d}	78.12 ± 0.03^{c}	5.76 ± 0.01^{a}
15%	64.97 ± 0.03^{c}	70.99 ± 0.03^{c}	78.26 ± 0.05^{c}	5.47 ± 0.03^{b}
20%	65.83 ± 0.05^{b}	71.98 ± 0.06^{b}	78.54 ± 0.01^{b}	5.25 ± 0.02^{c}
25%	67.04 ± 0.05^{a}	72.98 ± 0.05^{a}	79.27 ± 0.04^{a}	4.15 ± 0.03^{f}

Values are mean±standard error, different letters in the same column mean significant difference ($P < 0.05$), ^{a}is the maximum.

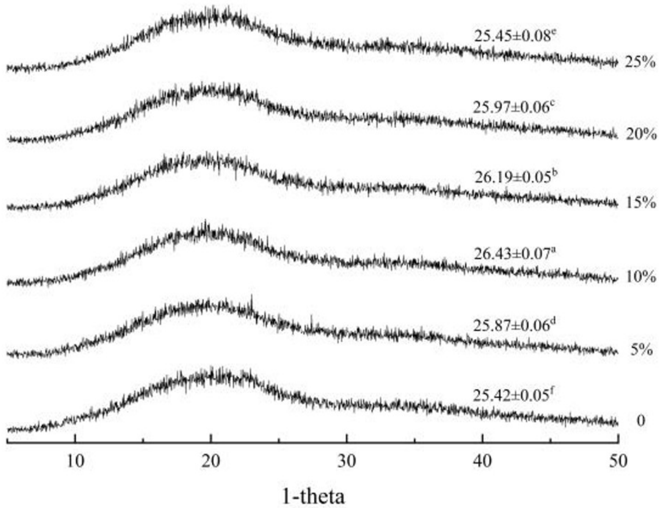

Figure 2. Pre-gelatinization with different proportions of carrot powder XRD pattern of Waxy rice starch gel.

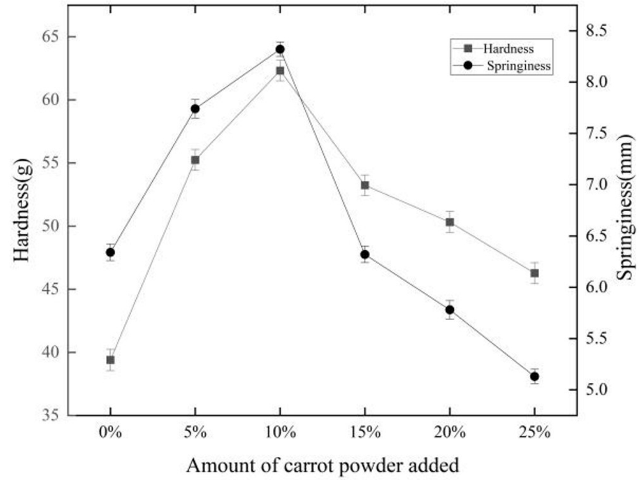

Figure 3. Effect of carrot powder on hardness and elasticity of PWRS gel.

3.3 Gel texture analysis of mixed system

Texture analysis is an important index for product evaluation, which can clearly reflect its physical characteristics (Graça et al., 2018).When the addition amount of carrot powder was less than 10%, the gel hardness of the mixed system increases, indicating that the gel strength was enhanced. The elasticity also increased gradually with the addition of carrot powder, which could result in the molecular interaction between carrot powder and PWRS, which leads to the improvement of their cross linking degree and the enhancement of gel network structure (Figure 3). The results showed that adding proper amount of carrot powder into PWRS could form stronger network structure, and the gel structure of the mixed system was more stable and difficult to deform. When the amount of carrot powder was more than 10%, the gel structure was difficult to form, so its hardness and elasticity also decreased.

3.4 Changes in dynamic rheology

Dynamic rheological properties mainly include storage modulus (G') and loss modulus (G"), which are mainly used to analyze the change rule of elasticity and viscosity of starch gel, also known as elastic modulus and viscosity modulus (Sandhu et al., 2020). That both elastic modulus and viscous modulus showed a rising trend with the increase of frequency and do not intersect each other (Figure 4). When the content of carrot powder was less than 10%, the tangent value of loss Angle was less than 1. The tangent value of loss Angle was the ratio of the viscous modulus to the elastic modulus, indicating that the elastic modulus was larger than the viscous modulus, and the solid properties were enhanced. When the content of carrot powder was more than 10%, the tangent value of loss Angle was greater than 1, indicating that the solid property of starch gel was weakened and the liquid property was enhanced. With the addition of carrot powder, the tangent value of loss Angle increased obviously, the viscosity continued to increase, and the structure of starch gel gradually changed to liquid, which was consistent with the change of texture.

3.5 Freeze-thaw stability analysis

Freeze-thaw stability is one of the important characteristics in the food industry. After continuous freezing and melting, gelatinized starch molecules are rearranged, and the bulk phase water of starch gel is released from the polymer network structure to form a spongy structure, which is called dehydration condensation (Mei et al., 2021). Dehydration condensation rate, also known as water extraction rate, is an important parameter to evaluate the stability of gel system. Dehydration condensation gradually increased in the first three freezing-thawing cycles, while remained unchanged in the last two freezing-thawing cycles shown in Figure 5. Dehydration condensation decreased when carrot powder content increased in a single cycle. The results could be attributed to the synergistic effect between carrot powder and PWRS, thus improving the gel rigidity. In the freezing and thawing process, the water separation rate from gelatinized starch was lower and lower, so carrot powder could improve the dehydration condensation of glutinous rice starch.

3.6 Microstructure analysis

The microstructure of gels mixed with different proportions after gelatinization and freeze drying was shown in Figure 6. The microstructure of PWRS gels is reticular or honeycomb (Aleixandre et al., 2021). With the increase of the amount of carrot powder, the pores in the gel microstructure continued to increase, and the pores were formed by the water molecules discharged from the samples after freeze-drying. It can be speculated that the water holding capacity of PWRS gel increased after the addition of carrot powder, and this property was strengthened with the increase of carrot powder. This result would be due to the influence of carrot powder as a high dietary fiber raw material, which contains a lot of cellulose and has a strong water absorption.

3.7 Changes in the state of water

The binding of water and matrix in low field NMR is reflected by longitudinal relaxation time (T_1) and transverse relaxation time (T_2). The shorter the transverse relaxation time (T_2) is, the

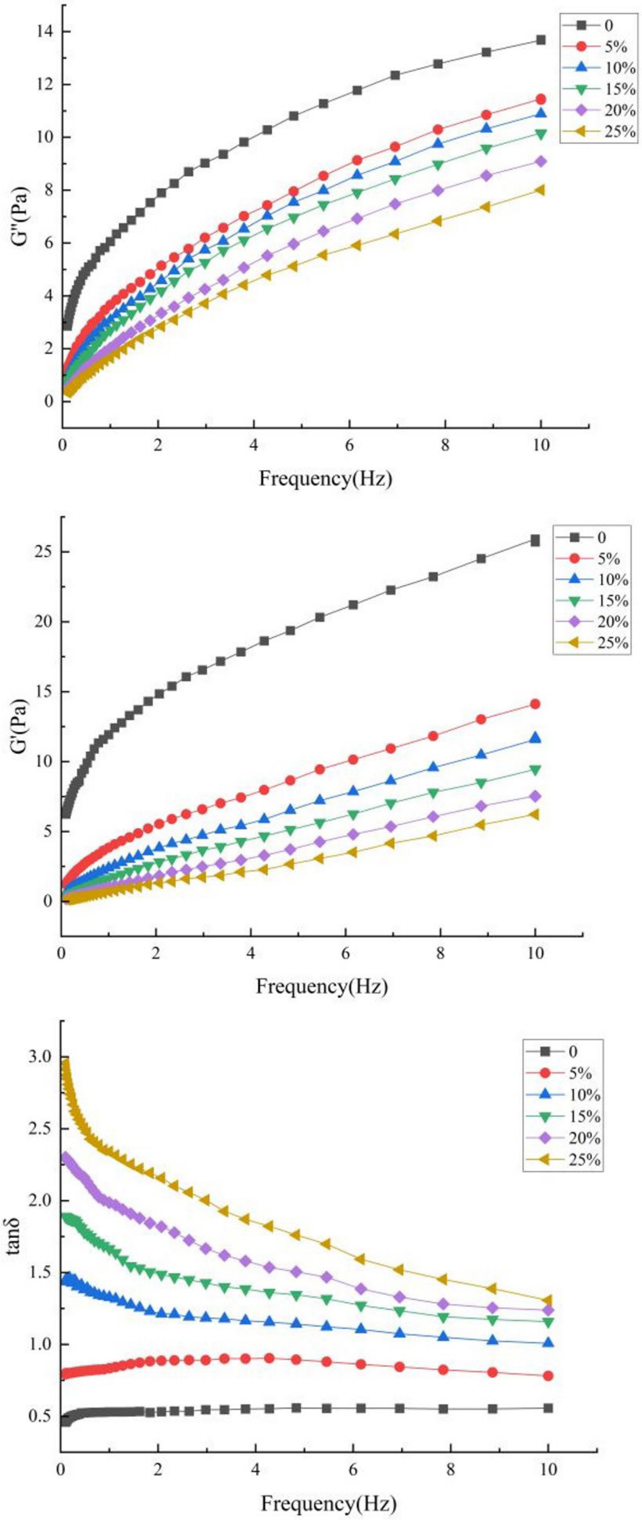

Figure 4. Effect of carrot powder on rheological properties of PWRS gel.

better the combination of water and matrix is, and the stronger the water holding capacity is. Table 2 presented the moisture state parameters of PWRS gel after adding different carrot powder. There were three kinds of moisture with different fluidity in T_2 of gels mixed with different proportion in certain relaxation

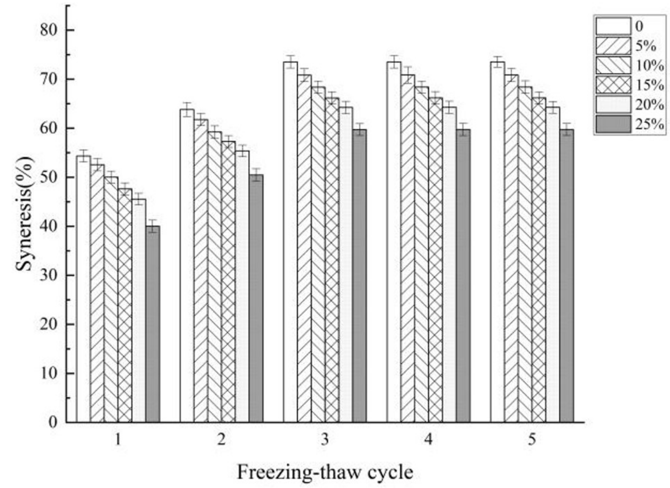

Figure 5. Effect of carrot powder on dehydration condensation rate of PWRS gel.

time. The mobility of T_{21} system was the weakest, because of the strongly bound water. T_{22} was characterized by weakly bound water with moderate fluidity. Free water was the most liquid in the T_{23} characterization system. A_{21}, A_{22} and A_{23} represented the contents of strongly bound water, weakly bound water and free water, respectively. According to the results in the table, T_{21} and T_{22} showed no significant change after the addition of carrot powder, while T_{23} showed a downward trend, A_{21} showed no significant change, A_{22} gradually increased, and A_{23} gradually decreased. It may be that the interaction between carrot powder and PWRS molecules improved the binding ability of PWRS matrix to water, resulting in the increase of weak binding water content and the decrease of free water content.

3.8 Sensory characteristics of PWRS gel

Different contents of carrot powder had different effects on the color, smell, taste, tissue morphology, elasticity and overall acceptability of gel products (Figure 7).When the content of carrot powder increased from 0 to 25%, the color, smell and taste scores of the samples increased, which may be due to the bright color and sweet taste of carrot powder. In addition, the tissue morphology, elasticity, and the overall acceptability score lowered after rising first, within the scope of this study. When the carrot powder content was 10%, there was three highest scores for tissue morphology, elasticity, and the overall acceptability. This was perhaps related to rich cellulose and high water absorbability of carrot powder. While carrot powder content was too high, gel structure was too difficult to shape. Therefore, adding appropriate carrot powder could improve the sensory quality of PWRS gel.

4 Discussion and conclusion

The aging of PWRS is mainly due to the reduction of the temperature of starch paste after gelatinization and the reaggregation of disordered starch molecules into ordered crystalline structure. Its aging is affected by a variety of factors, and gels with a certain network structure can be formed after

Table 2. Effect of carrot powder on water parameters of pre-gelatinized glutinous rice starch gel.

Amount of carrot powder added	T_{21}	T_{22}	T_{23}	A_{21}	A_{22}	A_{23}
0	0.52 ± 0.04^{d}	10.75 ± 0.52^{e}	285.42 ± 3.42^{a}	6.43 ± 0.26^{a}	4.56 ± 0.16^{f}	112.84 ± 0.46^{a}
5%	0.53 ± 0.01^{c}	10.76 ± 0.58^{e}	284.32 ± 3.15^{b}	6.25 ± 0.38^{b}	4.85 ± 0.08^{e}	112.21 ± 0.53^{b}
10%	0.54 ± 0.02^{b}	10.83 ± 0.62^{d}	282.18 ± 3.26^{c}	6.08 ± 0.32^{c}	5.37 ± 0.05^{d}	110.47 ± 0.38^{c}
15%	0.54 ± 0.08^{b}	11.14 ± 0.71^{c}	281.63 ± 3.57^{d}	5.76 ± 0.18^{e}	5.79 ± 0.16^{c}	109.73 ± 0.48^{d}
20%	0.55 ± 0.03^{a}	11.68 ± 0.65^{b}	280.27 ± 3.31^{e}	5.87 ± 0.29^{d}	6.25 ± 0.07^{b}	108.42 ± 0.54^{e}
25%	0.53 ± 0.01^{c}	12.09 ± 0.82^{a}	279.29 ± 3.05^{f}	5.71 ± 0.24^{f}	6.39 ± 0.12^{a}	107.29 ± 0.36^{f}

Values are mean±standard error, different letters in the same column mean significant difference ($P < 0.05$), [a]is the maximum.

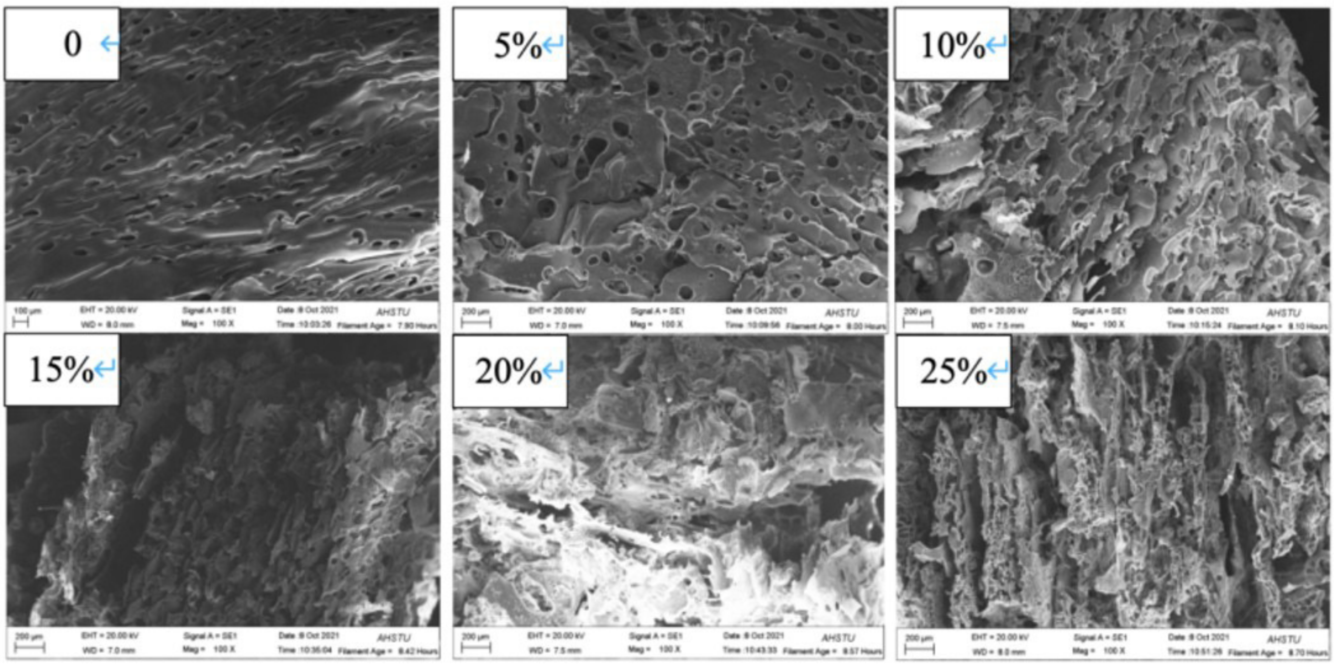

Figure 6. Effect of carrot powder on microstructure of PWRS gel.

aging and rejuvenation, whose texture qualities such as hardness, elasticity and chewiness have an important impact on the food quality of related products (Wongsa & Aichayawanich, 2020). At present, it is widely believed that the aging kinetics of starch can be simulated by Avrami equation. The aging kinetics of starch is divided into two dynamic changes: the rapid gelation of amylose and the formation of a double helix, which makes it irreversible crystallization; The short chains in amylopectin recrystallized at a slower rate than the amylose glue. Amylopectin is a high-branching structure with orderly molecular arrangement and stable hydrogen bond. Compared with amylose, the aging rate of amylose is significantly slower than that of amylose.

Starch aging is affected by a variety of factors, such as amylose and amylopectin, protein, moisture, sugars, lipids, salts, storage temperature, pH, etc (Huang et al., 2021). Food enterprises are faced with the problems of product aging and loss of commodity value in the process of starch food. Therefore, the law of starch aging has great research significance. Studies on the aging properties of glutinous rice starch at home and abroad mainly focus on the molecular chain structure, average chain length, water migration rate and distribution of amylopectin (Li et al., 2021), but a mature aging inhibition mechanism has not been cleared, mainly due to the numerous factors affecting the aging of starch, unknown process and complex system (Santos et al., 2020).

Our results showed that adding carrot powder in PWRS dilution effect of amylose content, its crystal structure has not changed, in addition, the carrot powder add from 0 to 10%, the content of carrot powder increased PWRS gel hardness and elasticity, viscous modulus and elastic modulus are on the rise and loss tangent value greater than 1, the solid properties, In the freeze-thaw cycle, the dehydration and condensation rate also decreased, the pores in the microstructure increased, and the water holding capacity increased, which resulted in changes in the combination of water and matrix in the mixed system, and the content of weakly bound water increased. The sensory properties of PWRS gel could also be improved with appropriate carrot powder. In conclusion, the addition of carrot powder in PWRS can improve the characteristics of PWRS, which may help delay the aging of glutinous rice products, and provide theoretical support for the product innovation and development of food enterprises.

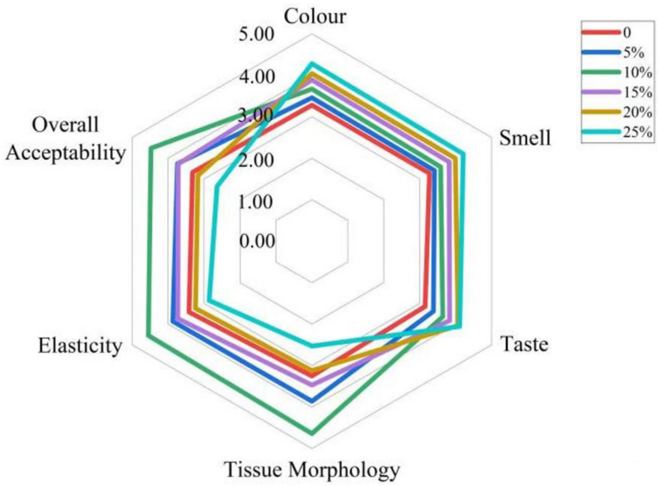

Figure 7. Sensory properties radar map.

Acknowledgements

This work was supported by the Science Research Program of Anhui University of Science and Technology (2021XCZX11) and the Innovation and Entrepreneurship Training Program of Anhui University Students (S202110879122).

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