

# Starch modification by tea polyphenols under ultrasonic power control: Digestive behavior and physicochemical characteristics

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## Research Article

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# Starch modification by tea polyphenols under ultrasonic power control: Digestive behavior and physicochemical characteristics

## ABSTRACT

Starch is the primary dietary carbohydrate, but excessive rapidly digestible starch (RDS) can cause sharp postprandial glycemic spikes. In this work, yam starch (YS)-tea polyphenol (TP) complexes were fabricated via ultrasonic treatment at gradient power levels (96, 192, 288, 384, and 480 W). The results demonstrated that ultrasonication significantly elevated the complexation index (CI) between YS and TP. Microstructural observation revealed that the obtained complexes exhibited irregular fragmented morphology with abundant porous structures, which facilitated the non-covalent embedding of TP molecules into the starch matrix. X-ray diffraction analysis confirmed that ultrasonic treatment promoted the formation of V-type inclusion complexes. Notably, the 288 W ultrasonic treatment produced the maximum relative crystallinity of 33.7%, and remarkably improved the thermal stability of the YS-TP complexes. Under this optimal 288 W condition, the resistant starch (RS) content increased from 30% in native yam starch to 38%, while the estimated glycemic index (eGI) decreased to 41.04. Enzyme activity assays indicated that the digestion-delaying mechanism of the complexes was primarily attributed to the reduced accessibility of digestive enzymes to starch substrates. Collectively, ultrasonication is validated as an efficient strategy for the scalable preparation of YS-TP complexes, which effectively attenuates postprandial glycemic response. This study provides a theoretical foundation for the development of high-loading functional starch-based materials.

**Keywords** Ultrasound Starch Tea polyphenols *In vitro* digestion

## Introduction

Starch, as the dominant dietary carbohydrate in human diet, provides sustained metabolic energy and maintains postprandial glucose homeostasis (Liu et al., 2026). Based on its digestibility, starch is generally categorized into rapidly digestible starch (RDS), slowly digestible starch (SDS), and resistant starch (RS). Nevertheless, excessive proportion of RDS in starch may trigger abrupt postprandial blood glucose spikes, leading to the increased risk of chronic metabolic disorders including type 2 diabetes and obesity (Kusumawardani et al., 2026). Numerous studies have confirmed that starch digestibility is primarily governed by its multi-scale structural features, which act as the core determinant mediating the

interaction and catalytic efficiency between starch and digestive enzymes. In this context, tailoring the multi-scale architecture of starch through starch modification, including physical, chemical, enzymatic and genetic treatments, has been considered as an effective strategy for reducing starch digestibility and modulating postprandial glycemic response (He et al., 2025).

Yam (*Dioscorea opposita Thunb.*) is considered the fourth largest tuber crop after potatoes, cassava, and sweet potatoes (Chen et al., 2026). Yam is rich in bioactive ingredients such as starch, dietary fiber, protein, vitamins, minerals, saponins and flavonoids. Among various starch sources, tiegun yam starch (YS) has attracted significant attention due to its unique health-promoting properties, including antioxidant, qi-invigorating, antihypertensive, and spleen-strengthening effects (Sahoo et al., 2023; Yuan et al., 2025). However, due to the poor physicochemical performance and inferior processing properties of natural yam starch, its functional, nutritional and physicochemical properties are still underutilized and underdeveloped (Yuan et al., 2025). In order to address these limitations and improve the functional properties of yam starch in food processing applications, modification techniques are commonly applied before practical use. Physical modification methods such as preparing complexes of starch and small guest molecules (including polyphenols, fatty acids, iodine) are safe, environmentally friendly and low-cost. As a practical method to improve the applicability of starch, it has received increasing attention (Xu et al., 2026).

TPs are polyhydroxy phenolic compounds extracted from tea leaves (Du et al., 2027). Accumulating studies have demonstrated that TPs exhibit multiple physiological benefits including antioxidant, anti-inflammatory, and anticancer properties (Zhu et al., 2025). However, the active groups in TPs are sensitive to environmental conditions such as oxygen, light, temperature, and pH, leading to the loss of functional activities and posing significant challenges in practical applications. In order to improve the stability of polyphenols and maximize their efficacy in functional foods, researchers typically use food grade carriers such as starch matrices to load TPs. In addition, the interaction between starch and TPs may affect the digestive, physicochemical and functional characteristics of starch. As a functional additive, TPs have been applied in starch modification. Such modification results in a single, left-handed helical inclusion complex termed V-type amylose; or a non-inclusive complex with weak binding forces mainly formed through hydrogen bonds (Li et al., 2026). Recent studies have found that starch can be modified by complexing with polyphenols to regulate hydrolysis and produce low glycemic index foods

(Kunyanee et al., 2022). However, the outcomes of these interactions and their effects on the starch properties are influenced by the specific processing techniques used in the formulation process as well as the structures of starch and polyphenols (Liu et al., 2025). Ultrasonic modification is an environmentally friendly and efficient non-thermal processing technology, which is increasingly being applied in the food industry. Besides, ultrasonication can cause cavitation effects, generating strong shear forces, high temperatures, and free radicals in the reaction medium (Xu et al., 2022; Rahaman et al., 2021). The substantial activation energy generated by the reaction can destroy the structure of starch, causing voids and cracks in starch granules, leading to the partial decomposition of starch granules. In addition, the influence of ultrasonic waves could also form pitting marks on the surface of starch granules, resulting in changes in the thermal stability, decomposition and gel properties of the granules (Xu et al., 2021; Li et al., 2021). This mechanochemical effect effectively destroys the structure of starch molecules and enhances their reactivity with other compounds. Xu *et al.* (2023) have systematically investigated the complexes made from three starches with different typical crystalline structures (corn, potato and pea) and rose polyphenols by ultrasonication with different power, established a positive correlation between ultrasonic power and complexing index of starch and polyphenols. Besides, researchers have revealed synergistic effects between ultrasonication and freeze-thaw treatment during the complexation of rice starch with polyphenols (Luangsakul et al., 2025). They further illustrated that ultrasonic treatment promoted the formation of V-type inclusion complexation and reduced the hydrolysis rate of starch. In recent years, green technology has become a promising strategy to prepare starch-polyphenol complexes. Till now, many studies have been reported on modified starch. However, systematic investigation is still needed on the starch modification method for preparing starch-polyphenol complexes by ultrasonic treatment. Thus, this study investigated the effect of varying ultrasonic power levels on the preparation of complexes between YS and TPs, specifically focusing on the alteration of complexation index, water hydration properties, crystal structure, microstructure and thermal properties. Besides, dynamic glucose release, polyphenol release and enzyme activity during *in vitro* digestion were also studied in this research. This study provided a theoretical basis for starch modification, which helped to provide more dietary choices for sugar control populations. In addition, it provided a theoretical and practical basis for the further development and high-value conversion of YS and TPs in food industry.

## **Materials and Methods**

### **Raw materials**

Tiegun yam was purchased from Jiaozuo Huaiputang Biotechnology Co., Ltd. (Jiaozuo, China); Tea polyphenols, absolute ethanol, sodium hydroxide and sodium acetate were purchased from China National Pharmaceutical Group Chemical Reagent Co., Ltd. (Shanghai, China); Folin-Ciocalteu reagent was obtained from Shanghai Macklin Biochemical Co., Ltd (Shanghai, China); Porcine pancreatic  $\alpha$ -amylase (CAS 9000-90-2, activity 9.4 U/mg), amyloglucosidase amylase (CAS 9032-08-0, activity 100 U/mg), 3, 5-dinitrosalicylic acid (DNS) were purchased from Shanghai Yuanye Biotechnology Co., Ltd. (Shanghai, China). All reagents used in this study were analytical grade.

### **Preparation of yam starch**

Native YS was extracted from fresh Tiegun yam tubers according to the method reported by Xu et al. (2026) with some modifications. Briefly, fresh yam tubers were washed, peeled and sliced. Then deionized water was added and homogenized with a crusher to obtain yam homogenate. The homogenate was passed through a 100-mesh sieve, and the collected filtrate was allowed to settle for 4 h at 25 °C, after which the supernatant was carefully discarded. A 0.2% (w/v) sodium hydroxide solution was added to the precipitate, and the pH of the mixture was adjusted to 9.0. After thorough magnetic stirring for 30 min, the mixture was allowed to settle completely, and the supernatant was removed. The resulting precipitate was repeatedly washed with deionized water no less than 3 times, and then washed with absolute ethanol no less than 2 times. Finally, the purified starch precipitate was collected and dried in a convection oven at 45 °C for 24 h. The dried starch was gently ground, and then passed through a 100-mesh sieve to obtain the final YS sample for subsequent modification experiments.

### **Preparation of yam starch-tea polyphenols (YS-TP) complexes**

2.0 g of YS was dispersed in 20 mL deionized water and stirred continuously at 95 °C for 30 min in a constant temperature magnetic stirrer for gelatinization. Next, treat the starch paste at 50 °C under ultrasound conditions with gradient power levels of 96 W, 192 W, 288 W, 384 W, and 480 W for 30 minutes. Subsequently, tea polyphenols (30%, w/w) were added and vortexed to mix evenly. The resulting mixture was further stirred in a water bath at 80 °C for 30 min. After the above treatments, the reaction system was centrifuged at 10,000 rpm for 10 min and dried in a 45 °C oven for 24 h. After gentle grinding, the obtained solid was passed through a 100-mesh sieve. The ultrasonic-treated samples were

labeled as YS-TP-U96, YS-TP-U192, YS-TP-U288, YS-TP-U384, and YS-TP-U480, corresponding to the ultrasonic power of 96 W, 192 W, 288 W, 384 W, and 480 W, respectively. The starch-polyphenol physical mixture prepared without ultrasonic treatment was labeled as YS-TP and set as the blank control group.

#### **Complexation index (CI)**

The CI of YS and TPs were calculated according to the method of Kusumawardani et al. (2026) with some modifications. Briefly, 0.1 mL of the reaction supernatant was diluted with 0.4 mL deionized water. Then, 0.1 mL of the dilution was mixed with 1 mL of tenfold-diluted Folin-Ciocalteu reagent and incubated in the dark for 6 min. Subsequently, 1 mL of 6% (w/v) sodium carbonate solution was added, and the mixture was allowed to stand in the dark for 1 h. The absorbance was finally measured at 765 nm. The CI value was calculated based on the gallic acid standard curve.

#### **Water hydration properties**

The water solubility (WS), swelling power (SP), and water absorption index (WAI) of the samples were according to the method of Liu et al. (2023) with some modifications. Briefly, 0.2 g of sample was weighted and mixed with 10 mL of deionized water. The suspension was magnetically stirred at 95 °C for 30 min. After cooling to room temperature, the mixture was centrifuged at 5000 rpm for 15 min. The supernatant was collected and dried to a constant weight. The dry mass of the soluble fraction was recorded, and the wet weight of the remaining sediment was also weighed simultaneously. The three indices were calculated according to the following formula:

$$\text{Water solubility (\%)} = \frac{\text{dry supernatant(g)}}{\text{dry sample(g)}} \times 100\% \quad (1)$$

$$\text{Swelling power (g/g)} = \frac{\text{wet sediment(g)}}{\text{dry sample(g)} - \text{dry supernatant(g)}} \quad (2)$$

$$\text{Water absorption index (g/g)} = \frac{\text{wet sediment(g)}}{\text{dry sample(g)}} \quad (3)$$

#### **Scanning electron microscopy (SEM)**

The SEM analysis was calculated according to the method of Luo et al. (2022) with some modifications. Dried starch powder was mounted on the conductive carbon adhesive tape affixed to the SEM specimen stub. Loose floating powder on the sample surface was gently blown off using a rubber bulb, and the sample was subsequently sputter-coated with a 5-10 nm thin gold layer. The microstructure of the

prepared samples was then observed under a ZEISS Sigma 360 field-emission scanning electron microscope at an accelerating voltage of 15 kV.

### **X-ray diffraction (X-RD)**

The crystal structure of starch samples was characterized on an X-ray diffractometer (Ultima IV, Rigaku Corporation, Japan) with Cu K $\alpha$  radiation. The operating conditions were set at 30 kV and 20 mA, with a 2 $\theta$  scanning range of 5-35 ° and a scanning speed of 4 °/min (Wang et al., 2024).

### **Thermal properties**

Thermal properties were measured using a differential scanning calorimeter (DSC 214 Polyma, NETZSCH-Gerätebau GmbH, Germany) (Raza et al., 2023). Each sample (3.0 mg, dry basis) was mixed with deionized water (1:2, w/v), sealed, and equilibrated at 4 °C for 24 h. Scans were performed from 30 to 120 °C at 10 °C/min under N<sub>2</sub> flow, using an empty sealed pan as reference. The onset (To), peak (Tp), and conclusion (Tc) temperatures, as well as the gelatinization enthalpy ( $\Delta H$ ), were recorded.

### **Digestive properties**

#### **Simulated *in vitro* digestion experiments (RDS, SDS, RS, and eGI)**

The *in vitro* digestion experiments were calculated according to the method of Yao et al. (2023) and Goñi et al. (1997) with some modifications. Briefly, 400 mg of YS-TP complex was mixed with 3 mL sodium acetate buffer (pH 5.2), 2 mL of freshly prepared mixed enzyme solution (308.5 mg  $\alpha$ -amylase and 10.0 mg amyloglucosidase, dissolved in 10 mL deionized water, pre-incubated at 37 °C for 10 min and filtered), and 6 glass beads in a 37 °C thermostatic oscillator. At 0, 20, 40, 60, 90, 120, 150, and 180 min, 0.2 mL of the digest was withdrawn and immediately mixed with 1.0 mL absolute ethanol to terminate the reaction. After centrifugation at 5000 rpm for 5 min, 0.25 mL of the supernatant was diluted with 1 mL deionized water, mixed with 2 mL DNS reagent, and vortexed. The mixture was boiled for 5 min, then rapidly cooled in an ice-water bath. After dilution with 9 mL deionized water, the absorbance was measured at 540 nm using a double-beam UV-Vis spectrophotometer (UV-1901, Persee General Instrument Co., Ltd., Beijing, China).

$$\text{Starch digested ratio} = \frac{G_t \times 0.9}{m} \times 100\% \quad (4)$$

$$\text{RDS} = \frac{(G_{20} - FG) \times 0.9}{TS} \times 100\% \quad (5)$$

$$196 \quad \text{SDS} = \frac{(G_{120} - G_{20}) \times 0.9}{\text{TS}} \times 100\% \quad (6)$$

$$197 \quad \text{RS} = 1 - \text{RDS} - \text{SDS} \quad (7)$$

$$198 \quad \text{HI} = \frac{S}{S_{\text{wb}}} \times 100\% \quad (8)$$

$$199 \quad \text{eGI} = 0.862 \times \text{HI} + 8.198 \quad (9)$$

200 where  $G_t$  is the glucose content at time  $t$  (mg), 0.9 is the conversion factor,  $m$  is the mass of starch (mg),  
 201  $FG$  is the glucose content (mg) in the sample before digestion,  $TS$  is the content of dry starch in the  
 202 sample (mg),  $S$  is the area under the hydrolysis curve,  $S_{\text{wb}}$  is the area under the hydrolysis curve of white  
 203 bread.

#### 204 **Release of TPs during digestion**

205 At predetermined time points (0, 20, 40, 60, 90, 120, 150, and 180 min), 0.5 mL of the digestive solution  
 206 was withdrawn and centrifuged at 5000 rpm for 10 min. Then, 0.1 mL of the supernatant was collected  
 207 and diluted with 0.4 mL of deionized water. Subsequently, 0.1 mL of the diluted sample and 1 mL of  
 208 tenfold-diluted Folin-Ciocalteu reagent were mixed and incubated in the dark for 6 min. After adding 1  
 209 mL of 6% (w/v) sodium carbonate solution, the mixture was kept in the dark for 1 h, and the absorbance  
 210 was measured at 765 nm (Liu et al., 2019).

#### 211 **Total enzyme activity**

212 The total enzyme activity was calculated according to the method of Wu et al (2023) with some  
 213 modifications. During *in vitro* digestion, 0.1 mL of the digestive solution was withdrawn at 0, 20, 40, 60,  
 214 90, 120, 150, and 180 min, respectively, mixed with 0.9 mL of 1% (w/v) starch solution, and incubated  
 215 at 37 °C for 30 min. The reaction was terminated by adding 0.5 mL of absolute ethanol, followed by  
 216 centrifugation at 4 °C and 10,000 rpm for 10 min. Then, 0.25 mL of the supernatant was collected and  
 217 diluted to 1 mL with deionized water. After adding 2 mL of DNS reagent, the mixture was vortexed  
 218 thoroughly, boiled in a water bath for 5 min, and immediately cooled in an ice-water bath to room  
 219 temperature. The solution was further diluted with 9 mL of deionized water, and the absorbance was  
 220 measured at 540 nm. The system without added starch solution was set as the negative control group.

#### 221 **Statistical analysis**

222 All experimental results are presented as mean  $\pm$  standard deviation (SD) from triplicate independent  
 223 measurements. Statistical analysis was performed using SPSS 18.0 (IBM, Chicago, IL, USA). One-way

analysis of variance (ANOVA) was carried out, and significant differences among groups were evaluated by Duncan's multiple range test at a significance level of  $p < 0.05$ . All figures were plotted using Origin 2021 (OriginLab Corporation, Northampton, MA, USA).

## Results and Discussion

### Complexation index

To investigate the effect of ultrasonic treatment on the binding behavior between YS and TPs, the content of TPs in the prepared complexes was quantitatively determined via the Folin-Ciocalteu method, and the CI was calculated accordingly (Ma et al., 2023). As shown in Fig. 1, the CI of the obtained samples exhibited a significant increasing trend with the elevation of ultrasonic power. The results showed that the CI of the YS-TP complex without ultrasonic treatment was only 1.86%, while a remarkable enhancement in CI was observed after ultrasonic intervention. Notably, as ultrasonic power increased, the CI between YS and TPs followed a trend of first rising and then declining, reaching a maximum value of 37.15% at 288 W. This phenomenon could be primarily attributed to the disintegration of swollen starch granules induced by ultrasonic treatment, which effectively facilitated the intermolecular interaction between starch and small-molecule ligands (Wang et al., 2025). Nevertheless, a sharp reduction in CI was detected when ultrasonic power further exceeded this optimal level. This was because abundant hydroxyl groups are distributed on the outer surface of starch chains, and TPs can interact with starch chains through hydrogen bonding and hydrophobic interactions. Excessive ultrasonic treatment will break the weak hydrogen bonds formed between starch chains and polyphenols, thus leading to a decrease in CI. In addition, ultrasonic treatment promoted the release of more amylose molecules from starch granules, while the amylose was easily depolymerized, which reduced the formation of starch-ligand complexes (Kou et al., 2019; Carmona-Garcia et al., 2009). These findings are consistent with previous reports focusing on the effect of ultrasonic treatment on improving the complexation between starch and polyphenols. It bears mention that light ultrasonic treatment corresponds to a longer wavelength and stronger penetrating power than high-frequency sonication, so it can more effectively disrupt the molecular structure of starch, and further enable starch to bind with polyphenol extracts at a higher efficiency (Xu et al., 2023). It can be concluded from the above results that appropriate ultrasonic power can significantly increase the complexation index of starch and polyphenols, while excessive ultrasonication will produce a counterproductive effect and reduce the final complexation index.

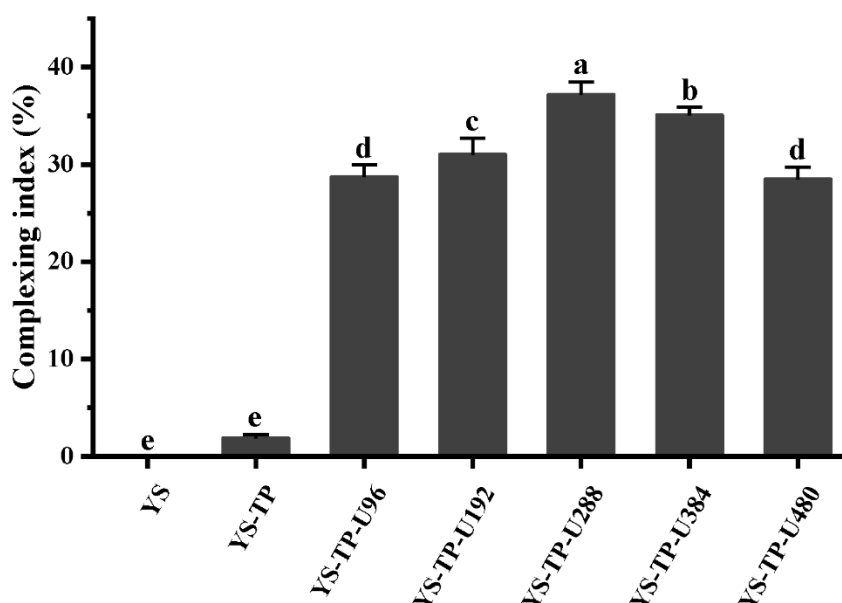


**Fig. 1** CI of YS-TP complexes following ultrasonication treatment

#### Water hydration properties

The hydration properties of starch are critical determinants of the texture and storage stability of food systems. The water solubility, swelling power and water absorption index of native YS and YS-TP complexes are shown in Table 1. Compared with the simple physically mixed YS-TP complexes, the water solubility, swelling power and water absorption index of YS-TP complexes prepared under ultrasonic power of 96–384 W decreased significantly ( $p < 0.05$ ). These reductions indicated that ultrasonic cavitation effect promoted hydrogen bonding cross-linking between TPs and YS, and consequently formed a more compact network structure in the system. This dense network effectively inhibited water penetration into starch granules and the leaching of dissolved starch molecules, thus reducing the swelling and solubility of starch. In addition, after the combined treatment of gelatinization and ultrasonication, the accessibility of YS molecules to TPs molecules were greatly improved, which facilitated the incorporation of TPs into YS matrix through non-inclusion interactions. The extensive hydrogen bonding formed between starch and polyphenols reduced the accessibility of hydroxyl groups on starch chains to water molecules (Mujtaba et al., 2022; Zhao et al., 2019b). Additionally, the hydrophobicity of certain polyphenols was attributed to their aromatic ring structures, which created hydrophobic regions and further inhibited starch-water interactions, leading to a decrease in water absorption capacity (Dai et al., 2024). However, no further significant decline in these three hydration indexes was observed when the ultrasonic power was increased to 480 W. This could be explained by the

fact that the starch-polyphenol reaction had reached a near-saturation state at 384 W, and the continuously increased ultrasonic intensity could not induce more effective binding sites to further reduce the hydration performance (Luangsakul et al., 2025).

**Table 1** The water hydration properties of YS and YS-TPs complexes

| Samples    | Water solubility (%)    | Swelling power (g/g)     | Water absorption index (g/g) |
|------------|-------------------------|--------------------------|------------------------------|
| YS         | 84.42±1.18 <sup>a</sup> | 12.84±0.20 <sup>ab</sup> | 35.02±0.24 <sup>ab</sup>     |
| YS-TP      | 87.70±1.29 <sup>a</sup> | 13.51±0.23 <sup>a</sup>  | 36.83±0.29 <sup>a</sup>      |
| YS-TP-U96  | 75.72±1.11 <sup>c</sup> | 12.10±0.18 <sup>bc</sup> | 32.72±0.26 <sup>c</sup>      |
| YS-TP-U192 | 73.47±1.07 <sup>c</sup> | 11.81±0.17 <sup>c</sup>  | 31.95±0.25 <sup>c</sup>      |
| YS-TP-U288 | 75.37±1.16 <sup>c</sup> | 12.33±0.19 <sup>bc</sup> | 33.36±0.27 <sup>bc</sup>     |
| YS-TP-U384 | 80.38±1.19 <sup>b</sup> | 11.94±0.18 <sup>c</sup>  | 32.28±0.25 <sup>c</sup>      |
| YS-TP-U480 | 89.06±1.33 <sup>a</sup> | 13.37±0.22 <sup>a</sup>  | 36.34±0.29 <sup>a</sup>      |

Significant differences within the same column are indicated by different superscripts ( $p < 0.05$ , based on Duncan's test)

### Scanning electron microscopy (SEM)

The SEM results of YS and YS-TP complexes after different power ultrasonic treatments are shown in Fig. 2. YS granules were elliptical or nearly spherical in shape, with regular morphology, smooth surface without wrinkles or pores, and uniform particle size. After gelatinization, the inherent morphology of starch granules was completely disrupted, with a significant increase in surface roughness and wrinkle density. Starch granules adhered and aggregated into irregular blocks, and the structure tended to become denser. The interaction between TPs and YS further induced the reorganization of particle structure. After ultrasonic treatment, the composite showed a fragmented irregular morphology with abundant porous structures. This phenomenon was primarily attributed to cavitation-generated shear forces, which enhanced molecular mobility and caused partial disruption of the granule outer surface (Wei et al., 2019). Such structural features facilitated the penetration of polyphenol molecules, enabling partial embedding of TPs into the inner lumen of starch chains via non-covalent interactions and ultimately improving the binding efficiency between polyphenols and starch (Zhao et al., 2019a). Under low-frequency ultrasonic treatment of 96 W and 192 W, the obtained complexes presented a loose structure with weak interparticle adhesion, and low-power ultrasonication mainly exerted a crushing effect on starch granules. When the ultrasonic power was increased to 288 W, the compactness of the composite aggregate was remarkably improved, the number of surface pores decreased, and the intermolecular cross-linking became more

compact. An appropriate ultrasonic power could promote the stretching of starch molecular chains, which was conducive to the sufficient binding between YS and TPs. Under high-power ultrasonication at 384 W and 480 W, the composite structure became loose again, with intensified particle fragmentation and a rough, highly porous surface. Excessive ultrasonic cavitation effect tended to damage the pre-formed composite structure and reduced its binding stability. The final results demonstrated that ultrasonication could significantly modify the microstructure of YS-TP complexes. Ultrasonic power directly determined the compactness and orderliness of the composite structure, while both excessively low and excessively high ultrasonic treatments led to a loose and fragmented morphology. A suitable ultrasonic power was critical for the construction of a well-organized and compact YS-TP composite structure.

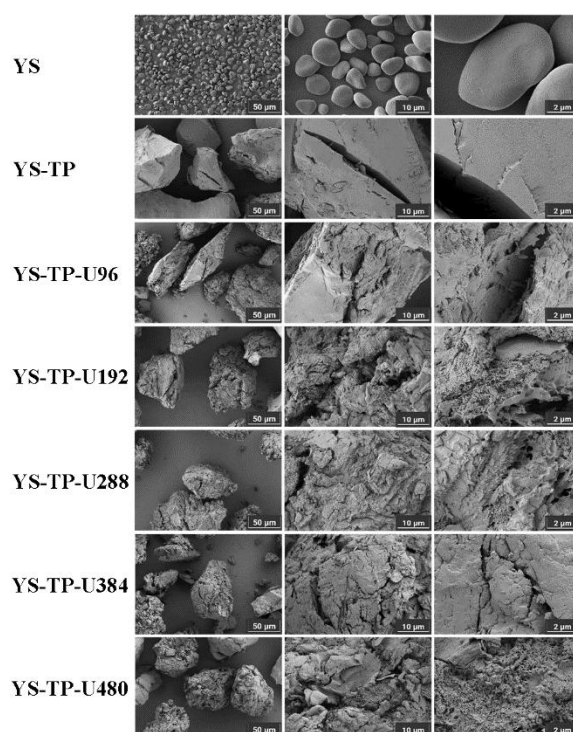


**Fig. 2** SEM images of YS and YS-TP complexes under ultrasonication treatments

### **X-ray diffraction (XRD)**

The XRD patterns of YS-TP complexes under different ultrasonic treatments are shown in Fig. 3. Native YS exhibited typical A-type crystalline peaks at  $15^\circ$ ,  $17^\circ$ , and  $18^\circ$ , with a relative crystallinity of 28.0 %. In A-type starches, polyphenols generally increase crystallinity via hydrogen bonding (Yongxing Li 2026). The typical diffraction peaks of YS disappeared after thermal gelatinization and subsequent complexation with tea polyphenols. YS and YS-TP complexes treated after ultrasonication showed new distinct diffraction signals near  $7^\circ$ ,  $13^\circ$ ,  $17^\circ$  and  $20^\circ$ . This suggested that the ordered crystalline structure

of starch was irreversibly destroyed, resulting in the disappearance of the original intense diffraction peaks of native YS. Notably, diffraction peaks at 7°, 13°, 17° and 20° in the XRD spectrum were the characteristic feature peaks of V-type inclusion complex, which provides unambiguous evidence for the successful formation of V-shaped starch-polyphenol inclusion structures (Wang et al., 2024; Zhao et al., 2019a; Chi et al., 2022). This result further implied that the orientation and rearrangement of the starch chains of YS and TPs were promoted by TPs, driving the TPs molecules to bind with YS in the form of V-type inclusion complexes (Qin et al., 2025). As the ultrasonic power increased from 96 W to 288 W, the crystallinity of the samples increased from 30.8% to 38.9%. However, further increasing ultrasonic power beyond 288 W induced a declining trend in crystallinity, and the crystallinity value dropped back to 28.3% when the ultrasonic power reached 480 W. This trend is highly consistent with the findings reported by Zhang *et al.* (2020), who demonstrated that appropriate ultrasonic treatment favoured the ordered arrangement of starch molecules, while excessive ultrasonic intensity disrupted pre-formed crystalline structures. Our experimental results were consistent well with this mechanism, verifying that moderate ultrasonic power promoted the growth of V-type crystals and improves overall crystallinity, whereas over-high ultrasonic power damaged the already established ordered crystalline architecture. Notably, the YS-TP-U288 sample possessed the highest crystallinity (38.9%) among all groups, which was strongly correlated with its highest resistant starch content observed in the digestion assay. This close correlation clearly indicated that a more intact and well-organized V-type crystalline structure conferred the starch-polyphenol complexes with stronger anti-digestion performance.

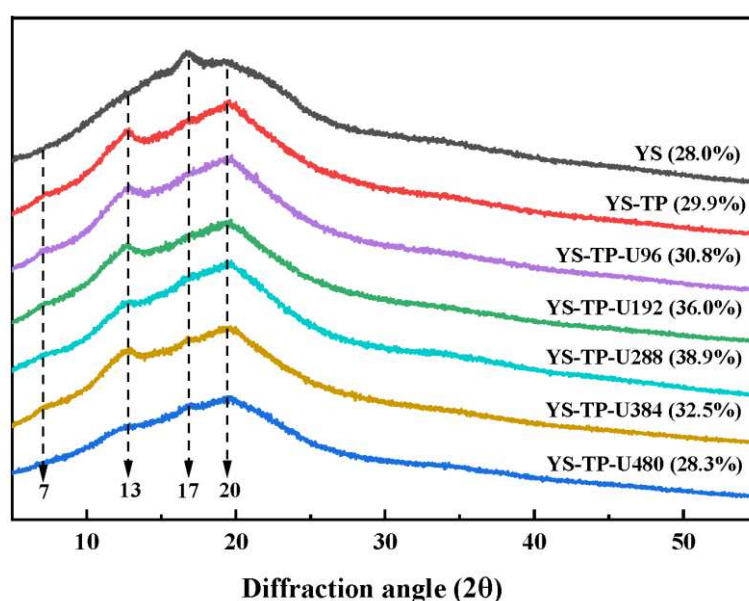


**Fig. 3** X-ray diffraction patterns of YS and YS-TP complexes under ultrasonication treatments

### ***In vitro* digestion characterization**

The *in vitro* digestion characterization of YS and YS-TP complexes treated after ultrasonication is presented in Fig. 4. Specifically, the hydrolysis rates of YS and YS-TP complexes increased rapidly in a short period of time, gradually leveling off until reaching equilibrium (Fig. 4A). TPs incorporation effectively inhibited starch hydrolysis, and this anti-digestive effect was further pronounced after ultrasonic processing. The eGI of native YS was measured at 49.81, which belongs to low GI food (Zhang et al., 2026). Polyphenol addition alone led to a marginal, non-significant eGI reduction ( $p > 0.05$ ), while ultrasonic treatment induced a significant eGI drop in the YS-TP complexes. This observation confirms that the formation of stable V-type inclusion complexes substantially reduces the enzymatic accessibility of starch chains, which aligns well with the findings reported in previous studies (Zhao et al., 2019a). When the ultrasonic power reached 288W, the eGI reached the lowest level of 41.04, which was 17.60% lower than that of YS (Fig. 4C). According to the Englyst method, the contents of RDS, SDS, and RS across all samples were calculated, and the results are shown in (Fig. 4B). The RDS content of untreated YS-TP was 44%, which decreased significantly to 43%, 36%, 31%, 36% and 39% after 96–480 W ultrasonic exposure, respectively. Meanwhile, the baseline RS content of YS-TP was 11%, which increased by 24%, 22%, 29%, 15% and 28% under the 96–480 W ultrasonic regimes. Notably, the 288 W treatment group yielded the lowest RDS content and the highest RS fraction among all tested conditions. Compared with native YS, both the TP-complexed samples and all post-ultrasonication complexes exhibited remarkably enhanced anti-digestion properties. This suggests that ultrasonic treatment can modify the molecular structure of starch and induce compact rearrangement of double-helix structures within starch granules, thereby lowering the overall starch hydrolysis rate and retarding amylopectin hydrolysis kinetics (Xu et al., 2021; Hasek et al., 2020). Additionally, the entrapped polyphenols exerted a steric shielding effect that protected starch molecules from digestive enzyme attack, creating a synergistic inhibition on the digestion behaviour of polyphenol-starch binary complexes (Xu et al., 2021). Most notably, the highest RS percentage was recorded at 288 W ultrasonic power. These results collectively demonstrated that a moderate ultrasonic intensity (288 W) could effectively promoted the intermolecular interactions between starch and polyphenols, facilitated the formation of more enzyme-resistant ordered structures, and consequently drove a significant elevation in RS proportion.

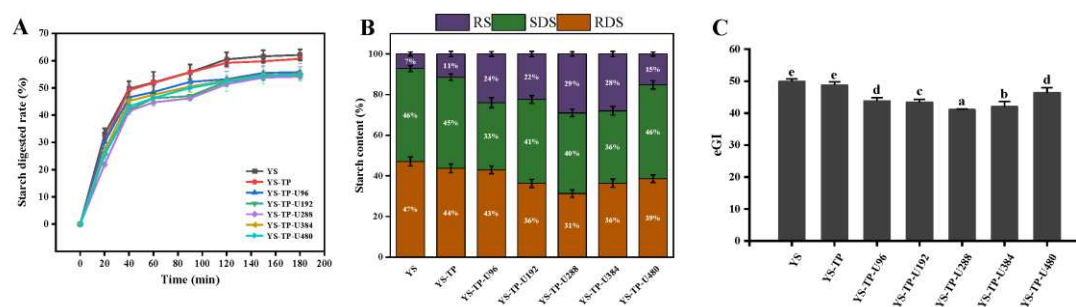


**Fig.4** *In vitro* digestion characterization of YS and YS-TP complexes under ultrasonication treatments.

**A:** Starch digestion curves; **B:** Starch fractions; **C:** Estimated glycemic index (eGI)

### Release of TPs during digestion

The mechanism of YS-TP complexes that significantly delayed the digestion of YS might due to the formation of V-type inclusion complexes between YS and TPs. Meanwhile, the polyphenols gradually released from the complexes during digestion may also inhibit the activity of digestive enzymes (Zhang et al., 2026). To further confirm its mechanism, we investigated the dynamic release behavior of TPs from the YS-TP system during *in vitro* digestion. The TPs release of YS-TP complexes under different ultrasonic treatments during digestion are shown in Fig. 5. It was noted that all YS-TP complexes exhibited a rapid release pattern in the initial 0–40 min and a gradual sustained release pattern in the subsequent digestion stage. This observation is consistent with the previous finding that TPs addition reduced the relative crystallinity of YS (Section 3.4), which weakened the overall structural ordering of the starch granule and thereby potentially facilitated the migration and release of bound TPs. The complex without ultrasonic treatment (YS-TP) showed the fastest release rate within 0-40 minutes, reaching a TPs release amount of 52 mg GAE/g starch at 40 min, and then plateaued at 56–58 mg GAE/g for the remainder of digestion. However, ultrasonication significantly altered the release behavior of TPs in a power-dependent manner ( $p < 0.05$ ). The cumulative TPs release of YS-TP-U288 during 40-120 min (76.0-79.5 mg GAE/g) was moderately higher than that of the native YS-TP complex, indicating a substantially improved bioavailability of bound polyphenols. In contrast, YS-TP-U480 exhibited a significantly lower TPs release than all other ultrasonic treatment groups at all time points, with a peak value of only 66.2 mg GAE/g, suggesting that excessive ultrasonic power either embedded polyphenols more firmly within the starch matrix or induced partial degradation of polyphenols. These release results showed a significant correlation with the RS content measured during digestion. Notably, YS-TP-U288

achieved the highest RS content while simultaneously maintaining the maximum polyphenol release. In comparison, YS-TP-U480 presented low TPs release but relatively high RS content, indicating that different ultrasonic power levels induced distinct binding modes between polyphenols and starch molecules.

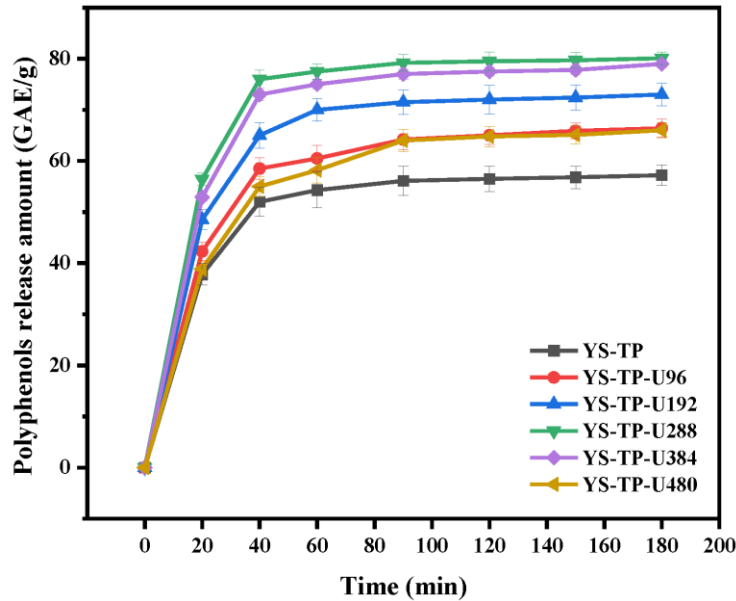


**Fig.5** Polyphenol release during *in vitro* digestion

### Total enzyme activity

To investigate the effect of TPs released from YS-TP complexes on digestive enzyme activity during *in vitro* digestion, dynamic changes in enzyme activity were monitored simultaneously alongside starch digestion assays. The temporal evolution of enzyme activity across all groups is presented in Fig. 6. The free YS group and all YS-TP complex groups exhibited a natural gradual decline in residual enzyme activity over the 0–180 min digestion period, while no significant intergroup difference in enzyme activity was observed at any matched time point ( $p > 0.05$ ). This finding demonstrated that the concentration of TPs released from YS-TP complexes during digestion was insufficient to exert direct inhibitory effects on the activity of digestive enzymes. Accordingly, the dominant mechanism by which YS-TP complexes delayed starch digestion was not enzyme inhibition, but rather the formation of V-type inclusion complexes between YS and TPs. Polyphenols were well documented to exert a broad spectrum of health benefits, among which the inhibition of digestive enzymes and potent antioxidant capacity were the most widely reported properties. However, like any chemical substance, polyphenols could also have harmful effects when combined with dosage, conditions, and environmental factors

(Tarko et al., 2023). According to our results, the anti-digestion mechanism of YS-TP complexes is due to physical structure modulation, rather than biochemical inhibition caused by TPs release. This structural-dependent mechanism can effectively avoid the potential digestive burden caused by digestive enzymes inhibition, especially for food-intolerant and elderly individuals with low digestive enzyme activity (Zhang et al., 2026).

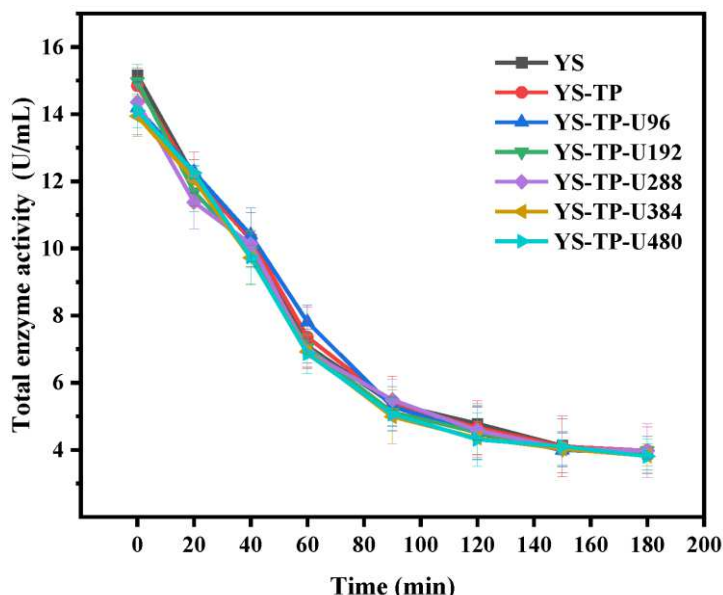


**Fig.6** Total enzyme activity of of YS and YS-TP complexes under ultrasonication treatments

### Thermal properties

The DSC parameters of native YS and YS-TP complexes prepared under different ultrasonic treatments are shown in Table 2. The gelatinization temperatures (initial temperature  $T_o$ , peak temperature  $T_p$  and end temperature  $T_c$ ) reflect the degree of crystallization and the crystalline structure of starch granules. The gelatinization enthalpy ( $\Delta H$ ) represents the energy required to separate the double helix structure in the starch crystallization region during the gelatinization process (Meng et al., 2025). Compared with native YS,  $T_o$ ,  $T_p$ , and  $T_c$  of YS-TP and YS-TP complexes under ultrasonic treatments increased significantly ( $p < 0.05$ ). These results confirm that TPs complexation and ultrasonic treatment synergistically enhanced the thermal stability of YS-TP complexes, which can be primarily attributed to the strong non-covalent intermolecular interactions formed between starch and polyphenols (Du et al., 2027; Wang et al., 2026). This more ordered molecular rearrangement of the starch-polyphenol matrix elevated the critical temperature required for crystalline phase melting, directly verifying the improved thermal resistance of the complexes. As the ultrasonic power increased from 96 W to 384 W, the

corresponding  $\Delta H$  increased from 2.66 J/g to 3.05 J/g ( $p < 0.05$ ), indicating that suitable ultrasonication can promote the formation of more ordered crystal structures between YS and TPs, and improve thermal stability. In addition, polyphenols are introduced into starch, which form new cross-linked structures between starch molecules through hydrogen bonding or hydrophobic interactions, thereby enhancing the binding between starch chains. This enhanced molecular interaction improves the thermal stability of starch granules. Nevertheless, the  $\Delta H$  of the 480 W ultrasonic group decreased significantly to 2.25J/g, suggesting that excessive ultrasonic power intensity disrupted the pre-formed ordered composite structure. A previous study by Zhang *et al.* found that TPs addition reduced the gelatinization enthalpy of corn starch and wheat starch, but increased the enthalpy of potato starch and tapioca starch. The difference in this result may be related to the unique intrinsic structural features of yam starch and its specific complexation mode with tea polyphenols (Zhang et al., 2025). Collectively, the ultrasonic power range of 288–384 W is identified as the optimal condition to maximize the thermal stability of YS-TP complexes.

**Table 2** Thermal properties of YS and YS-TP complexes under ultrasonication treatments

| Samples    | T <sub>0</sub> (°C)      | T <sub>P</sub> (°C)      | T <sub>c</sub> (°C)      | $\Delta H$ (J/g)       |
|------------|--------------------------|--------------------------|--------------------------|------------------------|
| YS         | 88.43±0.21 <sup>f</sup>  | 97.77±0.45 <sup>f</sup>  | 99.60±0.56 <sup>f</sup>  | 2.26±0.01 <sup>f</sup> |
| YS-TP      | 90.43±0.25 <sup>e</sup>  | 99.63±0.55 <sup>e</sup>  | 101.53±0.80 <sup>e</sup> | 2.46±0.26 <sup>e</sup> |
| YS-TP-U96  | 93.50±0.40 <sup>d</sup>  | 101.83±1.01 <sup>d</sup> | 103.10±1.14 <sup>d</sup> | 2.66±0.25 <sup>d</sup> |
| YS-TP-U192 | 102.73±0.25 <sup>b</sup> | 112.23±0.60 <sup>b</sup> | 114.60±0.40 <sup>b</sup> | 3.66±0.42 <sup>b</sup> |
| YS-TP-U288 | 108.63±0.31 <sup>a</sup> | 117.30±0.53 <sup>a</sup> | 119.50±0.36 <sup>a</sup> | 4.26±0.02 <sup>a</sup> |
| YS-TP-U384 | 97.40±0.80 <sup>c</sup>  | 106.53±0.25 <sup>c</sup> | 109.33±0.67 <sup>c</sup> | 3.05±0.03 <sup>c</sup> |
| YS-TP-U480 | 88.30±0.60 <sup>f</sup>  | 98.83±0.61 <sup>f</sup>  | 100.30±0.66 <sup>f</sup> | 2.25±0.01 <sup>f</sup> |

Significant differences within the same column are indicated by different superscripts ( $p < 0.05$ , based on Duncan's test)

## Conclusion

This study systematically investigated the effects of different ultrasonic power intensities (96–480 W) on the complexation behavior between YS and TPs. The complexation index, water hydration properties, thermal characteristics, crystalline structure, microstructure, *in vitro* digestibility, polyphenol release kinetics, and total digestive enzyme activity of native YS and YS-TP complexes were comprehensively evaluated. The results demonstrated that ultrasonic treatment significantly increased the CI of YS and TPs, effectively promoting the formation of V-type inclusion complexes. SEM observations revealed that ultrasonication transformed the microstructure of the complexes into irregular fragments with abundant pores, which facilitated the embedding of TPs molecules via non-covalent interactions. XRD analysis

further confirmed the appearance of characteristic V-type peaks at 7°, 13°, 17°, and 20°, accompanied by a notable increase in crystallinity. Meanwhile, the thermal stability of the complexes was significantly enhanced under moderate ultrasonic power (288 W), whereas excessive power (480 W) damaged the composite structure and led to a decrease in  $\Delta H$ . In terms of hydration properties, the YS-TP-U288 group exhibited moderate water solubility (75.37%) and swelling capacity (12.33 g/g), forming a relatively stable network-like structure. This sample also presented the highest crystallinity (38.9%) and the lowest hydrolysis rate during *in vitro* digestion. The RS content increased from 7% (native YS) to 29%, and the estimated glycemic index (eGI) decreased to 41.0. The release profile of TPs followed a biphasic pattern, characterized by rapid initial release followed by a sustained gradual phase. Moreover, the determination of total enzyme activity showed no significant differences among all treatment groups ( $p > 0.05$ ), confirming that the primary mechanism by which YS-TP complexes delay starch digestion is through physical barrier effects that reduce enzyme accessibility to starch substrates, rather than via direct inhibition of digestive enzyme activity. In summary, the YS-TP complexes developed in this study exhibit excellent thermal stability and favorable hydration properties, effectively slowing the rate of postprandial blood glucose elevation. These findings provide a solid theoretical foundation for the design and development of functional starch-based materials with high polyphenol loading capacity.

**Author Contribution** Dan Liang: Writing-original draft, Investigation. Mingzhu Wang: Formal analysis, Data curation. Xinhua Xie: Writing-review and editing. Lilin Cheng: Investigation, Formal analysis. Ziye Liu and Shuaishuai Zheng: Data curation. Zhilu Ai: Project administration, Supervision, Writing-review & editing.

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**Data Availability** Data will be made available on request.

## Declarations

**Competing interest** The authors declare no competing interests.

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