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# New starches from traditional Chinese medicine (TCM)—Chinese yam (*Dioscorea opposita* Thunb.) cultivars

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**Abstract**—The starches separated from two different *Dioscorea opposita* Thunb. cultivars were investigated for morphological, thermal and crystal properties. The shape of starch granules separated from different *D. opposita* Thunb. cultivars varied from round to oval or irregular. The surface of the starch granules appeared to be smooth without any fissures. The average particle diameter of starches from different *D. opposita* Thunb. cultivars was 40.3 and 38.7 μm for *D*. 47 and *D*. SXY starch, respectively. The transition temperatures ($T_o$, $T_p$ and $T_c$) and enthalpy of gelatinization ($\Delta H_{gel}$) were determined using differential scanning calorimetry (DSC). The *D*. SXY starch showed the lower $T_o$ (74.2 °C) and the broader *R* (12.4). $T_p$ and $T_c$ of starch from *D*. 47 were higher than that of *D*. SXY starch. $\Delta H_{gel}$ values (11.37 J/g) of *D*. 47 was higher than that of *D*. SXY starch (10.78 J/g). The crystal type of starches separated from two different *D. opposita* cultivars was a typical C-type pattern. The degree of crystallinity of two *D. opposita* cultivars starches was about 45.9% and 31.5%, respectively.


*Keywords:* *Dioscorea opposita* Thunb.; Starch; Morphology; Thermal; Crystallinity

Dioscoreae (Chinese name Shanyao), the rhizome of various species of genus *Dioscorea opposita* Thunb. (Dioscoreaceae), has been used as an important invigorant in traditional Chinese medicine (TCM) for many years.[1] *D. opposita* Thunb. was one of the most important substance with food and pharmaceutical functions. There were many *D. opposita* Thunb. cultivars planted in China especially in Henan province. In the rhizome of these *D. opposita* cultivars, starch was the main components making up to 20–60% content in the total biomass.[2,3]

Starch, the most important polysaccharide reserve in higher plants in the form of birefringent, semi-crystalline granules deserves a detailed research to understand better its biochemical and functional characteristics as well as variations. Extensive research has been conducted on the structure and functional properties of the Commercial starches obtained from crops due to their ready availability and their extensive utilization in food and non-food applications.[4,5] However, the starch from medicinal plants has not been paid enough attention by starch researchers. In order to understand the starch separated from medicinal plants, the starch separated from *Fritillaria* medicinal plants has been studied by our research group firstly.[6,7] In fact, there are many medicinal plants containing starch such as Rhizoma Dioscoreae, Rhizoma Curcuma Longa, Semen Ginkgo, Euphorbia Kansui, which all deserved detailed research on starch contained in them.

The objective of this work is to study the functional properties of two *D. opposita* Thunb. starches for food and medicine industry.

The starches separated from two *D. opposita* Thunb. cultivars differed significantly in granule size and shape when viewed by SEM. Scanning electron micrographs

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of the starch granules from different *D. opposite* Thunb. cultivars are illustrated in Figures 1 and 2.

SEMs of starches separated from different *D. opposita* Thunb. cultivars showed the presence of starch granules from small to large and round or oval to irregular or cuboidal with diameters ranges between 5–20 and 20–60 μm, respectively, for small and large granules. The surface of the granules appeared to be smooth with no evidence of any fissures. *D*. 47 starch showed the presence of a fairly large number of large-sized, cycloidal or elliptic-shaped granules while *D*. SXY starch showed the presence of many small-sized and oval or irregular granules.

Two representative curves of granule diameter of *D. opposita* Thunb. cultivars starches are shown in Figure 3. The diameter of majority of starch granules ranged between 7–60 and 7–50 μm with some granules having diameter in the range of 1.0–8 μm for *D*. 47 and *D*. SXY, respectively. According to the statistical result, *D*. 47 starch showed the presence of larger size granules with mean diameter of 40.3 μm whereas *D*. SXY starch had the smaller size granules with mean diameter of 38.7 μm. The results are in accord with the SEM analysis.

Physico-chemical properties, such as percent light transmittance, amylose content, swelling powder and water-binding capacity were significantly correlated with the average granule size of the starches separated from different plants.[11–13]

The results of DSC analysis of starches separated from different *D. opposita* Thunb. cultivars are summarized in Table 1.

The transition temperatures ($T_o$; $T_p$ and $T_c$), range ($T_c - T_o$), enthalpies of gelatinization ($\Delta H_{gel}$) and peak height indices (PHI) of starches from different *D. opposita* cultivars differed slightly. *D*. 47 starch showed higher $\Delta H_{gel}$ value of 11.37 J/g whereas *D*. SXY starch showed the lower $\Delta H_{gel}$ value of 10.78 J/g. *D*. SXY starch showed the lower $T_o$ (74.2 °C) while it was higher for *D*. 47 starch (75.4 °C). $T_p$ and $T_c$ of starches from two cultivars were 80.9, 85.5 and 81.5, 86.6 °C for *D*. 47 and *D*. SXY, respectively. The lower $T_o$ and the broader *R* for *D*. SXY starch may be attributed to the smaller granule size.[14] *D*. 47 starch showed the higher PHI and *D*. SXY starch showed the lower.

The X-ray diffractograms of starches from two different *D. opposita* Thunb. cultivars are presented in Figure 4.

In the diffraction spectra of the two starches separated from different *D. opposita* Thunb. cultivars, there were four strong diffraction peaks at 6.5°, 17.8°, 20.1° and 27.4° 2$\theta$. Of all the diffraction peaks, the peaks at

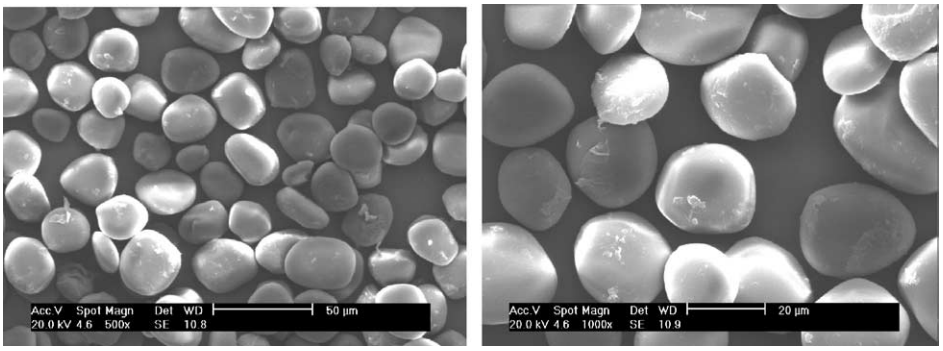


**Figure 1.** SEM of *D*. 47 starch granules.

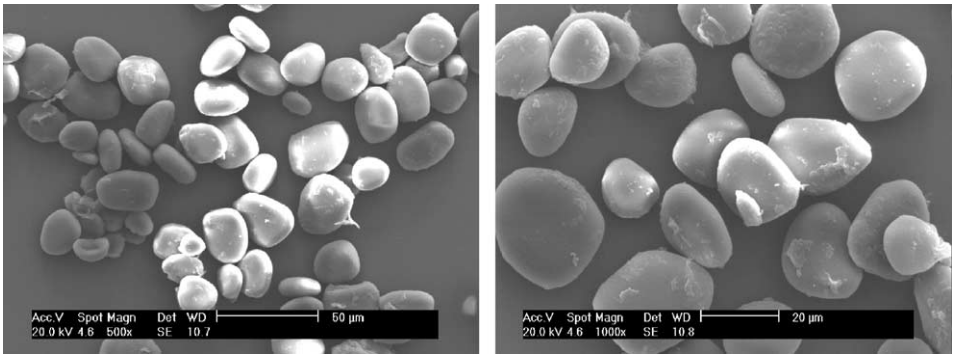


**Figure 2.** SEM of *D*. SXY starch granules.

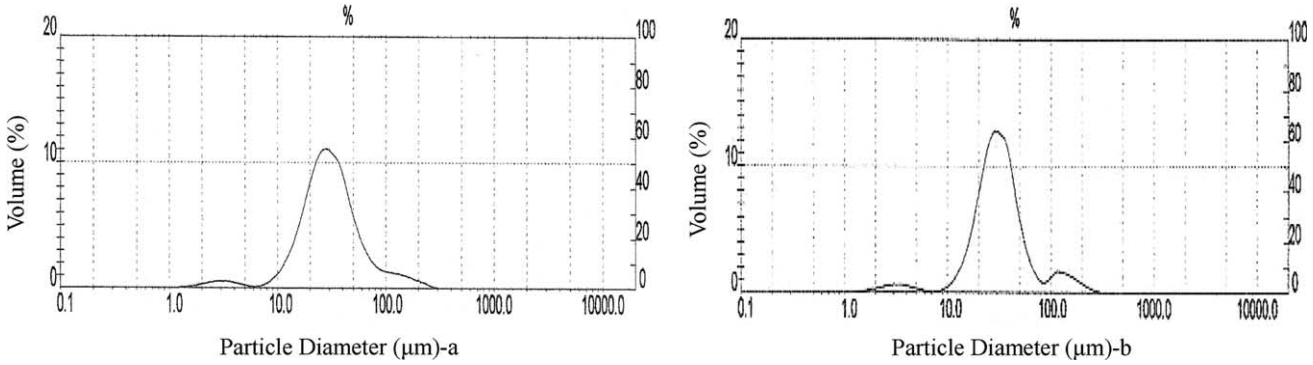


**Figure 3.** Particle size analysis of the starches separated from *D*. SXY (a) and *D*. 47 (b).

**Table 1.** Thermal properties of starches separated from two *D. opposita* Thunb. cultivars

| Samples | $T_o$ (°C) | $T_p$ (°C) | $T_c$ (°C) | $\Delta H_{gel}$ (J/g) | PHI | *R* |
|---|---|---|---|---|---|---|
| *D*. 47 | 75.4 | 80.9 | 85.5 | 11.37 | 2.07 | 10.1 |
| *D*. SXY | 74.2 | 81.5 | 86.6 | 10.78 | 1.48 | 12.4 |

$T_o$ = onset temperature, $T_p$ = peak temperature, $T_c$ = conclusion temperature, $R$ = gelatinization range ($T_c - T_o$); $\Delta H_{gel}$ = enthalpy of gelatinization (dwb, based on starch weight), PHI = peak height index $\Delta H_{gel}/(T_p - T_o)$.

around 2$\theta$ value of 6.5° were characteristic of B pattern. While at 27.4° 2$\theta$ only one peak appeared, which were indicative of the A pattern. Thus, the two *D. opposita* Thunb. starches were classified as C-type, which was a mixture of A-type and B-type.

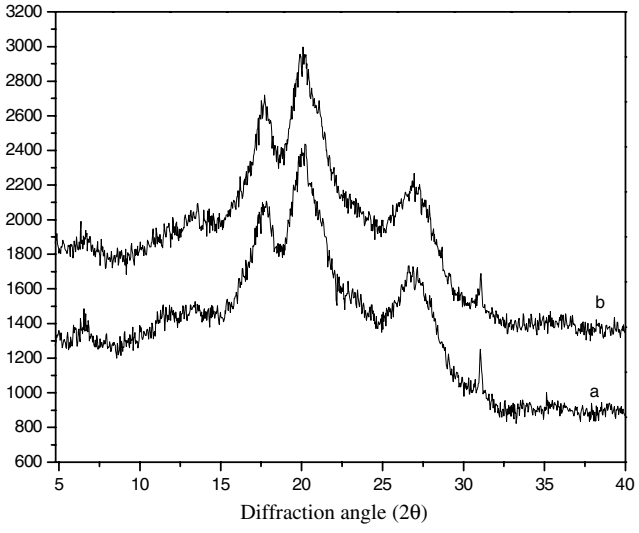


**Figure 4.** X-ray diffraction spectra of the two *D. opposita* Thunb. cultivars starches: (a) *D*. 47 and (b) *D*. SXY.

The corresponding X-ray diffraction parameters and crystallinity level calculated from the ratio of diffraction peak area and total diffraction area are given in Table 2.

The scattering angle, at which the diffraction intensities could be observed was 2$\theta$, and the *d* spacing was used to discriminate the planes of different sites. The degree of crystallinity of two *D. opposita* starches was 45.9% for *D*. 47 and 31.5% for *D*. SXY.

Starches separated from two *D. opposita* Thunb. cultivars showed obvious differences in morphological, thermal and crystalline properties. The shape of two *D. opposita* Thunb. starches varied from round or oval to irregular, similar to those of tuber starch granules. The average particle diameter of starches from different *D. opposita* Thunb. cultivars was 40.3 and 38.7 μm. These starches showed different $\Delta H_{gel}$ values and transition temperature, which were higher than the normal starches separated from corn, wheat and potato starches. The crystal type of *D. opposita* Thunb. cultivars starches was a C-type pattern.

**Table 2.** X-ray diffraction data of starches from different *D. opposita* Thunb.

| Samples | Diffraction peaks at 2$\theta$ values (° angle) (*d* spacing) | | | | Degree of crystallinity (%) |
|---|---|---|---|---|---|
| | 6° | 17° | 20° | 27° | |
| *D*. 47 | 6.55 (15.64 Å) | 17.87 (5.76 Å) | 20.18 (5.10 Å) | 27.04 (3.83 Å) | 45.9 |
| *D*. SXY | 6.74 (15.23 Å) | 17.92 (5.74 Å) | 20.23 (5.09 Å) | 27.14 (3.81 Å) | 31.5 |

## 1. Experimental

### 1.1. Materials

Two different *D. opposita* Thunb. cultivars (cv.), that is, *Dioscorea opposita* cv. 47hao (*D*. 47); and *Dioscorea opposita* cv. Songxianye (*D*. SXY) were provided by Henan Academy of Agricultural Sciences and were identified by Researcher Liu hongyan, Henan Academy of Agricultural Sciences, Henan province, China.

### 1.2. Starch isolation

For the separation of starch, the two different dried *D. opposite* Thunb. cultivars were washed, cut into small pieces and ground with a plant micro-muller, which were sieved with 160 mesh sifter. After having been sieved, *D. opposita* Thunb. powders were immediately steeped in water containing 0.1% $HgCl_2$ to prevent the microbial growth. After depositing, the supernatant was removed by suction and the settled starch layer was resuspended in distilled water. By seven or eight depositing and resuspending repeatedly, the slurry containing starch was centrifuged in wide-mouthed cups at 3000 rpm for 10 min. The supernatant was spilled and upper non-white layer was scrapped off. The white layer was resuspended in distilled water and recentrifuged for 3–5 times. The starch was then collected and dried at room temperature automatically.

### 1.3. Characterization

Scanning electron micrographs (SEM) were obtained with an environmental scanning electron microscope (ESEM, Philips XL-3). Starch samples were suspended in acetone to obtain a 1% suspension. One drop of the starch–acetone suspension was applied on an aluminium stub using double-sided adhesive tape and the starch was coated with gold powder to avoid charging under the electron beam after the acetone volatilized. An accelerating potential of 30 kV was used during micrography.

Particle size analysis of starches from different *D. opposita* Thunb. cultivars was done using a laser light scattering particle size analyzer (Mastersizer S, version 2.15, Malvern instruments Ltd, Malvern, UK). The focal length was 100 mm.

Thermal characteristics of isolated starches were studied by using a differential scanning calorimeter, DSC204, HP (NETZSCH, Germany) equipped with a thermal analysis station. Each of *D. opposita* Thunb. cultivars starches (3.5 mg, dry weight) was loaded into a 40 μl capacity aluminium pan (Mettler, ME-27331) and distilled water was added with the help of Hamilton microsyringe to achieve a starch–water suspension containing 70% water. Samples were hermetically sealed and allowed to stand for 1 h at room temperature before heating in the DSC. The DSC analyzer was calibrated using indium and an empty aluminium pan was used as reference. Sample pans were heated at a rate of 10 °C/min from 20 to 120 °C. The transition temperature reported were onset temperature ($T_o$), peak temperature ($T_p$) and conclusion temperature ($T_c$). The enthalpy of gelatinization ($\Delta H_{gel}$) was estimated by integrating the area between the thermogram and a base line under the peak. The gelatinization temperature range ($R$) was computed as ($T_c - T_o$) as described by Vasanthan and Bhatty.[8] Enthalpies were calculated on a starch dry basis. The peak height index (PHI) was calculated by the ratio $\Delta H/(T_p - T_o)$ as described by Krueger et al.[9]

X-ray powder diffraction measurements were done using Panalytical X'Pert Pro diffractometer (PANalytical, Holland). Each sample of *D. opposita* Thunb. cultivars starches was packed tightly in a rectangular glass cell (15 × 10 mm, thickness 0.15 cm). The samples were exposed to the X-ray beam from the X-ray generator running at 40 kV and 40 mA. The scanning regions of the diffraction angle 2$\theta$ were 4–40°, which covered most of the significant diffraction peaks of the starch crystallites. The other operation conditions were as follows: $\lambda = 1.78901$ Å, step size, 0.0330°, scan step time, 30.8451 s, divergence slit size, 0.2177°. The degree of crystallinity of *D. opposita* Thunb. starches was quantitatively estimated following the method of Nara and Komiy.[10] A smooth curve, which connected peak baselines was computer-plotted on the diffractograms (Fig. 5). The area above the smooth curve was taken as the crystalline portion, and the lower area between smooth curve and the linear baseline, which connected the two points of the intensity 2$\theta$ of 30° and 4° in the samples was taken as the amorphous section. The upper diffraction peak area and the total diffraction area over the diffraction angle 4–30° 2$\theta$ were integrated using Smadchrom software (Morgan and Kennedy Research, Australia). The ratio of the upper area to the total diffraction was taken as the degree of crystallinity.

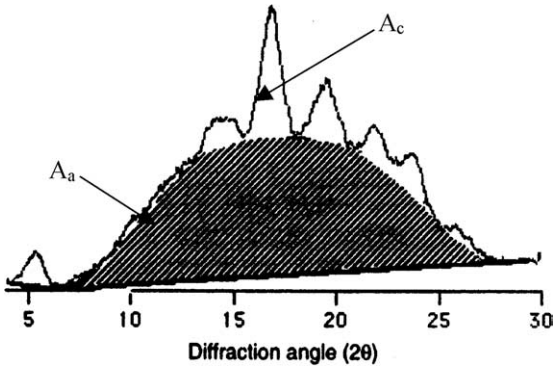


**Figure 5.** Calculation of the relative degree of the crystallinity.

The equation of the degree of crystallinity is as follows:

$$X_c = A_c/(A_c + A_a)$$

where $X_c$ refers to the degree of crystallinity; $A_c$ refers to the crystallized area on the X-ray diffractogram; $A_a$ refers to the amorphous area on the X-ray diffractogram.

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