CELLULOSE CHEMISTRY AND TECHNOLOGY

EFFECT OF MIXING CASSAVA, POTATO AND SWEET POTATO STARCHES ON THE PROPERTIES OF THEIR BLENDS

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In order to optimally improve as much as possible the properties of native starch and modified starch, different blends of cassava starch, potato starch and sweet potato starch were prepared to investigate the effect of blending on the acidolysis, acetylation and cross-linking degree, as well as other properties of the obtained starches. The results showed that cassava starch, potato starch and sweet potato starch differed in their sensibility to the acidolysis agent, cross-linker and acetylating reagent. Potato starch was more difficult to hydrolyze than cassava starch, while sweet potato starch was more difficult to acetylate than potato starch. The enthalpy change of the starch blend was greater than that of any of its components. The crystalline structure of the blend was closer to that of cassava starch rather than that of sweet potato starch, at the ratio of cassava starch to sweet potato starch of 1:1.

Keywords: mixing starch, acidolysis, acetylation, cross-linking, property

INTRODUCTION

Starches, including those extracted from corn, potato, cassava, rice, sweet potato, mung bean, pea *etc*., are employed in foods due to their good gelling and thickening performance. However, some of their drawbacks, such as low resistance to shear, high retrogradation[1] and poor freeze-thaw stability,[2] limit their use in industrial applications. Fortunately, these natural shortcomings can be overcome by modifications by chemical, physical and enzymatic methods.[3] In addition, a new and interesting way to improve the performance of native starches, enhancing their properties, is achieved by mixing different starches, because each starch has its peculiar functional properties, but it cannot provide a wider range of desirable characteristics for industrial application. For example, cassava starch has weak retrogradation and high viscosity, owing to its high content of amylopectin, compared with corn starch.[4,5] Potato starch and sweet potato starch also have high viscosity, but their retrogradation lies between that of cassava starch and that of corn starch. The paste of potato starch and cassava starch is transparent, but the paste of sweet potato starch is semi-transparent.[6-8] Moreover, the size of sweet potato starch particles is close to that of cassava starch and corn starch, but is smaller than that of potato starch. [9]

Acidolysis is a chemical modification method usually used to cut the molecular chains of native starches. The common reagents for acidolysis include hydrochloric acid,[10] sulphuric acid[11] and nitric acid.[12] After the acidolysis of starch, its viscosity and swelling power decreases, whereas its gelatinization temperature increases.[13] Acetylation will improve the freeze-thaw stability of starch by means of retarding the recrystalisation, that is, strengthening the bonds between the amylose and amylopectin molecules, and preventing water leaching out of the starch granules.[14] In addition, acetylation is able to raise the swelling power,[15] and reduce the pasting temperature.[16] Cross-linking is another modification method commonly used for starches, which can stabilize the structure of starch granules and restrict swelling. The cross-linking reagents usually include sodium trimetaphosphate, phosphoryl chloride, mixed anhydride of acetic and adipic acid and others.[17] Cross-linking can increase the gelatinization temperature of starch, but reduces the viscosity.[18]

Much research reported in the literature has focused only on various modifications of a variety of single starches, while the modification and properties of blends of different starches have not been discussed so far. Therefore, based on our previous work on the oxidation, hydroxypropylation,[19] acetylation and cross-linking[20-21] of high-amylose corn starch, in this study, we aimed to inexpensively and optimally improve the functional characteristics of different native starches by mixing them. Thus, we explored the effects of blending cassava, potato and sweet potato starches on the acidolysis, acetylation, cross-linking and performance of the obtained blends.

EXPERIMENTAL

Materials

Cassava starch (CS) was purchased from Desu Cassava Starch Factory (Vietnam). Potato starch (PS) was purchased from Nei Monggol Nailun Agricultural Science and Technology Co., Ltd. (China). Sweet potato starch (SPS) was purchased from Lulong Yidayuan Starch Co., Ltd. (China). Epichlorohydrin, sodium hydroxide, hydrochloric acid and acetic anhydride were obtained from Sinopharm Chemical Reagents Shenyang Co., Ltd. (China). All the reagents were of analytical grade.

Methods

Acidolysis, acetylation and cross-linking of starches

The acidolysis, acetylation and cross-linking of starch were carried out according to the previously published methods, with slight modifications.[22-24]

An amount of 30 g of blended starch (dry basis) was mixed with 53.3 g of distilled water in a 250 mL three-neck round bottom flask, equipped with a mixer. The slurry was heated to 45 °C and stirred for 5 min. 0.83 g of 36% (w/w) HCl was used, and the acidolysis was carried out for 4.0 h. After that, the slurry was neutralized to a pH of 6.5 with 4% (w/w) NaOH aqueous solution, and the slurry was filtered on a Buchner funnel. The filter cake obtained was washed with distilled water to totally remove any impurities, and then broken into very small pieces and dried for around 2.5 h under an infrared lamp. The dried powder was ground and screened. Finally, acidolyzed starch was obtained.

An amount of 30 g of blended starch (dry basis) was mixed with 53.3 g of distilled water in a 250 mL three-neck round bottom flask, equipped with a stirrer, and the mixture was heated to 25 °C and stirred for 10 min. The pH of the slurry was adjusted to 8.5 with 4% (w/w) NaOH aqueous solution. 1.8 g of acetic anhydride was slowly added into the suspension under constant agitation and constant pH, and the acetylation was carried out for 1.5 h. Thereafter, the pH of the slurry was adjusted to 6.5 with 5% (w/w) HCl. Similarly to the procedure described above, the filtration, washing, drying, grinding and screening were accomplished in order to obtain acetylated starch.

An amount of 30 g of blended starch (dry basis) was mixed with 53.3 g of distilled water and 3.0 g of anhydrous sodium sulfate in a 250 mL three-neck round bottom flask, equipped with a mixer, and the slurry was heated to 40 °C and stirred for 15 min. Afterwards, The pH of the slurry was adjusted to 10.5 with 4% (w/w) NaOH aqueous solution, 0.9 g of epichlorohydrin was added into the slurry and the cross-linking was allowed to proceed for 4.0 h. Then, the pH was adjusted to 6.5 with 4% (w/w) HCl. Next, the filtration, washing, drying, grinding and screening were performed as described above and cross-linked starch was obtained.

Cross-linking degree, acidolysis degree, substitution degree, swelling capacity and freeze-thaw stability

The cross-linking degree, acidolysis degree, substitution degree, swelling capacity and freeze-thaw stability were determined by previously published methods.[25-29] The cross-linking degree was assessed using the settling volume. The acidolysis degree was evaluated by alkali fluidity, which was determined using 1% (w/w) starch dispersion in 1% (w/w) NaOH. The paste of 4% (w/w) samples (dry basis) was thawed at room temperature for 4 h and then refrozen at -18 °C repeatedly up to five cycles in order to obtain the freeze–thaw stability.

Characterization

Differential calorimetry measurements were carried out using a DSC Q20 V24.4 Build 116 differential scanning calorimeter (TA Instruments, USA).[30] The crystalline structure was analyzed by a D8 ADVANCE Bruker X-ray diffractometer (Germany), in the reflection mode (angular region of 5 to 45º, steps of 0.03º).[31] The gelatinization behavior of the starches, with a mass concentration of 5%, was recorded using a MCR102 rheometer (the initial mixing speed of 960 rpm for ten seconds, normal mixing speed of 160 rpm).[32-33] The size distribution of dry starches was determined by a Mastersizer 3000 (Malvern Panalytical, UK).[34-35]

RESULTS AND DISCUSSION

Effect of blending CS, PS and SPS on acidolysis degree

For estimating the acidolysis degree of the blended starches, their alkali fluidity was analyzed. For the blend of CS with PS prepared for the acidolysis experiments (Fig. 1a), it was noted that the acidolysis degree of the mixed starch varied with increasing the amount of CS. When the amount of CS was 30%, the acidolysis degree of the mixed starch reached a maximum value. It confirmed that PS was more difficult to hydrolyze than CS. When CS was mixed with SPS (Fig. 1b), the acidolysis degree of the mixed starch changed insignificantly with increasing the amount of CS, which confirmed that the acid resistance of SPS was close to that of cassava starch. When SPS was mixed with PS for the acidolysis experiments (Fig. 1c), the acidolysis degree of the obtained blend could not be kept constant. However, when the amount of SPS was 40% or 50%, the fluidity of the mixed starch dropped to 66 mL, confirming that the acid resistance of SPS was much different from that of PS.

Effect of blending CS, PS and SPS on acetylation degree

The acetylation degree of different blends of CS, PS and SPS is shown in Figure 2. It can be noted that the tendency of DS variation of the blend made of CS and PS was basically consistent with that of the blend composed of CS and SPS, while the blend of SPS and PS exhibited a very different trend from the other two blends. This confirmed that PS and SPS were not as easily acetylated as CS, while SPS was more difficult to acetylate than PS.

Effect of blending CS, PS and SPS on cross-linking

The cross-linking degree of the blended starches was assessed using the settling volume. The evolution of the settling volume was similar for the blend that consisted of CS and PS and that of SPS and PS, but different from that of the CS and SPS blend (Fig. 3). This confirmed that CS and SPS were not easily cross-linked, compared with PS. The cross-linking activity of the three starches takes the following decreasing order: SPS > CS > PS.

Effect of blending CS, PS and SPS on size distribution

The effect of blending CS, PS and SPS on the size distribution of the blends is shown by the data listed in Table 1. From these data, it may be noted that, when increasing the mixing ratio of CS to PS, that of CS to SPS and of SPS to PS, the specific surface area of the blends increased, and the Dv(10), Dv(50) and Dv(90) of the blended starches dropped, except for the Dv(10) of the blend composed of CS and SPS. The decreasing sequence of the specific surface area of the starches was noted to be as follows: CS > SPS > PS, while the sequence of the mean sizes was PS > SPS > CS.

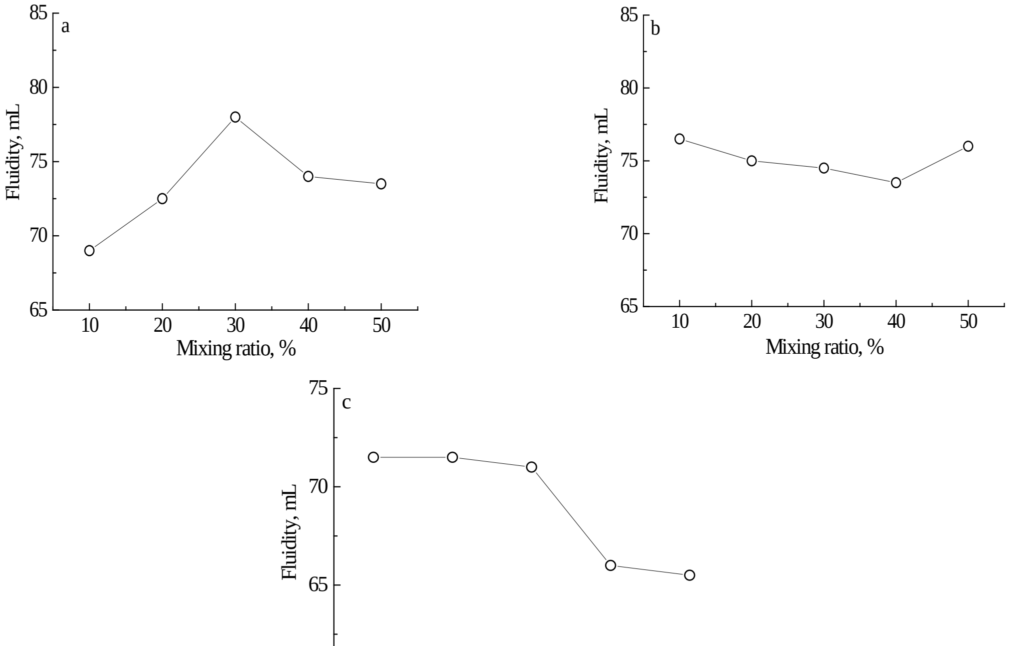


Figure 1: Effect of blending CS, PS and SPS on fluidity of acidolyzed starches; a) CS and PS blend, b) CS and SPS blend, c) SPS and PS blend

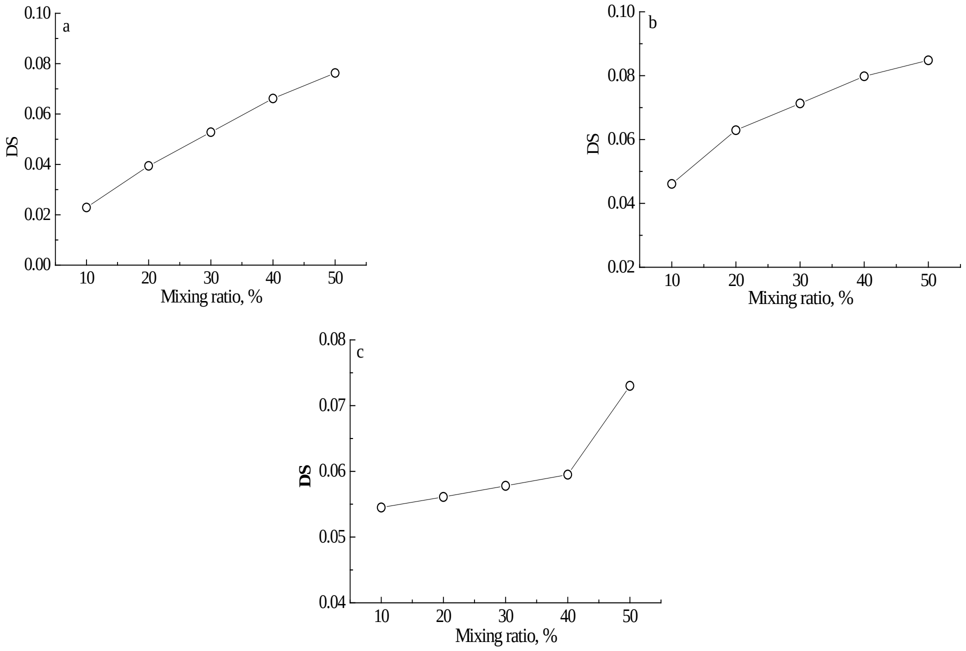


Figure 2: Effect of blending CS, PS and SPS on DS of acetylated starches; a) CS and PS blend, b) CS and SPS blend, c) SPS and PS blend

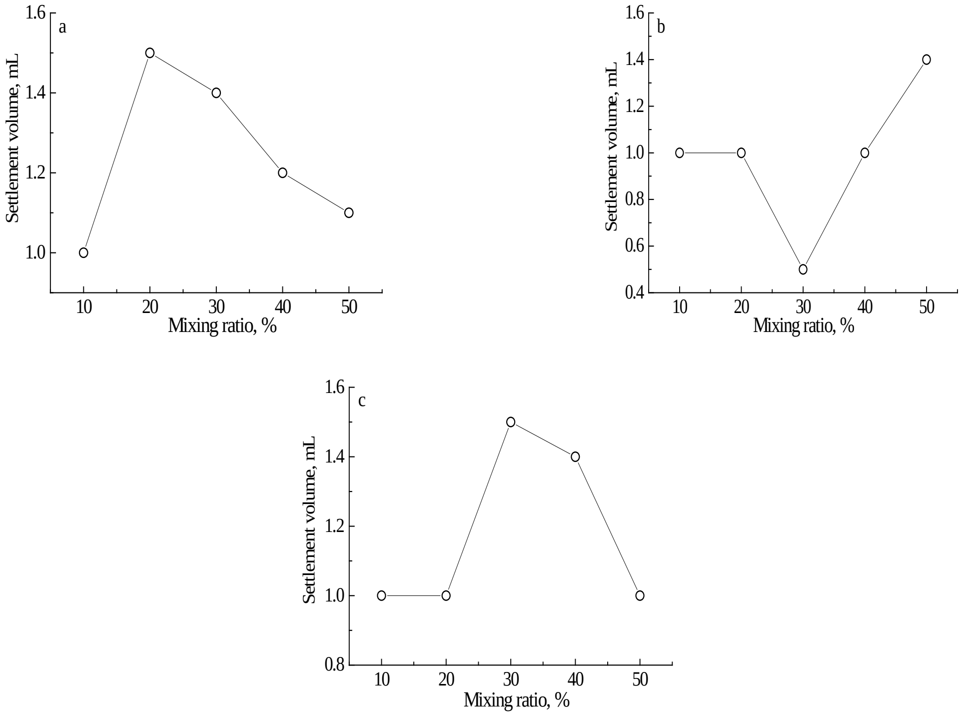


Figure 3: Effect of blending CS, PS and SPS on settling volume of cross-linked starches; a) CS and PS blend, b) CS and SPS blend, c) SPS and PS blend

Effect of blending CS, PS and SPS on gelatinization properties

The effect of blending CS, PS and SPS on the gelatinization properties of the obtained mixed starches is illustrated in Table 2. The data in Table 2 indicate that the pasting temperature of PS, CS and SPS was 65.9 °C, 56.0 °C and 71.9 °C, respectively. The breakdown and setback of the CS and PS mixed starches decreased, while the breakdown and setback of the CS and SPS mixed starches rose. Interestingly, the breakdown of the mixed starches also diminished as the mixing ratio of SPS to PS was increased. According to their breakdown, setback and peak viscosity values, the starches can be arranged in the following sequence: PS > CS > SPS. Meanwhile, considering the trough viscosity and end viscosity of the starches, their order changes as follows: PS > SPS > CS.

Table 1
Effect of blending CS, PS and SPS on size distribution

Blend	Mixing ratio, %	Specific surface area, $m^2.kg^{-1}$	Dv(10), µm	Dv(50), µm	Dv(90), µm
Mixing CS with PS	0	469.3	17.1	31.2	53.0
	30	676.1	11.4	24.6	47.0
	50	763.3	9.89	20.8	41.2
	100	987.8	8.76	15.5	25.9
Mixing CS with SPS	0	865.6	8.58	16.9	29.6
	30	942.5	8.55	16.4	28.4
	50	949.4	8.63	16.1	27.5
	100	987.8	8.76	15.5	25.9
Mixing SPS with PS	0	469.3	17.1	31.2	53.0
	30	609.5	12.0	25.7	47.8
	50	720.0	10.1	22.1	41.8
	100	865.6	8.58	16.9	29.6

Note: Dv(10), Dv(50) and Dv(90) are the average particle size when the cumulative percentage of particle size distribution reaches 10%, 50%, 90%, respectively

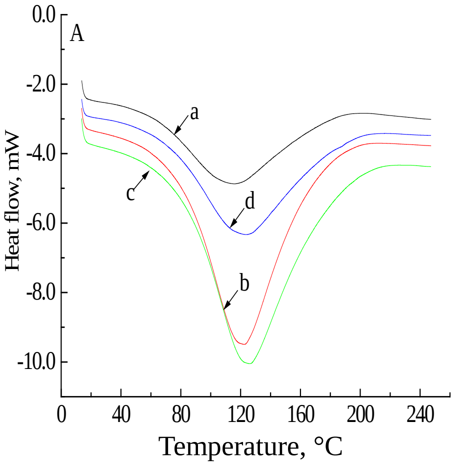
Table 2
Effect of blending CS, PS and SPS on key pasting parameters

Blend	Mixing ratio, %	Pasting temperature, °C	Peak viscosity, cP	Trough viscosity, cP	End viscosity, cP	Breakdown, cP	Setback, cP
Mixing CS with PS	0	65.9	3376	1220	1778	2157	558
	30	66.7	2677	1236	1813	1441	577
	50	66.3	1964	1063	1606	901	543
	100	56.0	1088	516	948	572	432
Mixing CS with SPS	0	71.9	772	646	1006	126	360
	30	68.4	944	626	991	318	365
	50	65.3	1023	599	990	424	391
	100	56.0	1088	516	948	572	432
Mixing SPS with PS	0	65.9	3376	1220	1778	2157	558
	30	76.8	1751	1114	1703	637	589
	50	64.4	1235	1002	1569	233	567
	100	71.9	772	647	1006	125	359

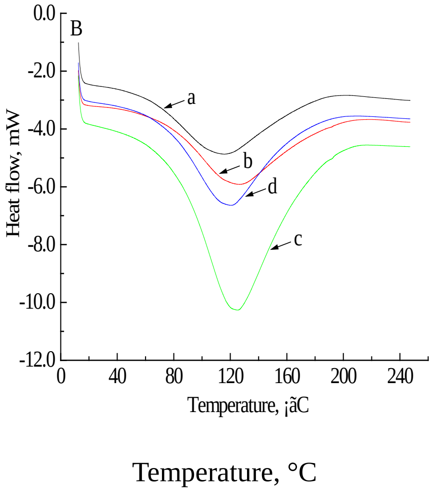
Effect of blending CS, PS and SPS on thermal properties

The peak intensity of PS was maximum, followed by those of SPS and CS (Fig. 4). When CS, PS and SPS were mixed to prepare different blends with proportions, the DSC curves of the mixed starches did not change proportionally with the increase in the mixing ratio of the starches. The data in Table 3 reveal that, as the ratio of CS to PS was increased, the enthalpy change of the mixed starch dropped regularly, but the concluding temperature and the peak temperature did not vary regularly when the ratio of CS to PS was 30% or 50%, while the concluding temperature and the peak temperature of the mixed starch were higher than those of CS and PS taken individually. For the mixed starch composed of CS and SPS, the enthalpy change was greater than that of CS and

SPS individual starches. When for the same blend, the mixing proportion of 30% was used, its concluding temperature was higher than that of SPS and PS taken individually. This finding confirmed that the thermal properties of certain starches can be improved by blending them together.



a: CS b: PS c: mixed starch (CS:PS = 3:7)
d: mixed starch (CS:PS = 1:1)



a: CS b: SPS c: mixed starch (CS:SPS = 3:7)
d: mixed starch (CS:SPS = 1:1)

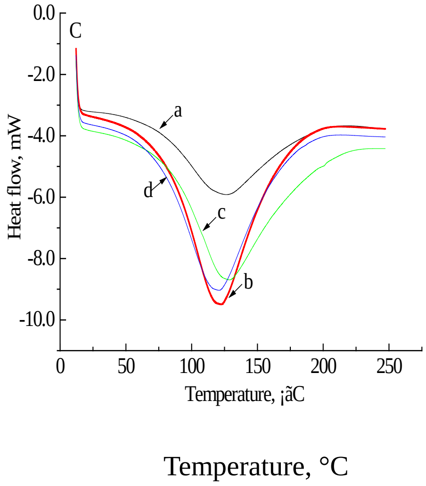
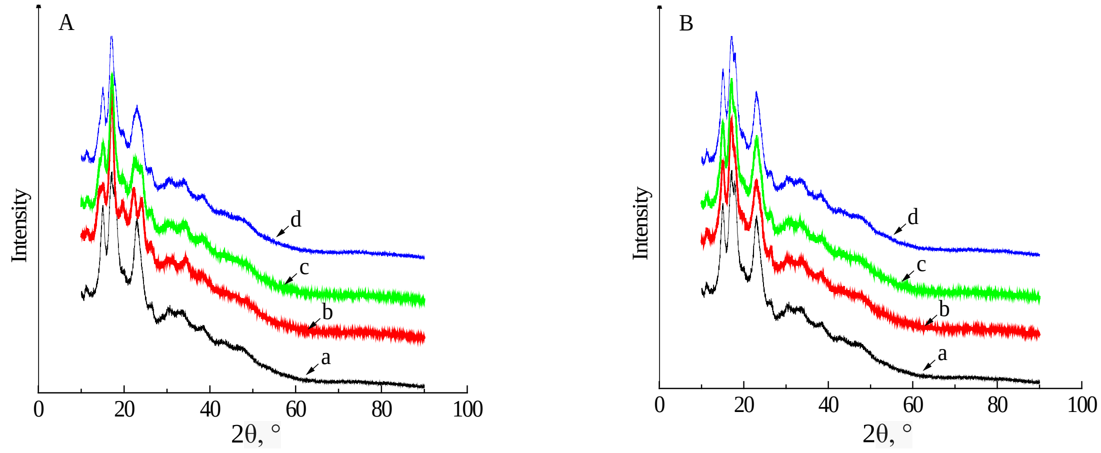


Figure 4: DSC curves for CS a: SPS b: PS c: mixed starch (SPS:PS = 3:7)
d: mixed starch (SPS:PS = 1:1) SPS and PS blend (C)

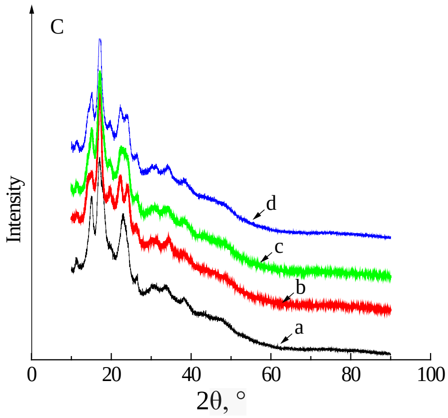
Table 3
Effect of blending CS, PS and SPS on key parameters of DSC thermodynamics

Blend	Mixing ratio, %	T_o, °C	T_p, °C	T_c, °C	ΔH, J•g^{-1}
Mixing CS with PS	0	82.04	122.23	174.69	386.4
	30	79.06	125.81	183.27	355.4
	50	68.75	123.43	186.15	290.9
	100	62.34	115.57	178.35	268.0
Mixing CS with SPS	0	74.75	126.06	187.15	326.3
	30	80.21	124.86	181.75	350.7
	50	70.24	120.29	173.72	393.1
	100	62.34	115.57	178.35	268.0
Mixing SPS with PS	0	82.40	122.23	174.69	386.4
	30	81.17	128.60	198.35	314.9
	50	73.46	119.62	172.83	372.6
	100	70.64	126.06	193.02	326.9



a: CS b: PS c: mixed starch (CS:PS = 3:7)
d: mixed starch (CS:PS = 1:1)

a: CS b: SPS c: mixed starch (CS:SPS = 3:7)
d: mixed starch (CS:SPS = 1:1)



a: SPS b: potato starch c: mixed starch (SPS:PS = 3:7)
d: mixed starch (SPS:PS = 1:1)

Figure 5: XRD curves of CS and PS blend (A), CS and SPS blend (B) and SPS and PS blend (C)

Effect of blending CS, PS and SPS on crystalline structure

The diffraction pattern of CS shows the four characteristic peaks at diffraction angles of 15.1°, 17.2°, 18.1° and 23.0°, which indicates that the crystalline structure belonged to an A-type starch. The diffraction peaks of PS appeared at 14.9°, 17.3°, 19.8°, 22.3° and 24.1°, which is diffraction pattern typical of a B-type starch. Meanwhile, the diffraction peaks of SPS, appearing at 15.3°, 17.3°, 23.0°, indicated that its structure belonged to a C-type. As regards the blends, the XRD pattern of the mixed starch composed of 30% CS and 70% PS was more similar to that of PS rather than to that of CS. However, the diffraction peaks of this blend at the diffraction angles of 22.3° and 24.1° almost merged into a single peak (for the mixing ratio of CS to PS of 3:7). When CS and PS were mixed in the ratio of 1:1, the blend exhibited only the peaks at 15.3°, 17.3°, 23.0°, and its structure became a C-type one. The XRD pattern of the CS and SPS blend (with a mixing ratio of 3:7) was closer to that of SPS rather than to that of CS. When the mixing ratio of CS to SPS was 1:1, however, the XRD pattern of the blend was analogous to that of CS rather than to that SPS. For the SPS and PS blend, mixed in the ratio of 1:1, the XRD pattern resembled that of PS rather than that of SPS.

Effect of blending CS, PS and SPS on swelling capacity

Table 4 shows the effect of blending CS, PS and SPS on the swelling capacity and freeze-thaw stability of the blends. According to the swelling capacity three individual starches at the temperature of 60 °C, they can be arranged in the following decreasing order: PS > CS > SPS. At the same time, in terms of the freeze-thaw stability, the three starches take the following sequence: PS > SPS > CS. The swelling capacity and freeze-thaw stability of PS was evidently greater than those of the other two starches. Therefore, when PS was mixed with

CS or SPS, the swelling capacity and the freeze-thaw stability of the blend were evidently improved. On the other hand, when CS was mixed with SPS, the freeze-thaw stability of the mixed starches varied insignificantly.

Table 4
Effect of blending CS, PS and SPS on swelling capacity and freeze-thaw stability

Blend	Mixing ratio, %	Solubility (60 °C), %	Swelling capacity (60°C), %	Syneresis rate, %
Mixing CS with PS	0	4.0	7.0	69.9
	30	3.6	6.2	74.1
	50	2.8	5.8	77.4
	100	3.2	3.2	81.1
Mixing CS with SPS	0	3.6	2.4	80.0
	30	2.0	3.2	80.2
	50	1.6	2.2	81.0
	100	3.2	3.2	81.1
Mixing SPS with PS	0	4.0	7.0	69.9
	30	2.4	5.2	75.8
	50	2.8	4.8	76.1
	100	3.6	2.4	80.0

CONCLUSION

The study allowed drawing the following conclusions. The acid resistance of SPS approached that of CS. CS was more easily acetylated than PS and SPS, while PS was more easily cross-linked than the other two starches. Blending different starches evidently influenced the pasting properties of the obtained mixed products. The enthalpy of the CS and PS blend dropped steadily, as the mixing ratio of CS to PS was increased. The crystalline structure of the same blend, at the ratio of CS (A-type) to PS (B-type) of 1:1, was indicative of an A-type starch, while that of the CS and SPS blend, at the mixing ratio of 3:7, revealed a structure closer to that of SPS instead of that of CS. The swelling capacity and freeze-thaw stability of the mixed starches did not change proportionally while increasing the content of one of the components in the blends.

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