

# The test of Tensile Properties and Water Resistance of a Novel Cross-linked Starch Prepared by Adding Oil-Flax

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**Abstract.** In this study, to solve the poor water resistance and the low mechanical properties of starch, a mixed-starch composite matrix which including glycerol, sorbitol, and urea, were prepared via single-screw extrusion, then adding oil-flax to improve its physical mechanical and used to a source of biodegradable plastics material. The composite matrix was systematically characterized using various analytic tools including XRD, SEM and TG. The composite showed a maximum tensile strength of 18.11Mpa and moisture absorption 17.67%, while the original starch matrix was only 12.51 Mpa and 24.98%, respectively.

## 1. Introduction

Nowadays, Thermoplastic starch (TPS) has been used in many fields, especially in textile industry. TPS is a source of biodegradable plastics material with advantages such as availability, low cost and machinability which is believed to replace conventional plastics in the areas of packaging, food, agriculture films and others. The widely use of TPS will relieve the pressures in resource and environment. It will also have a major significance in sustainable human development. But the poor water resistance and the low mechanical properties of starch limit its widespread application [1].

In this paper, we blended native corn starch with STMP cross-linked starch as the matrix material first. Prepare the composite matrix in the effect of the compound plasticizers which including glycerol, sorbitol, and urea through single-screw extrusion. Investigate the influence of the mass fraction of the cross-linked starch and the processing temperature on the properties of the composite matrix [2].

After mixing cross-linked starch, in the role of the plasticizing effect of the compound plasticizers and the shearing force of single screw, tensile strength of the mixed-starch matrix increased 44.76%, moisture absorption decreased 38.64 %. This proves that cross-linked starch has significantly enhanced effect on matrix. When the content of cross-linked starch increased over 15wt%, the increase in the tensile properties of matrix material trended to flat. Considering, the 15wt% content of cross-linked starch is most reasonable [3].

The mixed-starch composite matrix could straightforward to operate, it is easy to apply and maintain, affordable to implement, therefore has the potential to become widely employed for the textile industry.



## 2. Experimental

### 2.1. Materials

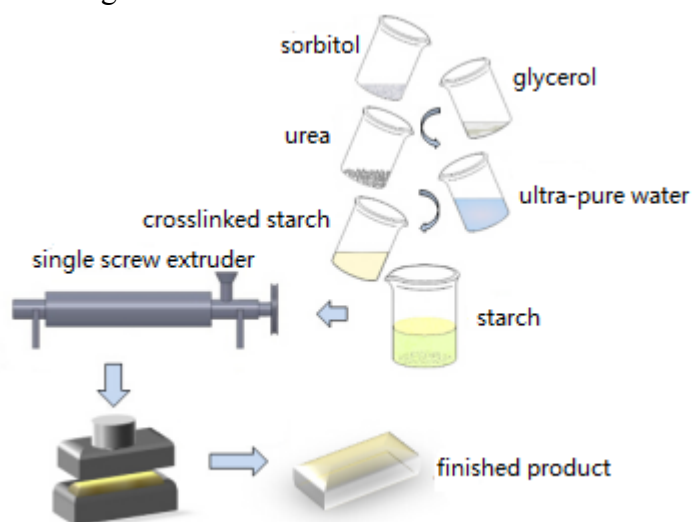
The thermoplastic Starch was kindly offered by Nanjing Haidexin technology, Co., Ltd. (Jiangsu, China). Crosslinked corn starch (purity $\geq$ 99%) was purchased from Zibo Haipeng Fine Chemical Co., Ltd. (Shandong, China). Urea, sorbitol and glycerin was obtained from Tianyi Chemical Reagent Institute Co., Ltd. (Tianjin, China). All of the other chemicals including isopropanol and ethanol were of analytical grade and purchased from Tianjin Kermel Chemical Reagent Co., Ltd. (Tianjin, China). All reagents and solvents were used as received.

### 2.2. Preparation of mixed-starch composite matrix

The procedure for the preparation of mixed-starch composite matrix is as follows:

(1). Pretreatment: The compound plasticizer was firstly immersed into the ultra-pure water for 24 h to remove the impurities and prepared by the proportion of glycerol, urea and sorbitol by 4:3:3. After drying in the oven, they were using a small electric blender stirring for 2 minutes, which was used as a matrix for the surface modification.

(2). Preparation step: Small screw extruder boot and heating treatment of the starch samples was carried out using a glycerol to screw infiltration, in the equipment, keep the speed of 130 rad/min, stable temperature at 120 °C after blending starch feeding screw extruder. The schematic of electron beam reactor was pictured in Fig 1.



**Figure 1.** Schematic of preparation of mixed-starch composite matrix.

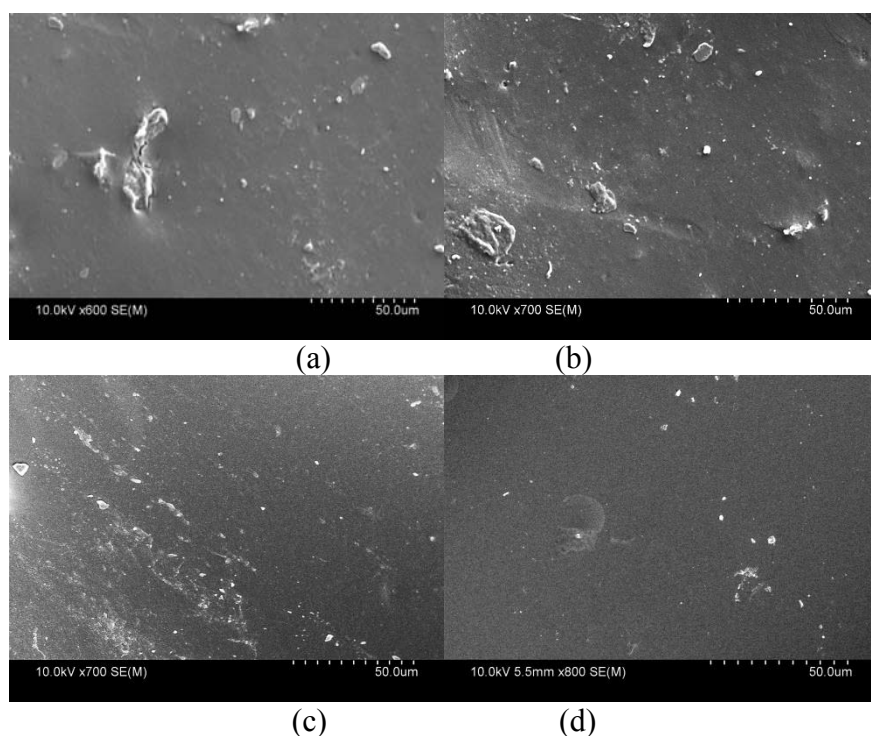
### 2.3. Characterization of mixed-starch composite matrix

High-energy-resolution XRD analyses were performed on a Kratos AXIS 165 spectrometer using monochromatic Al K $\alpha$  X-rays. Scanning electron microscope (SEM) investigations were performed by a Philips 505 M microscope. Matrix samples were frozen in liquid nitrogen, fractured to obtain fragments, mounted on disc holder with double sided tape and sputter-coated with thin gold film prior to SEM observation. The thermo-gravimetric was performed in a range of 0–800°C by 10°C min<sup>-1</sup> under nitrogen protection on a NETZSCH STA409PC instrument (Netzsch, Germany). Referring to experimental methods of tensile properties of GB1447283, tested tensile performance of coated fabrics on an Instron universal testing machine. Sample specification was 200 × 25 mm, thickness was 2.4 mm, the clamping distance was 100 mm, and loading speed was 2 mm/min. The composites were die-pressed to form cylindrical toroidal specimens with 7.0 mm outer diameter, and 3.0 mm inner diameter. The measurements of EM wave loss property for the specimens were carried out using a PNA 3629D vector network analyzer in the 30–6000 MHz range.

### 3. Results and discussion

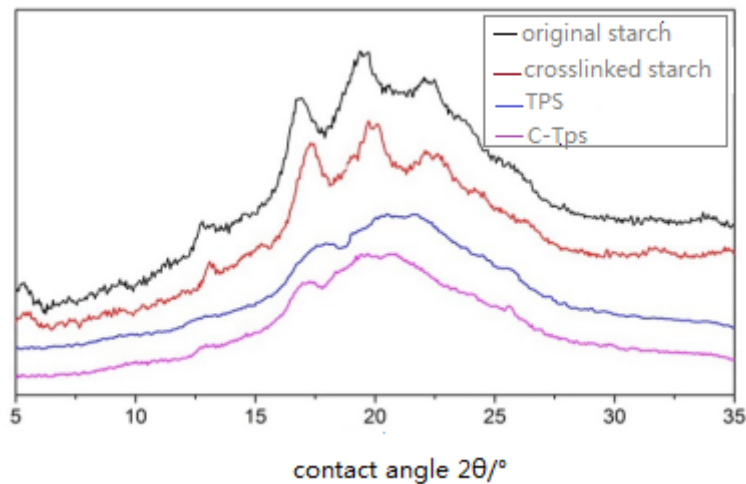
#### 3.1. Characterization of crosslinking starch

In order to further determine the morphology of the surface characteristics of original starch matrix after modification, TEM observation were conducted. A considerable difference between original starch and crosslinking starch with a mass fraction of 15 wt % in TEM observation (Fig.2 (a), (b), (c) and (d)) suggested a layer starch particles structure was formed on the starch material matrix surface randomly inside the liquid nitrogen, made the smooth surface of original starch became a microcrystalline fracture obviously.



**Figure 2.** SEM micrographs of (a) 120°C, 15%wt; (b) 130°C, 15wt %; (c) 140°C, 15wt % ; (d) 150°C, 15wt %

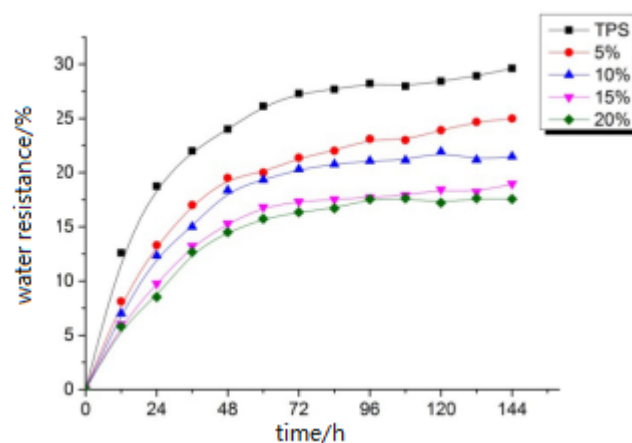
After crosslinking modification, we confirmed considerable chemical change of starch matrix by measuring XRD analysis spectrogram of the original starch, crosslinked starch, TPS and c-tps. Further characterization was carried out by XRD analysis. Figure 3 shows the differential spectrum which is obtained from original starch, crosslinked starch, TPS and c-tps, the spectrum was in good agreement with the original starch. Compared with the original starch matrix, the obvious appearance is the disappear of the diffraction spectrum diagram which can be seen that the crystallization of TPS, this is due to the small molecules of compound plasticizer into starch macromolecules and internal work, reduce the neat degree of starch molecules and would inhibit the recrystallization phenomenon at the same time [4].



**Figure 3.** XRD analysis of the original starch, crosslinked starch, TPS and c-tps.

### 3.2. test of crosslinking starch

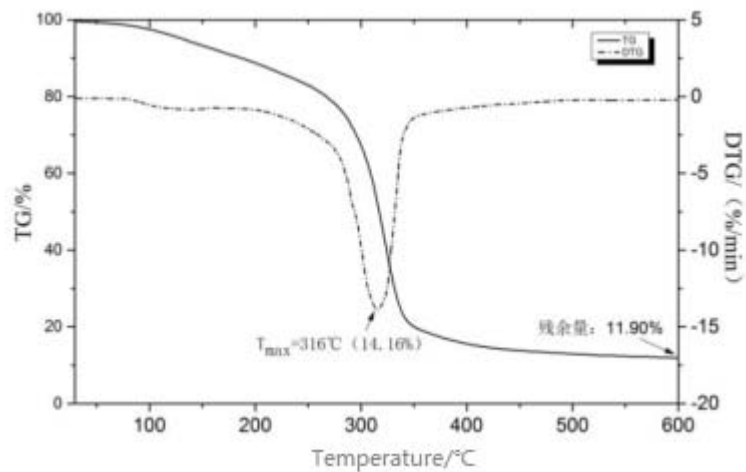
As shown in Figure 4, water resistance exhibited an increase from 0 to 29.61% with the crosslinking starch by 140 °C for different content preparation of the matrix material single screw extruder, which indicated that the water resistance of original starch matrix surface was greatly improved after crosslinking. After modification, it can be clearly seen from the figure that crosslinked starch can significantly improve the water resistance of matrix materials. The mixing of the mixture was 24.98%, 21.49%, 18.17% and 17.67%, respectively. The results pointed out that the crosslinking starch molecular polarity is low, under the processing conditions of 140 °C in matrix material distribution more uniform and to own a higher viscosity starch adhesive for mesh structure, which contribute its complete water resistance characteristic. Therefore, factors that may influence the water resistance processes should be considered to interpret the enhancement of the adding crosslinking starch hydrophilic group hydroxyl [5].



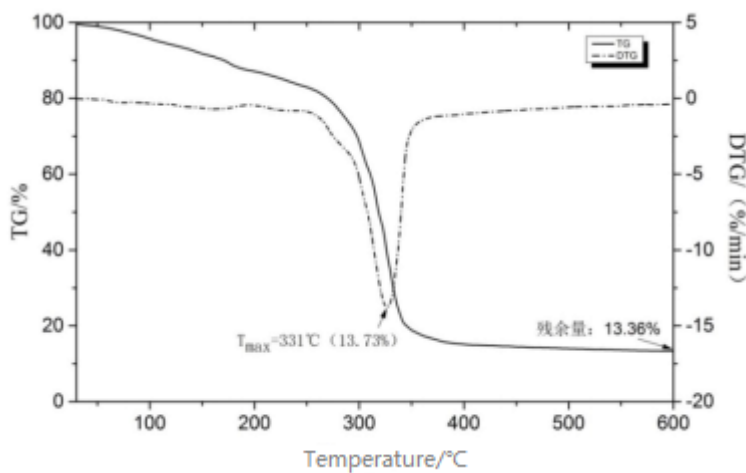
**Figure 4.** Water resistance of original crosslinking starch and crosslinking starch.

In order to study the stability of the TPS matrix against thermal conditions, the TG thermal analysis was carried out in the temperature range of 0 and 600°C at 10°C /min (Figure 5). The thermal

decomposition of sample was divided into two steps, including the decomposition of TPS matrix and C-TPS structure. The decomposition of TPS matrix from 100 °C to 280 °C; the decomposition of the C-TPS structure is from 280 °C to 350 °C, the peak melting temperature appears in the 315 °C. As it can be observed from these results, the C-TPS is stable up to 331 °C.



(a)

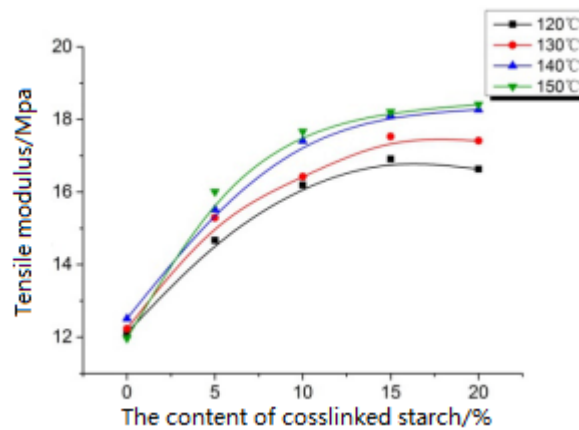


(b)

**Figure 5.** .TG curve of (a) TPS; (b)C-TPS, 15wt%, 140 °C

As shown in Figure 6, the physical mechanical of original starch matrix was very poor. It was reported that poor of physical properties and irreversible deformation of the original starch matrix was believed to be one of the main factors. It was found that the tensile modulus increased from 12.51 to 18.11 Mpa by adding oil-flax in its matrix, furthermore, the breaking strength and initial modulus by adding oil-flax in its matrix also showed the same trend, which indicated that the flexibility and elastic of crosslinking starch was improved significantly than that of the original starch matrix by adding oil-flax in its matrix. The crosslinked starch and starch mixed system helped the original starch matrix maintained its elasticity and capillary, hence improved its reusability. As a result, the physical

properties did not change significantly after regenerated, thus prolonging the lifetime of crosslinked starch [6].



**Figure 6.** Physical mechanical tests.

As shown in Figure 6, the physical mechanical of original starch matrix was very poor. It was found that the tensile modulus increased from 26.51 to 115.43 kg/cm<sup>2</sup> by adding oil-flax in its matrix with the crosslinking increased from 0 to 18.73%, furthermore, the breaking strength and initial modulus with the increasing of crosslinking also showed the same trend, which indicated that the flexibility and elastic of original starch matrix was improved significantly than that of the original starch matrix after modification. Adding oil-flax in its matrix helped the original starch matrix maintained its elasticity, hence improved its reusability. As a result, the physical properties did not change significantly after regenerated, thus prolonging the lifetime of starch matrix. The starch matrix after modification was still able to maintain its initial physical mechanical capacity (Table 1).

**Table 1.** Physical mechanical properties of starch matrix with different crosslinking by adding oil-flax in its matrix

Crosslinking (%)	Tensile modulus (kg/cm <sup>2</sup> )	Breaking strength (cN)	Initial modulus (cN/dtex)
0	26.51	0.71	0.35
6.4	43.28	1.22	0.48
9.5	76.41	2.48	0.92
15.4	99.85	3.14	1.24
18.73	115.43	3.76	1.48

#### 4. Conclusion

After mixing cross-linked starch, in the role of the plasticizing effect of the compound plasticizers and the shearing force of single screw, tensile strength of the mixed-starch matrix increased 44.76%, moisture absorption decreased 38.64 %. This proves that cross-linked starch has significantly enhanced effect on matrix. When the content of cross-linked starch increased over 15wt%, the increase in the tensile properties of matrix material trended to flat. Considering, the 15wt% content of cross-linked starch is most reasonable. On the basis of the above results, it could be concluded that the crosslinking thermoplastic starch was an effective biodegradable plastics material sorbent for the textile industry.

**Acknowledgments**

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**References**

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