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Effect of processing conditions on cell structure of starch-based foam

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Abstract. In this work, the effects of processing conditions on the cell structure were studied for starch foam through investigating the effect of water content and processing temperature and shear stress on starch-based materials. It was found that there was a critical point of water content (between 16-18%) where the expanding ratio changed significantly since the cell structure changed from open to closed. The lower water content was, the higher Tm of starch-based materials was, which resulted in lower melting strength and open cell structure. Conversely, the higher water content and the lower Tm resulted in higher melting strength and closed cell structure. The closed cell structure prevented moisture evaporation during foaming and kept the lower Tm, which resulted in foam shrinking when the materials was cooled down since negative pressure in the cell. The higher temperature not only increased the steam pressure but also decreased the melt strength of starch-based materials, which enhanced the open cell structure.

1. Introduction
Starch is the most promising natural polymer because of its inherent biodegradability, overwhelming abundance and annual renewability [1-9]. Starch-based polymers offer a very attractive low cost base for new biodegradable polymers due to their low material cost and ability to be processed with conventional plastic processing equipment [10, 11]. Since more and more countries have started to ban foam packaging materials from non-biodegradable resources, starch-based foam is an ideal replacement for these non-biodegradable materials, in particular application as loss fill. Due to the multiphase transitions of starch, the microstructures and mechanical properties of starch-based materials strongly depend on the processing techniques and conditions.

During starch foaming, a part of water containing in starch-based materials becomes gaseousness to create bubbles when the temperature is higher than boiling point of water, while other part of water acts as plasticizer to gelatinize starch and provide melt strength enabling the starch-based materials expansion to form foam structure. Phase transitions, thermal and physical properties of starch and dough or paste have been extensively studied. Previous investigations have showed that both glass transition (Tg) and melting (Tm) temperature, and physical properties, such as viscosity, melting strength and mechanical property, strongly depended on water content. However, there is less research focusing on how the water acts as both blow agent and plasticizer at same time during foaming. In this work, the effects of processing conditions on the cell structure were studied for starch foam through
investigating the effect of water content and processing temperature and shear stress on starch-based materials.

2. Experimental

2.1. Materials
The cornstarch supplied by Hengrui Starch Company (Luohe, China, with moisture content 13%w) was used in this work. The calcium carbonate (Yifeng Company, Guangzhou, China) was used (2%w) as a nucleation agent to ensure the uniformity of foaming cells. Tap water was used as plasticizer.

2.2. Sample preparation
In this work, the starch foams were prepared by two-step processing: 1) compounding starch-based pellets using a twin screw extrusion with co-rotating mixing screws (Keya, Nanjing, China) plasticized by injected water using a metering pump. 2) Starch foaming: the starch-based pellets with different moistures produced from first step were fed into a single screw extruder (Tongjia, Jining, China) with 35mm diameter and 30 L/D ratio. A die nozzle with 3mm diameter connected with the single screw extruder was heated to at 200°C.

2.3. Characterizations
A differential scanning calorimeter (DSC), Perkin Elmer DSC 4000 equipped with a low-temperature accessory, was used to study the thermal properties of the starch-based pellets with different moisture. The samples (~8mg) were weighed in high-pressure stainless steel pans and sealed with a gold-plated copper seal to avoid moisture evaporation. The samples were heated from 50°C to 200°C at a heating rate of 10°C/min. The water contents in the samples were measured by a moisture content apparatus MB-35 from Hongji Instruments (Shanghai China) at 130oC. The cell structures of starch foams materials were studied using a SEM (ZEISS, Oberkochen, Germany) that was operated at a voltage of 10.0kV. The specimens were cut from cross-section and tuck on a circular metal stubs with double side adhesive tape and then coated with gold using an Eiko Sputter Coater under vacuum.

3. Results and Discussions
Figure 1 shows the DSC thermograms of the starch-based pellets with different water content. It could be seen that all DSC thermograms presented phase transitions occurring over a quite broad temperature range due to the melting points with on-going evaporation of water from starch pellets. The results clearly showed that $T_m$ of the starch pellets increased gradually with decreasing water content as expected, similar as the glass transition temperature ($T_g$) of TPS, which has been widely reported. It has been noticed that the endothermal capacity of water evaporation decreased and the initial temperature of evaporation increased with decreasing water content in TPS. That indicated the lower water content containing in TPS the more difficulty of water was evaporated, which meant the water and hydroxyl group in starch molecular could form more the hydrogen bonds with a certain amount of water. When the water content was higher, there would be more and more free water evaporated easily. Previously study has also confirmed the lower water content in starch-based pellets the more chances of the starch molecular chain internally connected and crystallized via hydrogen bonds, where the water could be strictly inhibited and wrap in TPS resulting in lower speed of water evaporation.

The cell structures of the starch-based foams have been studied by SEM and showed in Figure 2. It was seen that all cells interconnected with each other and presented the same void patterns in successive slices. Open cell structure with diameter about 0.5-0.8 mm could be clearly identified in all samples. It has been noticed that thickness of the cell wall for sample containing lower water content (11%) was higher, which could be explained by the un-melted starch since the lower water content. This phenomenon was corresponded with surface observation. The sample containing meddle water content (13%) showed uniformed cell structures with thinner wall. The sample containing higher water
content (15%) showed uniformed cell structures with thinner wall. When the water content is larger than 18%, closed cells were obtained, in which the shrunken cells could be clearly identified, which resulted in higher density materials.

![Figure 1](image1.png)

**Figure 1.** Effect of water content on melting temperature (Tm) of starch-based materials (water content was increased from A to G).

![Figure 2](image2.png)

**Figure 2.** Photographs of starch foams prepared from starch-based pellets with different moisture contents (left: 11%; middle: 13%; right: 15%), respectively.

### 4. Conclusions

During starch foaming water acted as both blow agent and plasticizer at same time. There were generally two regions when the starch came out from extruder die: 1) foaming; and 2) cooling. It was found that there was a critical point of water content (between 16-18%) where expending ratio decreased significantly since the cell structures changing from open to closed cell. The lower water content was, the higher Tm of starch-based materials was, which resulted in lower melting strength and open cell structure. Conversely, the higher water content and the lower Tm resulted in higher melting strength and closed cell structure.

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