

## **CHARACTERIZATION OF STARCH PHARMACEUTICALS BY DSC COUPLED TO A PHOTOVISUAL SYSTEM**

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### **Abstract**

The present work proposes evaluation of the gelatinization processes of starch by means of DSC coupled with a photovisual system. The use of DSC, TG and DTA for a fast and efficient evaluation of the starch is suggested. The DSC curves of starch gels with water contents of 20, 30, 40 and 50% (mass/v) exhibited different phase transitions, corresponding to the gelatinization processes at the different water contents for the different lots. The DSC-photovisual system confirmed calorimetric behaviour differences between the starch lots studied.

**Keywords:** DSC-photovisual, gelatinization, starch

### **Introduction**

In most tablet formulations, starch comprises a significant proportion by mass of the total composition. Starch powders consolidate to form compacts primarily through plastic deformation, and the compacts also demonstrate some elasticity, with time-dependent behaviour [1–6]. The physicommechanical properties of starch are influenced by the chemical composition, the degree of polymerization, the degree of crystallinity, the moisture content, the particle size and the shape [2, 3]. The chemical, physical and mechanical properties of starch have been thoroughly investigated by numerous authors [4, 5]. The available data allow comparisons of the different starch sources. However, the data do not include thermal properties, though the recently introduced DSC-photovisual system technique is now available to the pharmaceutical industry.

### **Experimental**

Four lots of starch were obtained from the storeroom of the Laboratory of Pharmaceutical Technology of the Federal University of Paraíba. Samples were sieved to

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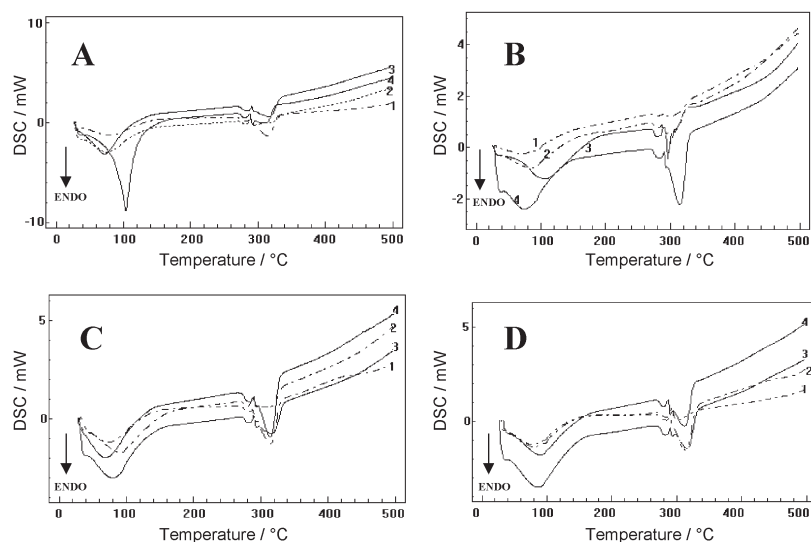
100 mesh, collected in porcelain cups, mixed with distilled water in proportions of 20, 30, 40 and 50% (mass/v) and homogenized for 10 min.

The DSC curves of the starch were obtained with a Shimadzu calorimeter, model DSC-50, coupled to a photovisual system consisting of an Olympus microscope connected to a Sanyo camera, model VCC-D520, under a nitrogen flow of  $50 \text{ ml min}^{-1}$ , at a heating rate of  $5^\circ\text{C min}^{-1}$ , up to  $500^\circ\text{C}$ .

These parameters were based on the conventional laboratory program and an appropriate method. Transition temperatures were recorded from a plot of heat flow vs. temperature ( $30\text{--}500^\circ\text{C}$ ). The peak temperature ( $T_p$ ) of gelatinization was determined from the DSC curve with Tasys software from Shimadzu. The reaction heat was determined by using the area of the peaks between the onset temperature ( $T_0$ ) and the end-temperature from the DSC curve. Samples ( $2.00 \text{ mg}$ ) were weighed to the nearest  $\pm 0.01 \text{ mg}$  and sealed in aluminium pans. The images were captured by means of DSC coupled to the photovisual system under similar conditions of conventional DSC. Medium and standard deviation ( $sd$ ) values were determined from triplicates of DSC curves. The instrument was calibrated via the melting points of indium ( $156.6\pm 0.3^\circ\text{C}$ ) and zinc ( $419.6\pm 0.3^\circ\text{C}$ ) standards. The heat flow and enthalpy were calibrated via the heat of fusion of indium ( $28.59\pm 0.30 \text{ J g}^{-1}$ ) under the same conditions as for the samples.

## Results and discussions

Starch contains an abundance of hydroxy groups; each anhydro glucose unit contains two secondary hydroxy groups and a large majority contain primary hydroxy groups. The hydroxy groups are potentially able to react with any chemical capable of react-



**Fig. 1** DSC curves of different lots of starch with water contents (mass/v) of 50% (A), 40% (B), 30% (C) and 20% (D); 1 – Lot 01, 2 – Lot 02, 3 – Lot 03, 4 – Lot 04

ing with water. All starches are hygroscopic and rapidly absorb atmospheric moisture [7]. The DSC curves of starch gels containing 20, 30, 40 and 50 (mass/v%) water (Fig. 1) displayed the distinct and characteristic endothermic peaks of the gelatinization processes (Table 1). There were significant differences between the endothermic peaks of the different starch lots. Starches from different plant sources differ in amylose/amylopectin ratio. These differences modify the physical properties of the starches and the various types may not be interchangeable for a given pharmaceutical application [8].

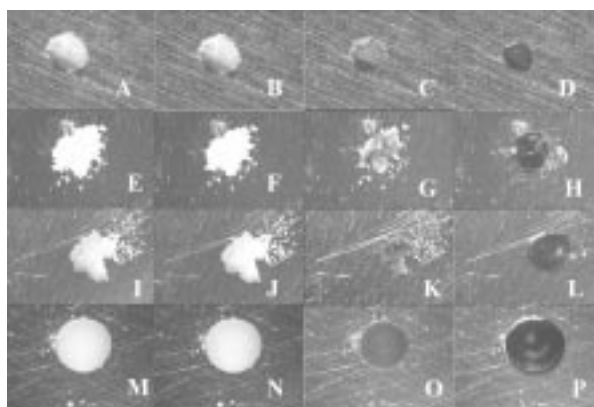
**Table 1** Gelatinization temperatures of different lots of starch

Water content/ mass/v/%		Lot number			
		01	02	03	04
50%	$T_p/^{\circ}\text{C}$	79.4 <sup>(a1)</sup>	72.3 <sup>(a2)</sup>	75.8 <sup>(a3)</sup>	104.2 <sup>(a4)</sup>
	$T_0/^{\circ}\text{C}$	33.5	43.6	37.7	42.6
	End/ $^{\circ}\text{C}$	122.4	115.9	112.5	128.6
	Reaction heat/ $\text{J g}^{-1}$	233.5 <sup>(b1)</sup>	345.9 <sup>(b2)</sup>	388.7 <sup>(b3)</sup>	395.8 <sup>(b4)</sup>
40%	$T_p/^{\circ}\text{C}$	73.7 <sup>(c1)</sup>	79.9 <sup>(c2)</sup>	92.9 <sup>(c3)</sup>	68.8 <sup>(c4)</sup>
	$T_0/^{\circ}\text{C}$	37.6	48.9	46.5	38.5
	End/ $^{\circ}\text{C}$	119.0	158.7	116.6	115.7
	Reaction heat/ $\text{J g}^{-1}$	194.7 <sup>(d1)</sup>	351.2 <sup>(d2)</sup>	343.4 <sup>(d3)</sup>	387.4 <sup>(d4)</sup>
30%	$T_p/^{\circ}\text{C}$	78.3 <sup>(e1)</sup>	86.4 <sup>(e2)</sup>	79.4 <sup>(e3)</sup>	91.4 <sup>(e4)</sup>
	$T_0/^{\circ}\text{C}$	40.0	39.2	43.3	45.0
	End/ $^{\circ}\text{C}$	121.3	123.3	137.9	199.2
	Reaction heat/ $\text{J g}^{-1}$	180.8 <sup>(f1)</sup>	217.8 <sup>(f2)</sup>	330.3 <sup>(f3)</sup>	299.5 <sup>(f4)</sup>
20%	$T_p/^{\circ}\text{C}$	68.2 <sup>(g1)</sup>	71.2 <sup>(g2)</sup>	74.3 <sup>(g3)</sup>	85.0 <sup>(g4)</sup>
	$T_0/^{\circ}\text{C}$	38.5	46.2	43.3	41.7
	End/ $^{\circ}\text{C}$	115.7	116.0	137.9	133.3
	Reaction heat/ $\text{J g}^{-1}$	119.2 <sup>(h1)</sup>	159.1 <sup>(h2)</sup>	138.8 <sup>(h3)</sup>	169.1 <sup>(h4)</sup>

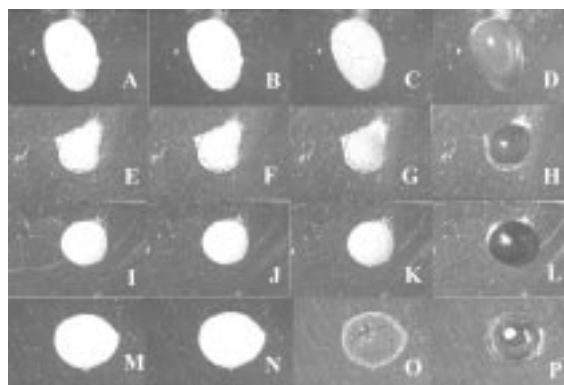
Standard deviation (a1–a4) (*sd*) of ( $\pm 0.4, 0.2, 0.5, 0.3$ ); (b1–b4) *sd* of ( $\pm 1.3, 1.4, 1.7, 1.5$ ); (c1–c4) *sd* of ( $\pm 0.2, 0.4, 0.5, 0.3$ ); (d1–d4) *sd* of ( $\pm 1.3, 1.6, 1.4, 1.5$ ); (e1–e4) *sd* of ( $\pm 0.6, 0.4, 0.5, 0.3$ ); (f1–f4) *sd* of ( $\pm 1.5, 1.3, 1.7, 1.3$ ); (g1–g4) *sd* of ( $\pm 0.2, 0.6, 0.3, 0.5$ ) and (h1–h4) *sd* of ( $\pm 1.5, 1.3, 1.7, 1.3$ )

Lot 1 (Fig. 2) underwent no alteration in volume and no change in colour at 68°C, corresponding to the gelatinization (picture B), but darkening occurred at temperatures above 268–295°C (pictures C and D). Lot 2 (pictures E, F, G and H) exhibited a small volume expansion at around 72°C, corresponding to gelatinization (picture F), followed by the evolution of gases at 295°C (picture H). Lot 3 did not reveal an alteration in volume or a change in the initial colour at 74°C (picture J). Lot 4 (pictures M, N, O and P) maintained the same initial volume up to 85°C (picture N) and exhibited darkening at 268°C (picture O), followed by decomposition with a volume reduction at temperatures above 295°C (picture P), due to volatilization.

Studies with the calorimeter coupled to the DSC-photovisual system (Fig. 3) on the four lots of starch gels with a water content of 50% revealed the most marked physical and chemical changes as a function of temperature. Pictures A, B, C and D, corresponding to lot 1, demonstrated a well-defined thermal behaviour in the gelatinization process (picture B). Lot 2 (pictures D, E, F and H) underwent slight darkening at 268°C (picture O), followed by decomposition with a volume reduction at temperatures above 295°C (picture P). Lots 3 and 4 displayed different thermal behaviour. Pictures J, K and L, corresponding to lot 3, revealed its well-defined thermal behav-



**Fig. 2** Pictures of starch gels with a water content of 20%, obtained by means of DSC coupled to the photovisual system. Lot 1: pictures A (room temperature) B (68°C), C (268°C) and D (295°C). Lot 2: pictures E (room temperature), F (71°C), G (268°C) and H (295°C). Lot 3: pictures I (room temperature), J (74°C), K (268°C) and L (295°C). Lot 4: pictures M (room temperature), N (85°C), O (268°C) and P (295°C)



**Fig. 3** Pictures of starch gels with a water content of 50%, obtained by means of DSC coupled to the photovisual system. Lot 1: pictures A (room temperature) B (79°C), C (268°C) and D (295°C). Lot 2: pictures E (room temperature), F (72°C), G (268°C) and H (295°C). Lot 3: pictures I (room temperature), J (75°C), K (268°C) and L (295°C). Lot 4: pictures M (room temperature), N (104°C), O (268°C) and P (295°C)

our in the gelatinization process at 75°C (picture J) with decomposition (picture K). Lot 4 (pictures M, N, O and P) manifested a gelatinization process with volume expansion followed by the evolution of gases at 295°C (picture P).

The DSC-photovisual system revealed the phase transitions and allowed the microscopic characterization of the starch gels subjected to increasing temperature. Figures 2 and 3 for starch gels with water contents of 20 and 50% evidenced the following stages: (a) the granules hydrate and swell (b), the mixture undergoes a colour change, (c) there is a rapid increase in volume, and (d) linear molecules diffuse out of the ruptured starch granules as they decompose.

Analysis of the data obtained from DSC coupled to the photovisual system confirmed differences between the starch lots. The comparison of starch samples from four different lots revealed the value of thermal analysis in the quality control of the raw material.

## Conclusions

Analysis of the data furnished by DSC coupled to a photovisual system confirmed differences between starch lots from the same manufacturer.

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