

## The role of drying process on corn-starch dust explosion

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The design of dust explosion venting and suppression systems requires the knowledge and then the evaluation of the thermo-kinetic parameters as the deflagration index ( $K_{St}$ ) and the maximum attainable pressure ( $P_{max}$ ), by standardized experiments. However, during industrial processes dusts may undergo structural changes which affect their hazard. In order to fully characterize the risk linked to their handling, the evaluation of the explosion parameters should be then evaluated keeping into account these effects. In the present work, we performed an experimental study for the explosion of several corn-starch samples obtained by different drying processes generally employed in the food and pharmaceutical industries.

### 1. Introduction

In several industrial food and pharmaceutical processes, starch may undergo to several transformation by means of grinding, heating, drying, depending on the final required properties (molecular structure, particle size, humidity content).

With specific reference to the food industry, starch undergoes to cycles of heating and drying treatment (Carter et al., 1997). The heating temperature is higher than the gelatinization temperature ( $T \geq 50^\circ\text{C}$ , Sablani et al., 2007) where the starch changes its structure (hot drying). However, gelatinization of starch is required for food applications because it makes starch accessible for enzymes.

On the other hand, when considering the manufacture of solid pharmaceutical such as tablets, granules, pills and capsules, starch should be dried avoiding the gelatinization process (Takeo et al., 1983). Hence, the drying process is performed at a temperature lower than gelatinization-starting temperature. Hence, vacuum drying method and the vacuum freeze-drying method are used (Takeo et al., 1983; Spieles et al., 1995).

Whatever the drying processes (vacuum drying, vacuum freeze-drying, and hot drying) the starch structure and properties are completely different with respect to the initial un-handled starch and then the correspondent explosion hazard may vary significantly. It is indeed acknowledged that the explosion properties depends on the dust structures and physical properties (Eckhoff, 2003).

Despite this consideration, many literature data for the explosion parameters of standard corn-starch have been published, but the hazard of the “processed –corn-starch” is not accomplished.

In the present paper we produce different samples of corn-starch by changing the drying process treatment. We then perform explosivity tests to evaluate the effect of the drying process on the explosion parameters ( $K_{St}$  and  $P_{max}$ ).

## 2. Experimental

### 2.1 Materials

The corn starch was purchased by Sigma Aldrich (CAS 9005-25-8). We performed the drying of the corn starch in two methods. The first method consists on drying the corn starch by heating at 57°C for about one hour in vacuum conditions ( $P=86$  mmHg) in a Rotavapor system, Laborota 4002 Heidolph; the dried sample obtained is named Sample A. The second method consists on drying the corn starch at ambient temperature ( $T=20$  °C) in vacuum for 7 h and then lyophilizing it at -50°C and  $10^{-4}$  mmHg for 15h (sample B).

We then characterized the as-received sample (sample C) and the dried samples (samples A and B) in terms of water content, particle images, size distribution and explosivity.

### 2.2 Equipments and methods

Laser granulometry Sympatec HELOS SUCCELL was utilized for the determination of particle size distributions of samples A, B and C. Water or ethanol was used as solvent. The sample structure was analysed by means of the Scanning electron microscopy (SEM, Philips mod. XL30).

Water contents of corn starch samples was determined by simultaneous TG/DSC (TA Instruments SDTQ600) coupled with a mass-spectrometer QMS 200 (PFEIFFER VACUUM). Tests were performed in the temperature range 25-230°C, at heating rate of 10 K/min and air flow rate of 100 cm<sup>3</sup>/min (STP).

The nearly spherical '20 l sphere', with rebound nozzle introduced by Siwek (1985) was used for the explosivity tests of corn starch (Figure 1). In the spherical vessels the ignition source is located in the centre of the sphere and the dust is injected from a separate container. Two pyrotechnic igniters (5kJ) and ignition delay of 60 ms were adopted. The experimental procedure has followed ASTM E1226 (2000).

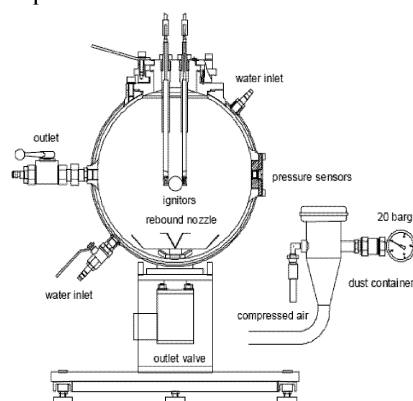


Figure 1. Siwek 20l equipment for the determination of dust explosion parameters.

### 3. Results

Figure 2 shows the cumulative and particle size distribution of the three analyzed corn starch samples. Results of particle distribution obtained by using water as solvent are similar to those obtained by using ethanol, and are not reported here for the sake of brevity. From Figure 2, the median diameter of starch results to be about 15  $\mu\text{m}$ , and only a very small fraction of particles has a diameter less than 5  $\mu\text{m}$ . The distribution is mono-modal and equal for all samples. Hence, the sample treatment does not alter the particle size distribution.

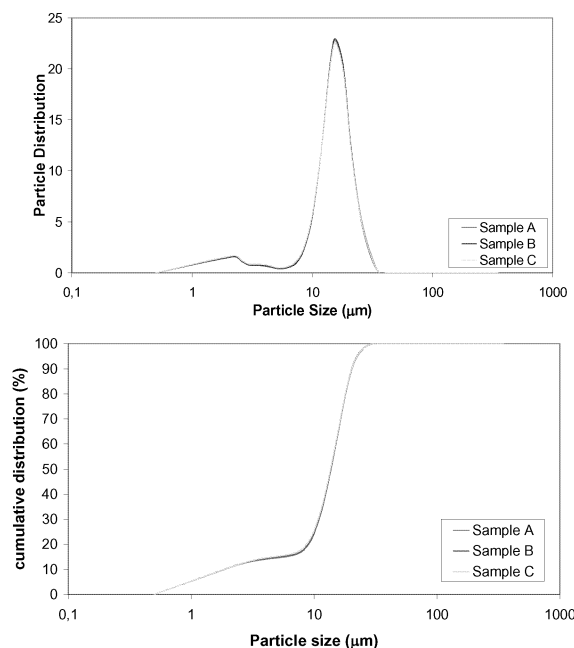


Figure 2. Particle and cumulative distribution as measured by laser granulometry.

In Figure 3, the results of TG and DSC analyses are reported. In the temperature range investigated, all the samples show a weight loss and a correspondent endothermic DSC signal due to the loss of water. This result has been confirmed by MS signals (here not reported). More specifically, the weight loss of corn starch as received (sample C) was 12.1 % while that of dried samples was 10.5 % and 8.0 %, respectively for sample A and B. Moreover, it is worth noting that the derivative weight, and the correspondent heat flow, shows a peak at 70 °C for sample C and for slightly dried sample A, whereas for the peak is shifted at higher temperature (about 85°C) for sample B. This result demonstrates that during lyophilization treatment (sample B), the more loosely linked water is lost.

#### 3.1 Explosion test results

We tested all the three samples in Siwek bomb in order to evaluate the deflagration index ( $K_{St}$ ) and the maximum pressure ( $P_{max}$ ). In Figure 4 the measured deflagration index ( $K_{St}$ ) is plotted vs. the dust concentration for all samples.

The un-treated sample (C) has the highest  $K_{St}$  value. Conversely, the dried samples have lower  $K_{St}$  values at all the concentrations investigated. In particular, the structural change during drying (sample A) affects the deflagration index of corn starch more effectively with respect to water content (sample B).

The maximum values of  $K_{St}$  and  $P$  are attained at a concentration equal to about  $1000 \text{ g/m}^3$  which is significantly higher than the stoichiometric concentration reported in the literature ( $C = 230 \text{ g/m}^3$ ). This apparent discordance may be due to particle size and agglomeration tendency of corn starch. To this regard, Figure 6 shows  $K_{St}$  values reported in the literature versus the particle diameter. The  $K_{St}$  obtained in this work, for all samples, are also shown.

For most of the literature data no information are available about the moisture content and the structure of the sample. The literature data of Figure 6 are summarized in Table 1. It is important noting that the data of van Wingerden & Stavseng, (1996) are not reported Figure 6 as consistently off the trend.

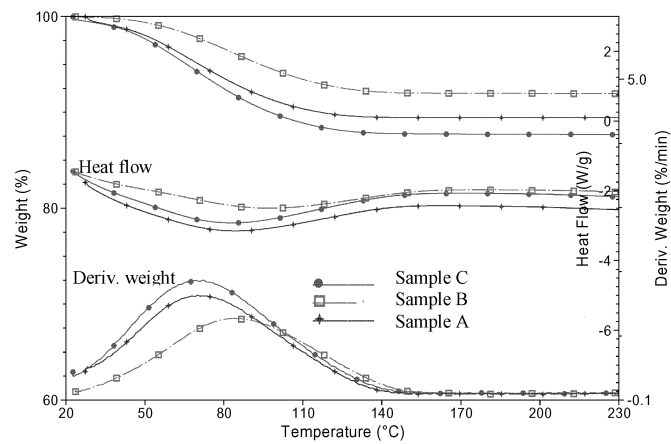


Figure 3. TG/DSC curves of different corn starch samples.

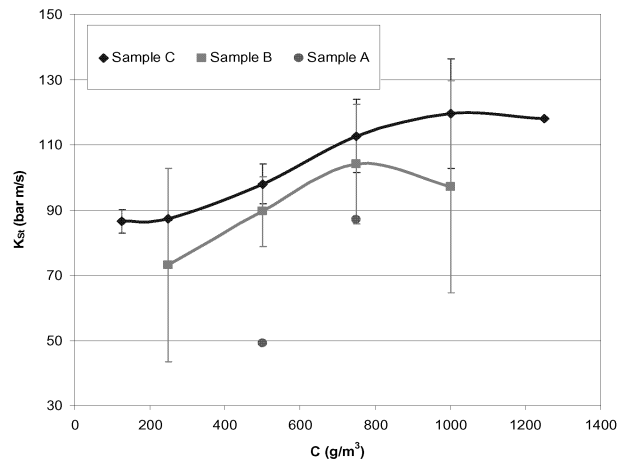


Figure 4 – Deflagration index of corn-starch samples as function of the dust concentration measured in the Siwek bomb (20 lt).

In Figure 5 the maximum pressure is also plotted vs. the dust concentration for all the samples.

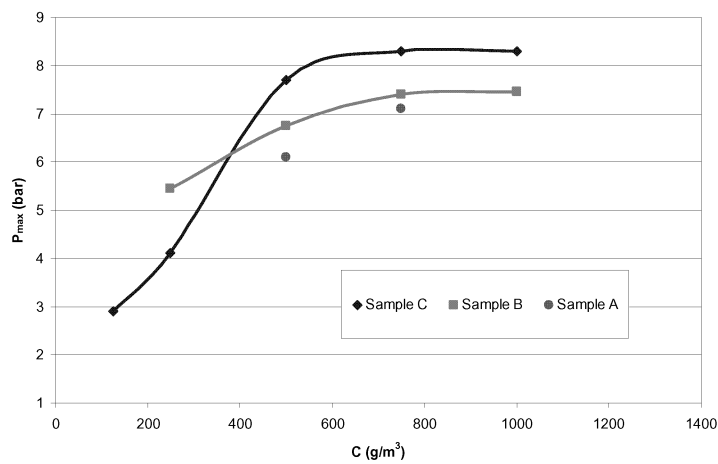


Figure 5 – Maximum pressure of corn-starch samples as function of the dust concentration measured in the Siwek bomb (20 l).

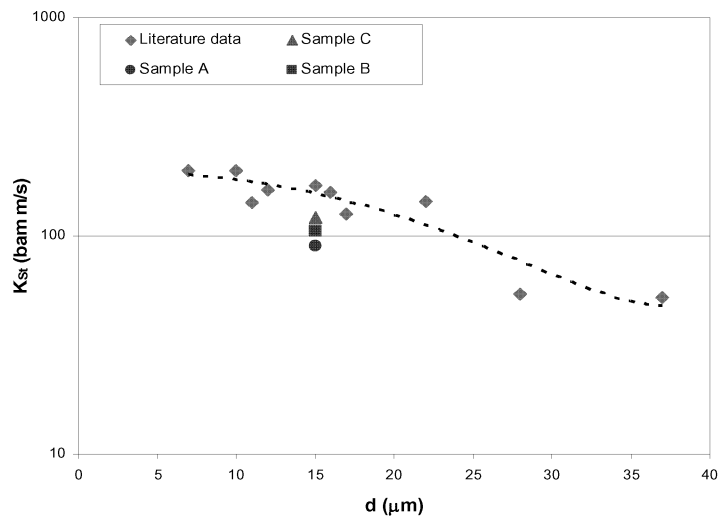


Figure 6 – Comparison between  $K_{St}$  values present in the literature and obtained in this work as function of the particle diameter.

In Table 2 the properties of the investigated samples are given. It is found that it is not possible to link the trend of  $K_{St}$  to the moisture content.

Table – 1 –  $K_{St}$  values as function of particle diameter – Literature data

Reference	d ( $\mu\text{m}$ )	$K_{St}$ (bar m/s)	moisture (%)
<i>NFPA 68</i>	7	200	not specified
<i>Proust, 1993</i>	28	54	not specified
<i>Eckhoff, 2003</i>	37	52	11.0
<i>BG, 2007</i>	10	200	not specified
	11	141	
	12	161	
	15	169	
	16	158	
	17	125	
	22	143	
<i>Eckhoff, 1991</i>	16	158	not specified
<i>van Wingerden &amp; Stavseng, (1996)</i>	50	161	dried

Table 2 – Summary of samples investigated properties.

Sample	d ( $\mu\text{m}$ )	H <sub>2</sub> O (% wt.)	$K_{St \text{ max}}$ (bar m/s)
A	15	10.5	90
B	15	8.0	100
C	15	12.1	110

To get insight into this behavior, Figure 7 shows the SEM images of all the analysed corn-starch samples. In the case of raw corn starch (Figure 7, top) particles are linked, forming agglomerates whose dimension is about 20  $\mu\text{m}$ . When decreasing the water content by drying process, the agglomeration of particles increases, where agglomerates of dimensions of about 60 and 80  $\mu\text{m}$  are visible, respectively (Figure 7, medium and bottom).

From these results it may be argued that lower value of  $K_{St}$  exhibited at lower water content (sample A and B) has to be addressed to the higher propensity of these sample to give rise to agglomerates.

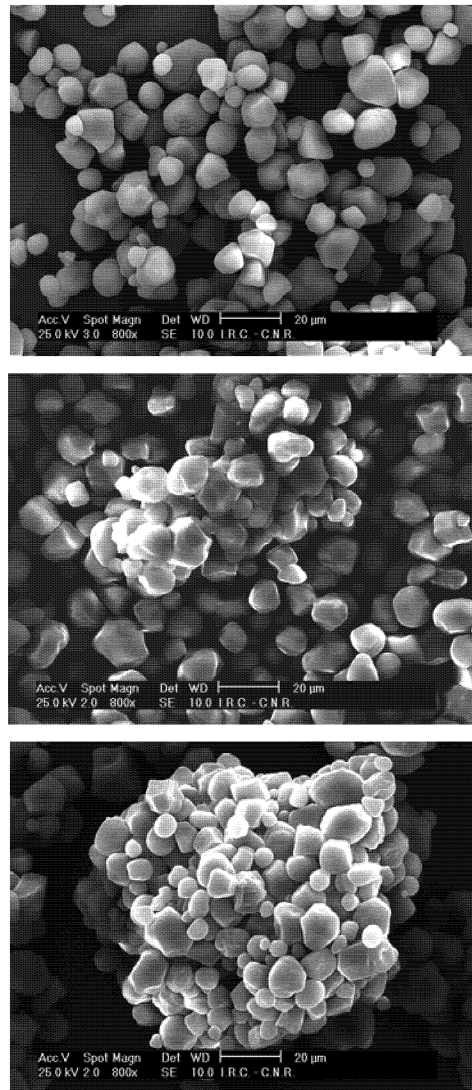


Figure 7. SEM image of sample C (top); sample B (medium) and sample A (bottom)

#### 4. Conclusions

The study of the hazard of corn-starch dust has been performed by measuring the deflagration index and the maximum pressure in the Siwek bomb (20 l).

Different samples of corn-starch have been obtained by means of typical drying processes encountered in the food and pharmaceutical industries. It was found that the hazards of the corn-starch dust may decrease depending on the drying method, as  $K_{St}$  reaches the value of 90 bar/m s starting from 120 bar m/s for the un-dried sample.

The hazard of corn-starch dust is then strongly dependent on the method of processing. Care must be taken when conservative choices for the dust explosion counter-measures are designed.

## 5. Acknowledgments

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## 6. References

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