

## THERMAL ANALYSIS AND SAFETY IN RELATION TO FOOD PROCESSING

A. Raemy, P. Lambelet and J. Loeliger

Research Department, NESTEC S.A., CH-1800 VEVREY (Switzerland)

### ABSTRACT

The adaptation of thermal analysis techniques to study exothermic phenomena and self-ignition of foodstuffs, and the use of special tools to simulate and analyze dust explosions are described. The examples shown should provide a better understanding of the mechanisms involved in phenomena which can lead to the bursting of an autoclave, to fires or to dust explosions during food processing operations.

### INTRODUCTION

It is well-known (ref.1) that risks of exothermic reactions, fires and explosions are of concern in organic powder products such as foods as well as for vapours or gases. Today, many methods enabling the study of exothermic phenomena, self-ignition, combustion and dust explosions can be used to analyze these phenomena in foods; in particular, thermal analysis makes important contributions.

### THERMAL ANALYSIS OF EXOTHERMIC PHENOMENA

The instruments for studying exothermic reactions can simply be containers fitted with sensors which follow the rising temperature of a heating bench, or autoclaves with additional pressure sensors. But techniques such as differential thermal analysis (DTA), differential scanning calorimetry (DSC) or adiabatic calorimetry allow one to obtain more detailed thermodynamic information, especially enthalpies.

However, thermal analysis techniques sometimes have to be applied in unusual ways so as to effect measurements under conditions close to those of the process being studied. Thus for instance, Fig. 1 shows the plan of an experiment designed to study food samples in contact with supercritical CO<sub>2</sub> by high pressure DTA, i.e. at a temperature above 31°C and a pressure above 72.9 bar. It can be used to simulate solid-liquid extraction with supercritical gases as solvent (ref.2).

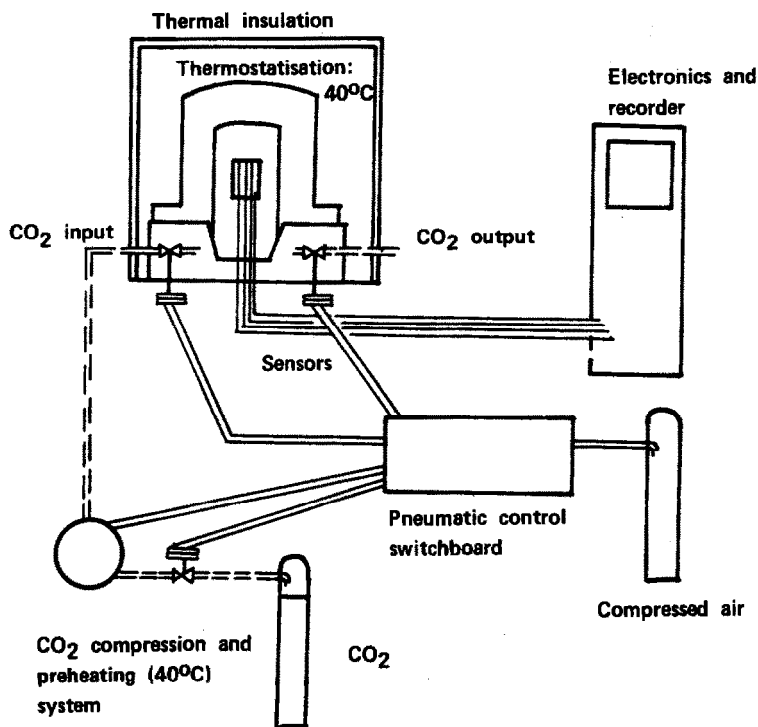


Fig. 1 Schematic of a DTA experiment under supercritical CO<sub>2</sub>. High pressure DTA instrument Netzsch.

Furthermore, in order to detect exothermic phenomena corresponding to sample pyrolysis, it is often useful to modify the atmosphere above the sample by increasing the pressure (of an inert gas) or to analyze the food by heating it in sealed cells. This prevents endothermic phenomena such as vaporization of the water remaining in the powder and can maintain the sample in a condensed phase at temperatures higher than its usual boiling point (ref.3). Fig. 2 presents the calorimetric curve of cellulose, a commonly found food carbohydrate, heated in a sealed cell. Thermal decomposition, shown by the exothermic peak, is completed below 300°C, which is not the case in a cell open to atmosphere (ref.4). The measured enthalpy of decomposition is approximately 650 Jg<sup>-1</sup>.

To detect oil or fat oxidation, it is desirable to increase oxygen availability by analyzing the samples under oxygen flow or oxygen pressure (ref.5).

Similar considerations apply in calorimetric studies concerning fermentation; aerobic as well as anaerobic conditions have to be fulfilled, depending on the kind of microorganism investigated (ref.6).

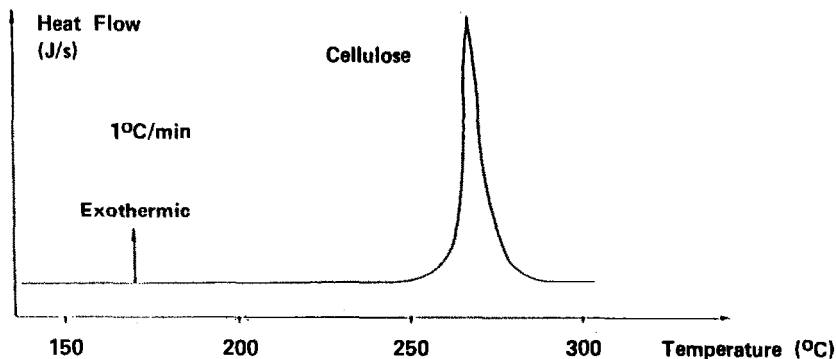


Fig.2 Calorimetric curve of cellulose heated in a sealed cell. Heat flow calorimeter Setaram C80.

#### SELF-IGNITION, COMBUSTION

Exothermic phenomena such as decomposition or fat oxidation can lead to self-ignition of the product if sufficient oxygen is available. Several techniques can be used for studying self-ignition of a sample; the simplest are plates or ovens heated to known temperatures (ref.7). Today, some thermal analysis instruments also allow the analysis of products under ignition conditions, i.e. either by sweeping the sample with air or oxygen, or by keeping it under oxygen pressure (ref.8). The parameter determined by these techniques is the ignition temperature of a powder in deposit (or in layer).

To find safe parameters of industrial operations, in particular temperature ranges, it is important to analyze the processed products under particularly stringent conditions. As an example, Fig. 3 presents the DTA curve of cellulose (Fluka AG, Buchs, Switzerland; No. 22197) heated and burnt under 25

bar of oxygen. Before saturation, the  $\Delta T$  curve shows the thermal runaway leading to self-ignition at a very low temperature: 240°C. When measuring

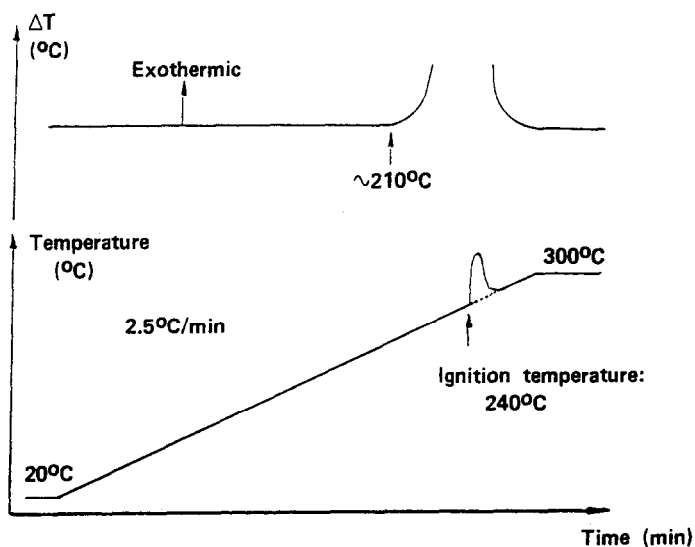


Fig.3 DTA curves of cellulose heated and burnt under 25 bar of oxygen. High pressure DTA instrument Netzsch.

under oxygen pressure, self-ignition temperatures will decrease with increasing pressure. In Fig. 4, the influence of oxygen pressure between 5 and 25 bar on self-ignition temperatures of cellulose powder is shown. At pressures lower than 4 bar of oxygen, cellulose did not ignite. It is not surprising, if one considers the very drastic conditions, that all temperatures indicated in Fig. 4 approach the lowest value found in the literature : 254°C (ref.9).

Finally, the bomb calorimeter enables the study of the combustion of foods: an adequate igniting system and high oxygen pressure guarantee maximum combustion; the heats of combustion obtained are about  $39 \text{ kJg}^{-1}$  for fat,  $23 \text{ kJg}^{-1}$  for protein and  $17 \text{ kJg}^{-1}$  for carbohydrate.

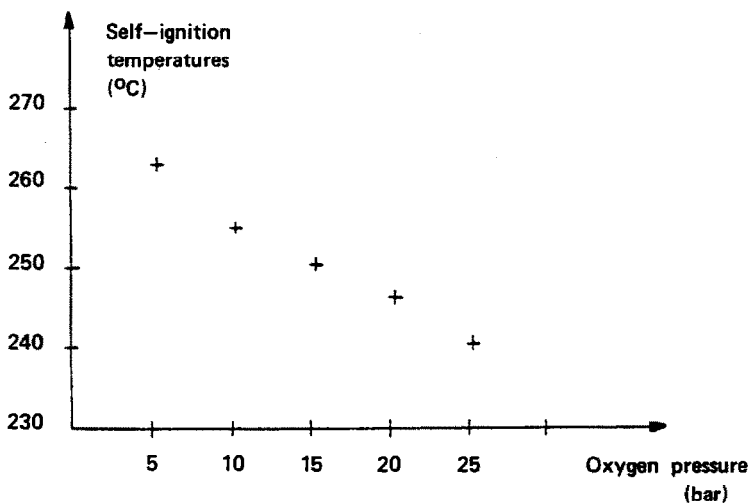


Fig.4 Self-ignition temperatures of cellulose as a function of oxygen pressure. High pressure DTA instrument Netzsch.

#### SIMULATION OF DUST EXPLOSIONS

Several types of tools allowing simulation and analysis of dust explosions (closed containers of volume  $V$  ( $m^3$ )) are described in the literature (ref.1,10): among the best known are Hartmann's tube, the 20 l sphere and the  $1 m^3$  cylinder. In these instruments, the product is dispersed before being ignited. Several parameters are determined from the recording (see Fig. 5) of rising pressure as a function of time, i.e. : maximum explosion pressure (bar), maximum pressure increase rate (bar/s), minimum and maximum explosive concentrations ( $g/m^3$ ). The  $K_{ST}$  value (bar m/s), a dust characteristic, is obtained from the maximum pressure increase rate by the formula :

$$(dp/dt)_{\max} \cdot V^{1/3} = K_{ST}$$

This value enables one to classify particular products into categories expressing the violence of a possible explosion; it is also used for calculating vent areas. Finally the minimum ignition energy (J) of a powdered product is determined with the help of electrical sources of ignition of known energy.

Data concerning dust explosion parameters of food products can be found in the literature (ref.10,11).

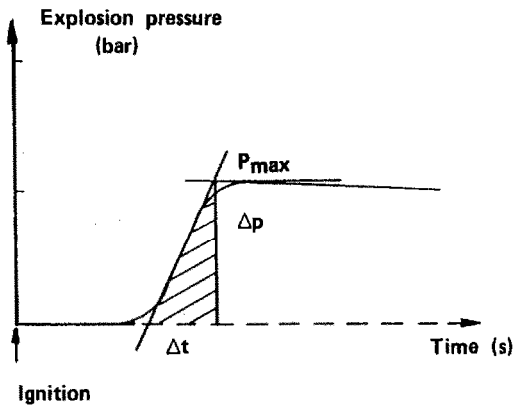


Fig.5 Explosion pressure rise as a function of time.

#### CONCLUSION

Today it is certain that thermal analysis techniques give valuable information on safety problems in relation to food processing at elevated temperatures. The operations mainly concerned are roasting, solid-liquid extraction and in particular high-temperature drying. A thorough knowledge of the physical and chemical properties of the foods helps in defining adequate preventive measures, thus providing for greater safety in these industrial operations.

#### ACKNOWLEDGEMENTS

The authors gratefully acknowledge many discussions with I. Horman and the skilled help of Miss I. Froelicher in the practical work.

#### REFERENCES

- 1 K.N. Palmer, *Dust explosions and fires*, Chapman and Hall, London, 1973.
- 2 G.M. Schneider, E. Stahl and G. Wilke, *Extraction with supercritical gases*, Verlag Chemie, Weinheim, Germany, 1980.
- 3 R.J. Seyler, *Thermochim. Acta*, 41 (1980) 55.
- 4 D. Dollimore and J.H. Hoath, *Thermochim. Acta*, 45 (1981) 87.
- 5 R.L. Hassel, *JAACS*, 53 (1976) 179.
- 6 A.E. Beezer, *Biological microcalorimetry*, Academic Press, New York, 1980.
- 7 E.C. Synnott, J.B. Glynn and T.C. Duane, *J. of Food Engineering* 3 (1984) 151.
- 8 A. Raemy and J. Loeliger, *Thermochim. Acta*, 85 (1985) 343.
- 9 D. Davies, A.R. Horrocks and M. Greenhalgh, in D. Dollimore (Ed.), *Proc. 2nd Eur. Symp. Therm. Anal.*, Aberdeen, 1981, Heyden, London, 1981, p. 588.
- 10 W. Bartknecht, *Explosionen*, Springer Verlag, Berlin, 1980.
- 11 P. Field, *Dust explosions*, Elsevier Scientific Publishing Co., Amsterdam (1982).