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# SELFIGNITION OF POWDERS STUDIED BY HIGH PRESSURE DIFFERENTIAL THERMAL ANALYSIS

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## ABSTRACT

The technique of differential thermal analysis was used, in an original way, for studying spontaneous ignition and combustion of powders. The measurements were performed with a high-pressure differential thermal analysis instrument. Selfignition temperatures of powders were determined by analysing the samples under a high pressure of oxygen (25 bar). Selfignition temperature values are given for powders which are of interest in the food industry.

## INTRODUCTION

Dust explosions and fires are known hazards (ref.1-2) in many different industries, in particular also in the food industry. This powder inflammability study tries to elaborate better defined safety conditions for high-temperature processing operations such as roasting.

Studies of spontaneous combustion of powders very often have involved sample deposition on a heating plate or into a furnace (ref.3) heated to a known temperature. The data obtained by these tests are generally compiled as minimum ignition temperatures for dust layers.

It has been proposed recently (ref.4-5) to perform similar measurements of selfignition temperatures by using differential thermal analysis (DTA) techniques with oxygen atmosphere. The samples were heated under air flow or in static air. The advantages are the well-defined measuring conditions, in particular the precise heating rates possible with DTA instruments.

In the present study high-pressure DTA was used; many different food materials were heated under 25 bar of oxygen.

## EXPERIMENTAL PROCEDURES

Instrument. The instrument used in this study was the 404 H high-pressure DTA apparatus [constructed by Netzsch following a Claudy and Bousquet (C.N.R.S.) patent]. The furnace and measuring systems are presented in Fig. 1. The furnace is surrounded by a water cooled high-pressure autoclave. The gas volume around the furnace is about 400 ml. The complete equipment has already

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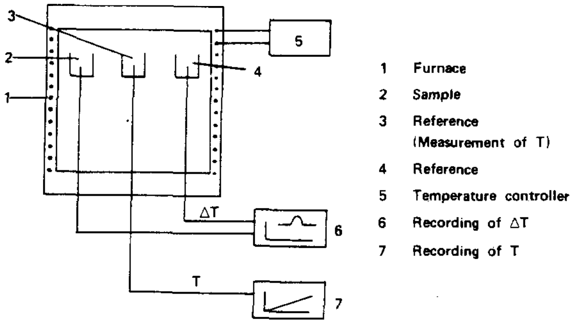


Fig. 1 Schematic representation of the Netzsch DTA instrument

been described (ref.6). It can be used for analysing samples in atmospheric air or under constant pressures of gas. Heating rate was generally 2.5 °C $min^{-1}$ and the temperature range between 20 and 300 °C was scanned.

Samples. Samples of about 0.5 g were analysed. All materials were of commercial origins. Particle size was determined with the Hiac PA-720 instrument.

RESULTS AND DISCUSSION

In an oxygen atmosphere, many powders could be brought to spontaneous ignition. To assure a complete combustion of the sample, a large excess of oxygen was required. To obtain this complete combustion of the food material studied, 25 bar of oxygen pressure was chosen. These conditions also reduce endothermic phenomena such as vaporisation of the water often contained in food products.

During burning of the product in the DTA furnace a great amount of energy is liberated (about 39 $kJg^{-1}$ for fat, 23 $kJg^{-1}$ for protein and 17 $kJg^{-1}$ for carbohydrate) : it is the heat of combustion of the food material. Its value is generally determined with calorimetric bombs. Because of this considerable heat release, the ΔT-curve, normally exploited, increases until saturation. During sample combustion, however, the temperature curve shows an exothermic peak or "spike" because the furnace (see Fig. 1) is heated above the programmed temperature value. The temperature where this peak appears indicates ignition of the product. Precise determination of the selfignition temperature is possible, and because of the well-defined conditions, good reproductibility is obtained. Fig. 2 shows the combustion of whole wheat flour in 25 bar oxygen : the upper curve is the saturated ΔT-response, the lower curve is the furnace response measured by the thermocouple under the third cell

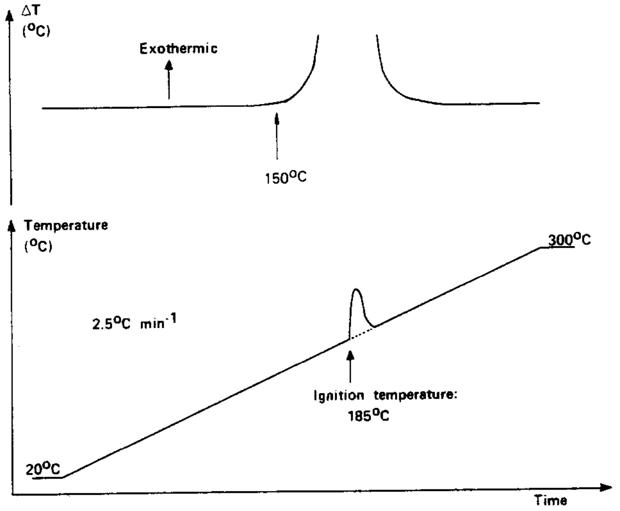


Fig. 2 DTA curves of whole wheat flour heated and burned under 25 bar of oxygen.

(filled with the same inert material as the reference cell). Depending on the sample, this furnace temperature can rise, during the combustion to about 10 to 40 °C above the programmed temperature.

During heating and even during sample combustion, the pressure stays virtually constant inside the fixed volume autoclave.

Table 1 shows the results obtained for the different food constituents and for some composite foods.

TABLE 1. Selfignition temperatures of foods determined by DTA. Atmosphere, 25 bar oxygen.

| Products | Diameter (μm) | Selfignition temperatures (°C) |
|---|---|---|
| Guar gum | 94 | 190 |
| Cellulose | 78 | 240 |
| Maltodextrin MD 33 | 63 | 260 |
| Cocoa butter | --- | 240 |
| Palm oil | --- | 245 |
| Linseed oil | --- | 215 |
| Whey protein | 227 | 195 |
| Casein | 57 | 185 |
| Chicory (dried) | 265 | 160 |
| "Soluble" cocoa | 45 | 155 |
| Cocoa shells | 195 | 160 |
| Soya (freeze dried) | --- | 150 |
| Wheat flour (whole) | 163 | 185 |
| Wheat bran | --- | 170 |
| Whole milk powder | 156 | 150 |

Carbohydrate. Carbohydrate which melt, like sucrose or fructose, did not ignite spontaneously. Only polysaccharides like cellulose, maltodextrins or gums ignite under the applied conditions. Iota-carrageenan explodes without burning (only 20 % of the sample weight is lost at the end of the run), at about 165 °C.

Fat. Selfignition of fats and oils, at elevated temperatures (generally above 200 °C) is a well known phenomenon. Our results confirmed this fact despite the rather unfavourable conditions for selfignition (low contact surface between oil and oxygen).

Protein. Two samples of milk protein (casein and whey protein) were analysed. Both ignited spontaneously at relatively low temperatures (around 190 °C).

Composite foods. A great deal of food samples were studied. Some of them, like milk powder, "soluble cocoa" or dried chicory roots ignited spontaneously at low temperatures (around 160 °C). Other foodstuffs, in particular whole beans or grains did not ignite under our conditions.

## CONCLUSION

By using the Netzsch high-pressure DTA equipment in an original way, the spontaneous combustion of powders is clearly detected, selfignition temperatures are precisely determined and the measuring conditions are well-defined.

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