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# THERMOANALYTICAL STUDIES OF HEAT AND ENZYME TREATED CEREAL PRODUCTS

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## ABSTRACT

The detection of heat damage with DSC is possible after separation of the temperature resistant components of the grain, e.g. on gluten. For sample preparation a new way has been chosen which differs from the frequently described methods for testing protein-water systems.

The reactions of gluten in the range 20 to 200°C are endothermal. Undamaged gluten shows a 5 to 40% higher reaction enthalpy than damaged gluten (dependent on the kind of damage).

The endothermal reaction peak temperatures for untreated gluten samples are higher (ca. 120°C). With the DSC differences on two gluten samples could be detected which could not be observed with former test methods

The heat influence on grain changes the structure of the protein and this can be clearly detected by DSC.

## INTRODUCTION

Drying grain to produce supplies of grain suitable for bread making is in many areas a necessity. The reason for the current study of the drying process is that expensive energy is needed for artificial drying, the time pressure during harvest, and the possibility of heat damage to the grain. Topics of interest are

- drying temperatures
- drying media to remove the moisture
- detection of heat damage.

The removal of moisture at predetermined temperatures and in controlled atmospheres is an ideal application for thermogravimetry and simultaneous thermal analysis. Tests on the complete grain are necessary to maintain its relevance to the real situation. Only by using large sample quantities can the natural variations of single grain analysis be avoided.

Using calorimetric methods it is possible to detect heat damage on grain after separation of the temperature resistant components of the grain, e.g. on flour, even better on gluten.

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## EXPERIMENTAL

Heat damage was studied on wheat, wheat flour and wheat gluten. The necessary damage on the complete grain was partially carried out by rapid heating in the thermobalance at exactly reproducible grain temperatures.

Gluten was available as pre-dryed powdery product, as well as in wet plastic state (table 1).

Of the powdery samples marked A1 and A2, A1 was untreated and A2 enzymatically damaged. The wet gluten samples, marked R10 and R13, were both of the summer wheat type "Ralle", R13 was obtained from wheat grain which had been treated at 150°C for 5 minutes. Further wet gluten samples C15 and C17 were obtained from untreated flour (C15) as well as from flour (C17) treated at 150°C for 5 minutes. All gluten samples were vacuum dryed at ambient temperature and treated for the same time (10 min) before thermal analysis independent of their different initial moisture contents.

The drying process on wet wheat was recorded by the thermobalance (Netzsch STA 409). For this sample crucibles with a volume to 3,4 $cm^3$ were used to be able to analyse simultaneously a larger number of grains (approx. 40 wheat grains). Heating rates were up to 2 K/min, the atmosphere used was dry air (*dynamic 100 $cm^3/min$*).

The tests on flour and gluten were carried out using a heat flux DSC (Netzsch DSC 444) in a static nitrogen atmosphere in the temperature range 20 ... 200°C at a heating rate 5 K/min. Sample crucibles used were open pans of pure aluminium, sample quantities were 20 ... 70 mg for gluten, the reference cell was empty. The DSC 444 was operated with the data acquisition system 414/1 and computer HP 85 with plotter. Peak integration in preselectable ranges and determination of characteristic temperatures was carried out automatically with this data system.

## RESULTS AND DISCUSSION

The vacuum drying of the gluten samples already shows differences between treated and untreated samples. Independent to their initial moisture contents, there were noticeable differences in mass loss between treated and untreated gluten: damaged samples always showed a higher mass loss than untreated samples (table 1).

Table 1

| Gluten | mass loss |
|---|---|
| Powdery samples | |
| A 1 untreated | 1,45% |
| A 2 enzym. damaged | 1,6 % |
| Plastic samples | |
| R 10 untreated | 14,3 % |
| R 13 heat damaged | 37,3 % |
| C 15 untreated | 10,9 % |
| C 17 heat damaged | 14,1 % |

The results obtained from vacuum dried samples indicate that when damaged the water is less tightly bonded to gluten. This effect can be very clearly noticed from the high initial moisture of wet gluten samples R10 and R13 as well as C15 and C17.

The trace for gluten in the temperature range 20 ... 200°C (fig. 1) shows an endothermal peak. The untreated dry gluten A1 shows an endothermal peak with the peak onset at 30°C and ending at 170°C. Peak temperature is 92,5°C. The damaged gluten A2 shows a similar peak (fig. 2) with peak temperature at 95°C. However, the reaction enthalpy for the damaged gluten is much smaller; it is reduced by 29% compared with the untreated gluten.

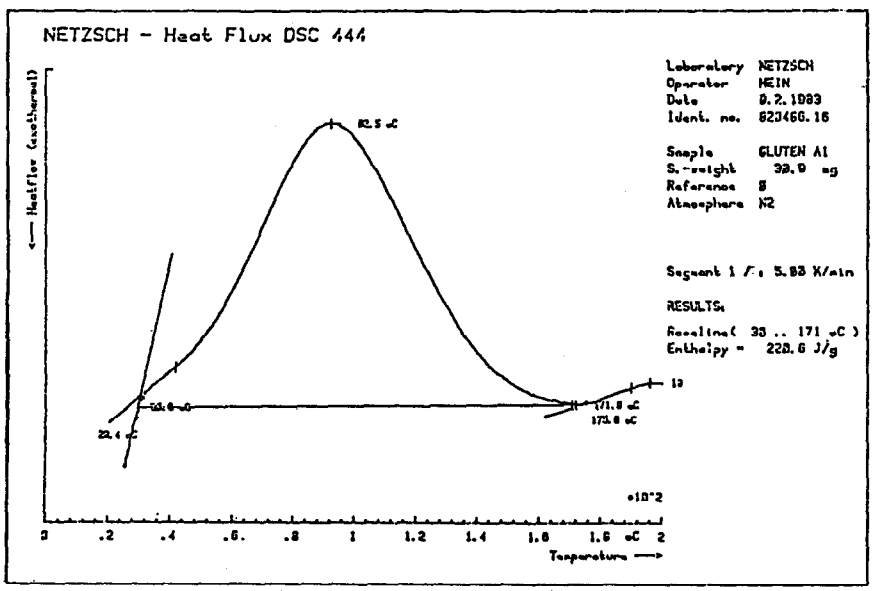


Fig. 1. Wheat gluten A1 (dry gluten) endothermal reaction enthalpy 220,6 J/g

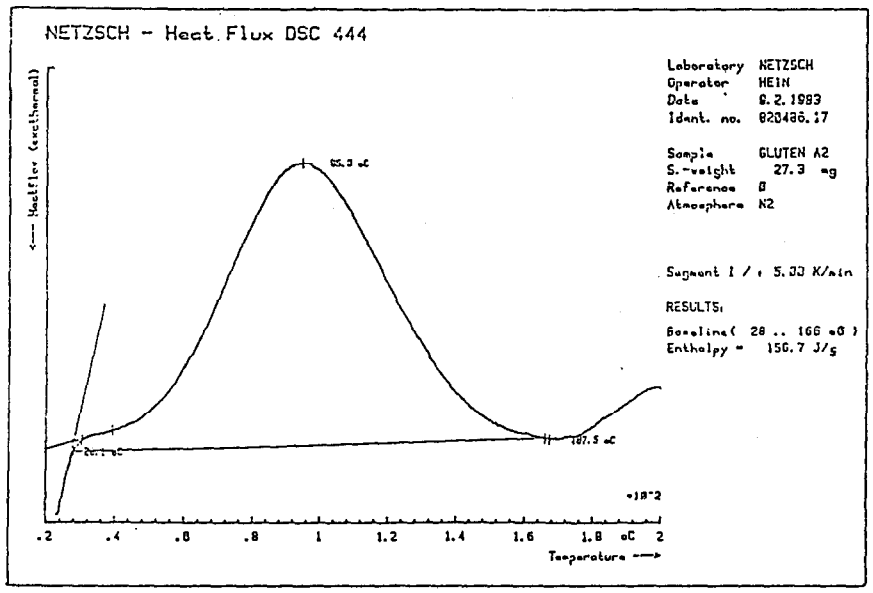


Fig. 2. Wheat gluten A2 endothermal reaction enthalpy 156,7 J/g

Untreated wet gluten samples always showed a structured (double-) peak with main maximum at approx. 120 ... 130°C (fig. 3). Treated wet gluten - obviously dependent on the degree of damage - gave lower peak temperatures, a simpler peak shape (R13) and lower reaction enthalpy. Fig. 3 shows a direct comparison of the untreated sample R10 and the heat-treated wet gluten R13.

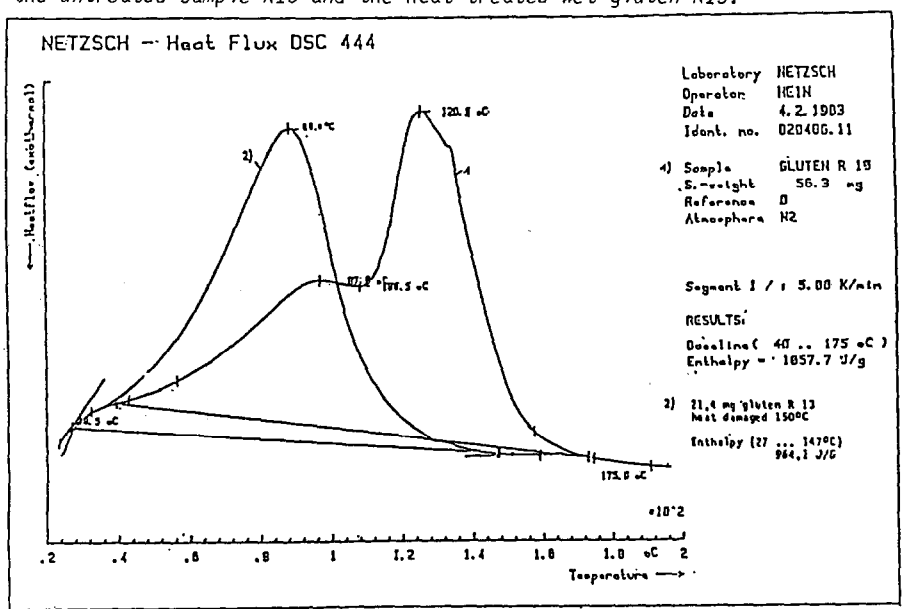


Fig. 3. Wheat gluten R10 endothermal reaction enthalpy 1057,7 J/g
Wheat gluten R13 endothermal reaction enthalpy 964,1 J/g

For the other plastic gluten C17 (fig. 4) which was washed out of heat-treated flour (150°C, 5 min) the corresponding distinct differences were not found between the treated and untreated samples C15 and C17 (fig. 5).

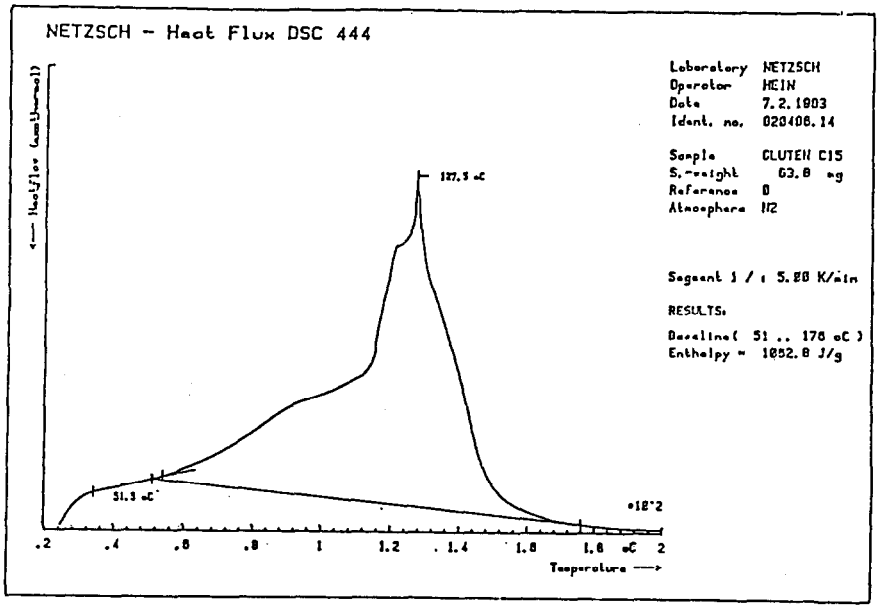


Fig. 4. Wet gluten C15 endothermal reaction enthalpy 1062,8 J/g

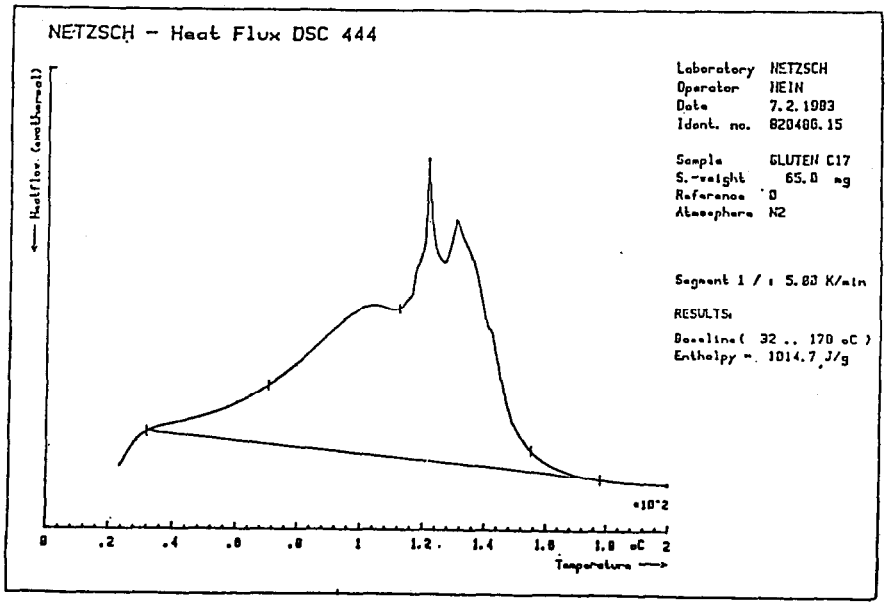


Fig. 5. Wet gluten C17 endothermal reaction enthalpy 1014,7 J/g

## CONCLUSION

Treating flour and grain under the same conditions, i.e. 150°C for 5 min in a thermobalance results in greater damage to the gluten extracted from grain than that extracted from flour.

These subtle structural damages can be clearly seen using DSC. For gluten samples the DSC shows better results than commonly used test methods which can determine no difference at all for the samples extracted from flour.

The basic structure changes of gluten are the subject of further work.

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