

# Effect of microwave radiation on physico-chemical properties and structure of potato and tapioca starches

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The time–temperature profiles of selected starch–water systems subjected to microwave processing were established and the effect of microwave radiation on the physico-chemical properties and structure of potato and tapioca starches was studied. The experimental starch samples were examined by the Brabender rheological method, light microscopy, scanning electron microscopy and X-ray diffractometry. Microwave radiation was evidenced to affect the temperature and moisture contents of the experimental starches, with a strong correlation between the moisture content and the rate of temperature rise. An isothermal transformation was revealed with the samples of moisture contents over 20%, causing a rise in the gelatinisation temperature of the starch and a drop in its solubility in water. The most pronounced change was observed in the case of potato starch: its crystal structure changed from type B to type A. The tapioca starch underwent similar but less marked changes. © 1998 Published by Elsevier Science Ltd. All rights reserved

## INTRODUCTION

There are a number of applications in the food industry to which microwave processing can be applied. Since it is quite competitive in cost compared with other methods of heating, it has been used for thawing of frozen foods, drying, baking, rendering, pasteurisation and sterilisation. Microwave irradiation seems applicable to starch processing but thus far it has not been used on a commercial scale.

Microwaves are known to be a non-ionizing energy capable of generating heat deep inside the penetrated medium by the “molecular friction” in an alternating electromagnetic field. Thus any design of microwave processing must include not only thermal properties of the product to be processed, which are relatively insensitive to temperature differences, but also a number of interrelated electric properties, which vary extensively with the processing frequency and time–temperature profiles of the product. At microwave frequencies, the basic electric characteristics, i.e. the dielectric constant and the loss factor, are largely determined by the salt and moisture contents of the

product (Mudgett, 1986). Therefore dry food components, e.g. unmodified starches, are thought to be electrically inert (Miller *et al.*, 1991). Most of the experiments carried out on starch–microwave radiation interaction have pertained to the systems of high water content (Rashed Khan *et al.*, 1979; Zylema *et al.*, 1985; Miller *et al.*, 1991). The only research done on the dextrinisation and chemical modification of starches by microwave irradiation is that reported by Muzimbaranda & Tomasik (1994).

The chemical modification of solid starches is usually carried out in rotating roasters. The basic parameters to be considered while designing the process include the temperature and moisture content of the reaction mixture. The suggestion of applying microwave ovens instead of rotating roasters seems promising, but requires a detailed study of the effect of microwave processing on starch. The purpose of this study was to establish the time–temperature profiles of selected model starch–water systems subjected to microwave processing and to examine the effect of microwave radiation on the physico-chemical properties and structure of potato and tapioca starches.

## EXPERIMENTAL

### Microwave irradiation of starch samples

Commercial potato (superior standard, produced in Poland) and tapioca (super high grade, produced in Thailand) starches were air-dried or moistened to obtain samples of adequate moisture. 200 g samples were irradiated in 600 ml glass beakers, open or sealed with a perforated polyethylene foil designed for microwave ovens. The irradiation was carried out in a DeLonghi microwave oven (Italy), 800 W microwave output power and 2450 MHz microwave frequency. The experiments were effected with about 10% output power (the

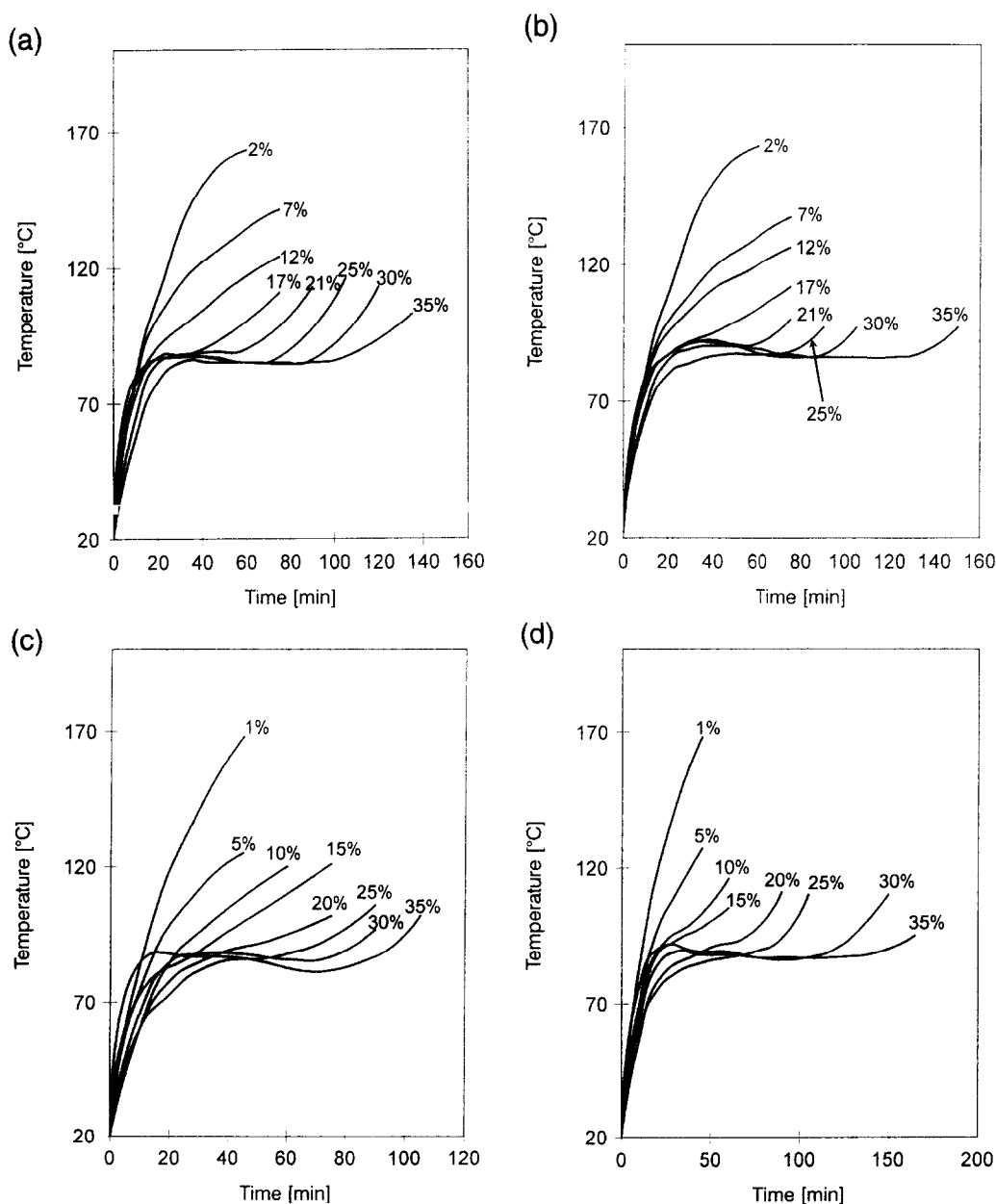
minimum level). The temperature of the starch layer was taken periodically with a mercury thermometer.

### Rheological properties

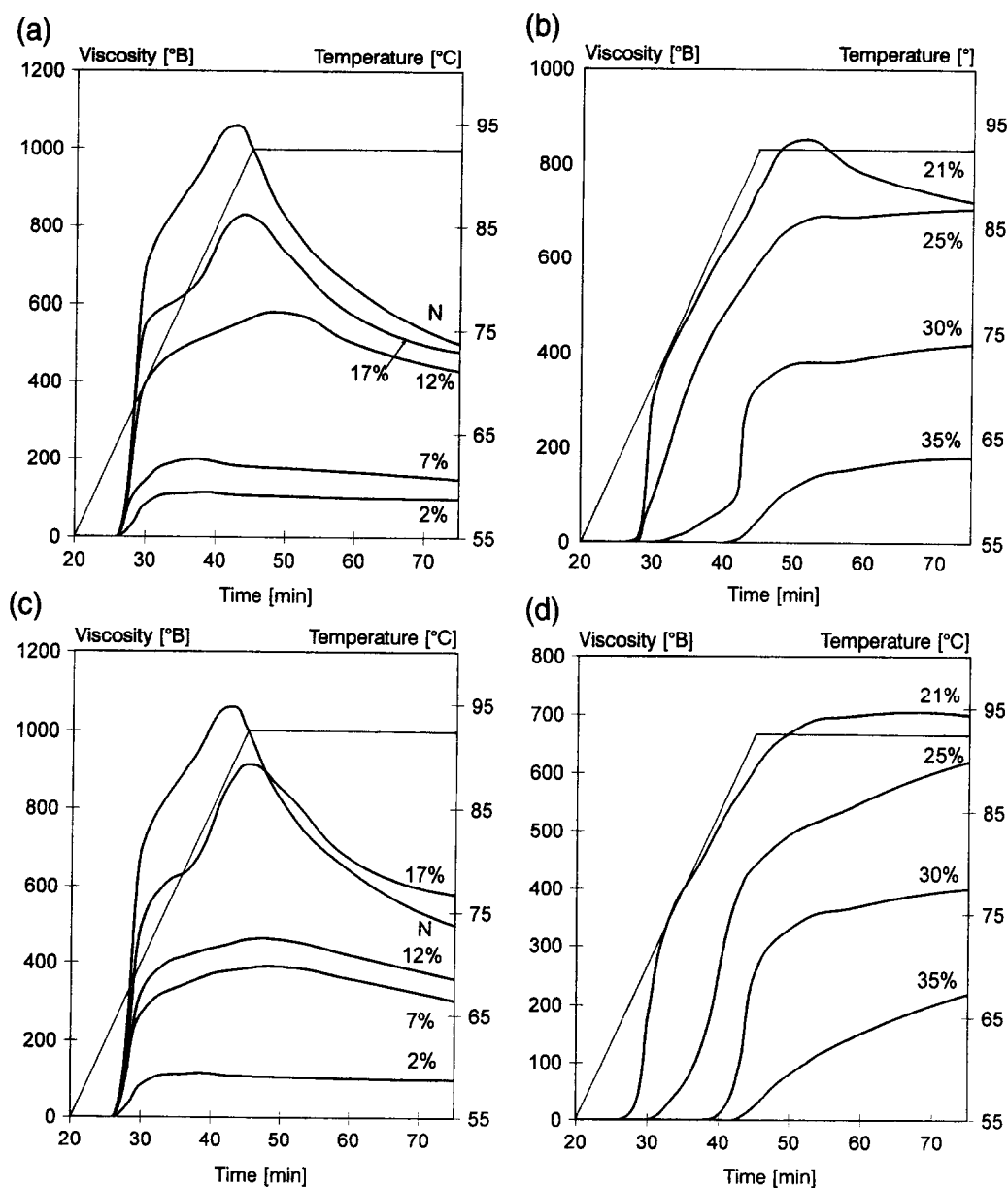
The course of gelatinisation was followed with a Brabender viscograph under the following conditions: measuring cartridge 0.07 Nm, heating/cooling rate 1.5°C/min, thermostating 30 min.

### X-ray diffractometry

X-ray diffractometry was effected with an X-ray diffractometer type TUR 62 Carl Zeiss Germany under



**Fig. 1.** Time-temperature profiles of potato starch irradiated in open (A) or in sealed (B) beakers and of tapioca starch irradiated in open (C) or in sealed (D) beakers.



**Fig. 2.** Brabender viscosity curves for 5% solutions of microwaved potato starch of moisture contents 2–17% irradiated in open beakers (A), of moisture contents 21–35% irradiated in open beakers (B), of moisture contents 2–17% irradiated in sealed beakers (C) and of moisture contents 21–35% irradiated in sealed beakers (D). N = native starch.

the following conditions: X-ray tube CuK $\alpha$  (Ni filter), voltage 30 kV, current 15 mA, scanning from  $\Theta = 2$  to  $18^\circ$ .

### Microscopic examination

The starch samples to be examined by light microscopy were prepared by the smear method. To this end starch suspensions were heated at the initial gelatinisation temperature (as measured according to Brabender), and at  $95^\circ\text{C}$ . A drop of the resulting paste was applied to a microscope slide and, on cooling, the smear was stained with iodine according to Kaczyńska *et al.* (1993) and observed with a Nikon FX light microscope.

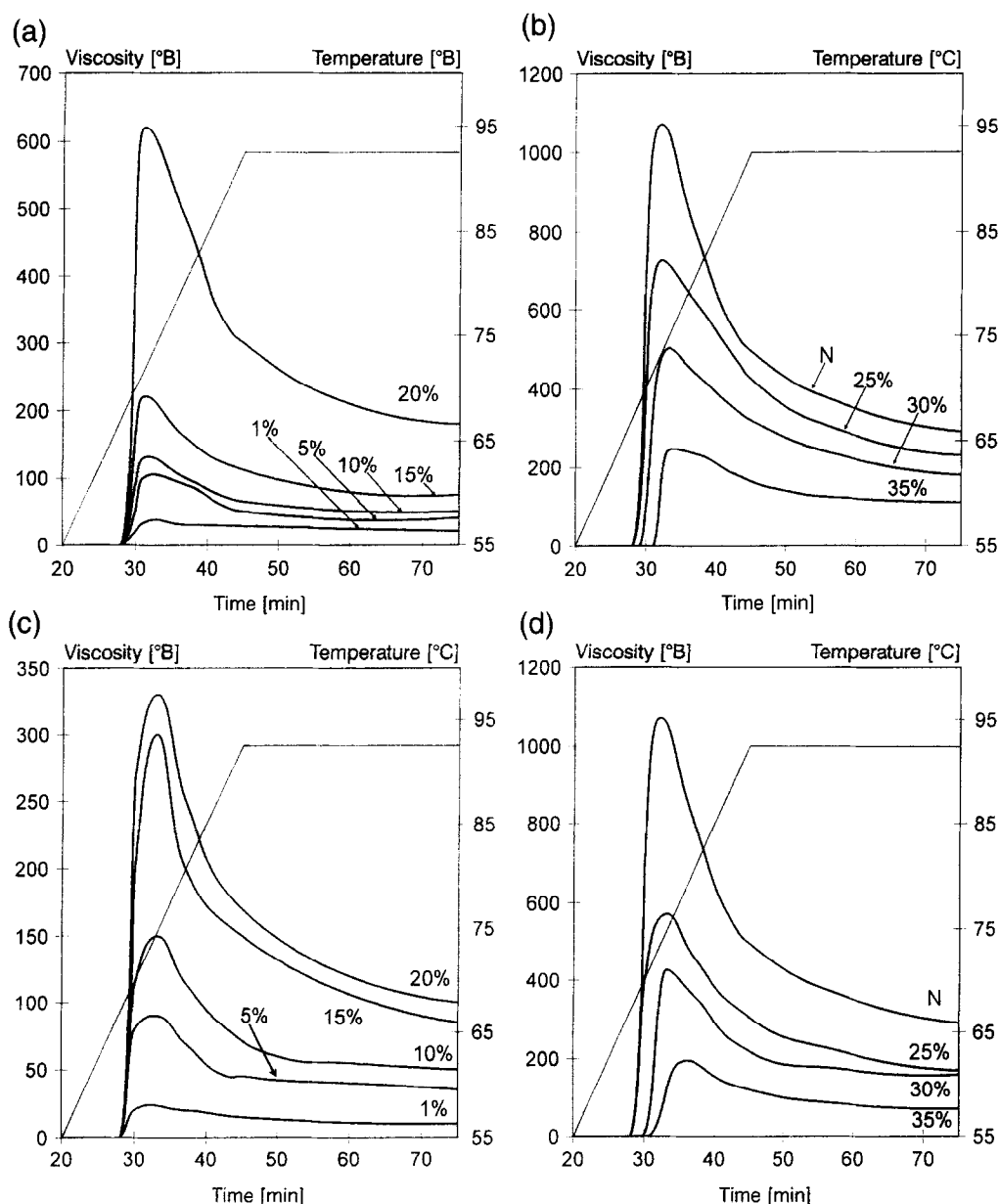
The starch samples to be examined by scanning

electron microscopy (SEM) were prepared according to Fornal (1985) and observed with a Jeol JSM 5200 microscope.

## RESULTS AND DISCUSSION

### Time–temperature profiles

A strong correlation was found between the water content of the starch samples and the character of their time–temperature profiles (Fig. 1). With the samples of low moisture content (1–5%), a rapid rise in temperature was observed while for those with a higher



**Fig. 3.** Brabender viscosity curves for 8% solutions of microwaved tapioca starch of moisture contents 2–17% irradiated in open beakers (A), of moisture contents 21–35% irradiated in open beakers (B), of moisture contents 2–17% irradiated in sealed beakers (C) and of moisture contents 21–35% irradiated in sealed beakers (D). N = native starch.

moisture content (7–15%) the rise was much less pronounced. This observation contradicts the opinion that dry starch is electrically inert (Miller *et al.*, 1991) and can be explained by high specific heat of water.

In the case of samples of moisture content over 20% a plateau was observed. The plateau interval length was found to rise with the rise in moisture content. This observation points to some kind of isothermal transformation occurring in the starch samples. The plateau interval lengths were also higher with the samples irradiated in sealed beakers as compared to those processed in open beakers. These observations prove that water affects the course of isothermal transformation of starch considerably.

### Rheological properties

A strong correlation was found between the moisture contents of the irradiated starch samples and their Brabender curves (Figs 2 and 3). On irradiation the dry starch samples showed a marked drop in viscosity while their gelatinisation temperatures remained unchanged. In the case of samples higher in moisture, up to 20%, the drop in viscosity was less pronounced. The Brabender curves for the microwaved samples of moisture content about 20% were almost identical with those for native starch.

Quite different results were obtained with the samples of moisture content ranging from 20% to

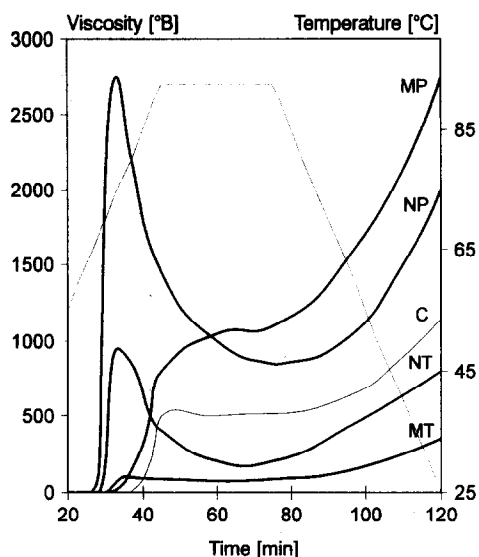


Fig. 4. Brabender viscosity curves for 8% solutions of starch samples of moisture content 35% irradiated in sealed beakers as compared to the native ones. NP= native potato starch; MP=microwaved potato starch; NT= native tapioca starch; MT=microwaved tapioca starch; NC= native corn starch.

35%. At these moisture contents a drop in viscosity and a rise in gelatinisation temperature were observed. The samples irradiated in sealed beakers showed a higher rise in gelatinisation temperature than those processed in open beakers. These observations point to a marked correlation between the plateau interval length in the time-temperature profiles and the gelatinisation temperature of the experimental starches. They also suggest that the isothermal transformations that occurred in microwave-irradiated samples affected their gelatinisation temperature and probably some other physico-chemical properties. From the Brabender curves (Fig. 2D and 3D) it can be concluded that the changes in the samples of potato starch were more pronounced. The gelatinisation temperature of the moist potato starch rose to 88°C (Fig. 2D), while that of tapioca starch only rose to 72°C (Fig. 3D). The shape of the Brabender curves for the moist potato starch differed greatly from that for the native starch. The pasting curves of tapioca starch were very alike and, apart from an insignificant drop in viscosity, no changes were observed.

The Brabender viscosity curves (Fig. 4) prove that native potato and tapioca starches change on irradiation from tuber to cereal type. Native potato and tapioca starches exhibited pasting characteristics typical of tuber starches, with a rapid increase in viscosity within a narrow temperature range and the occurrence of a viscosity peak. The samples of moisture content of 35% showed on irradiation pasting characteristics typical of cereal starches, with a progressive rise in viscosity over a wide temperature

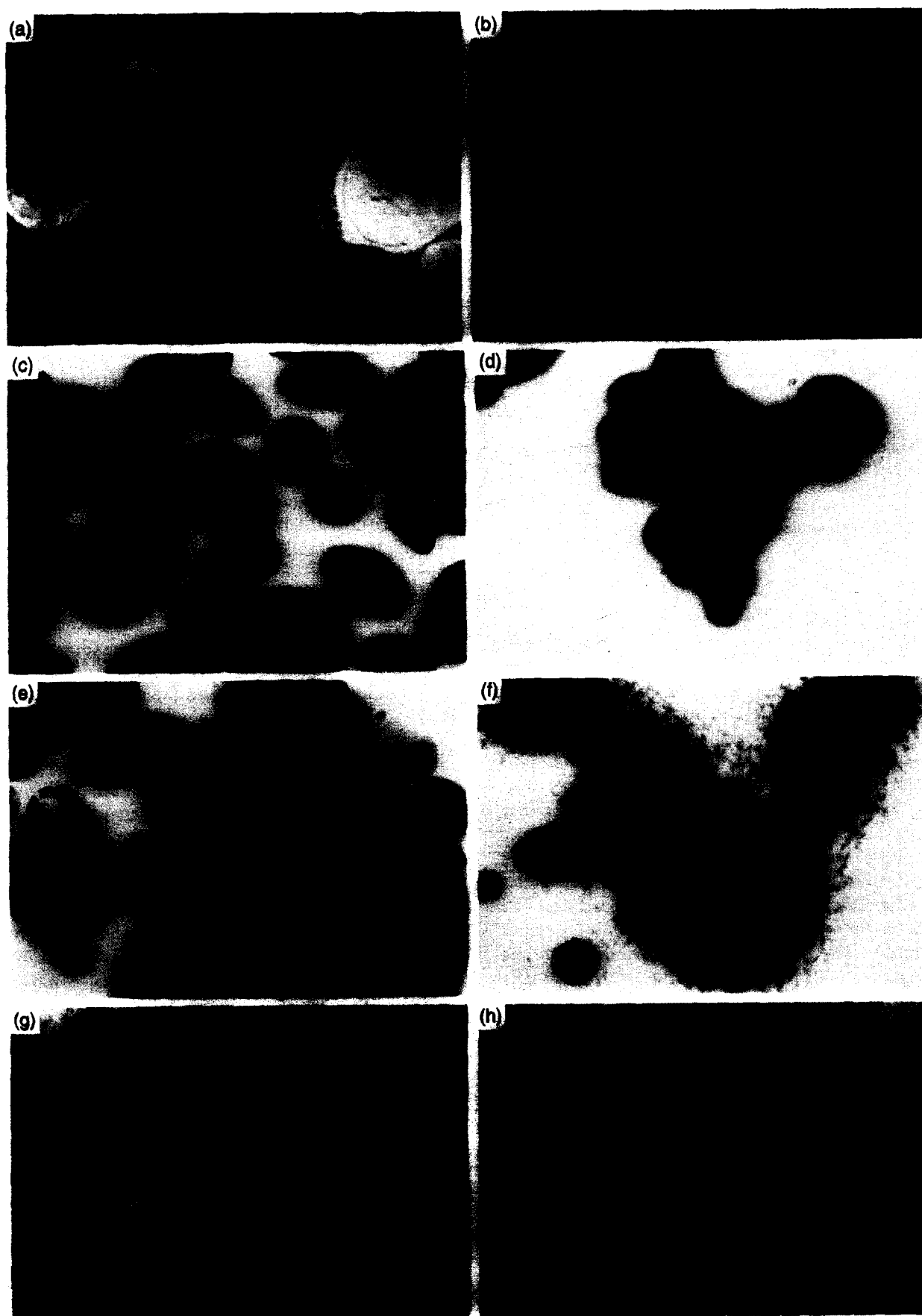
range and the lack of a viscosity peak. These changes suggest structural changes within starch granules.

### Microscopic observations

The above observations were confirmed by light microscopy. The samples of native potato starch heated at 68°C (Fig. 5A) gave an image characteristic of the early stages of gelatinisation, i.e. amylose escape from starch granules. When heated at 90°C (Fig. 5B) the granules of the native potato starch were almost fully soluble. With microwaved starch the image was different. At 68°C (Fig. 5C) there were no symptoms of gelatinisation and at 90°C (Fig. 5E) the image was typical of the initial gelatinisation phase, i.e. an amylose escape from starch granules. The microscopic images of the native potato starch heated at 68°C (Fig. 5A) or microwaved at 90°C (Fig. 5E) were almost the same. These changes were found to affect the solubility of starch. In other words, starch-starch bonds in the microwaved samples were stronger than in the unprocessed ones. As evidenced by microscopy, the extent of structural changes in tapioca starch was less than in potato starch. In the case of microwaved tapioca starch heated at 68°C (Fig. 5F), low but appreciable amounts of amylose were leached out of the granules. At 90°C (Fig. 5H) the samples exhibited marked solubility.

The microwaved potato and tapioca starches heated at 75°C (Fig. 5D and 5G) showed different behaviour patterns. Thus the samples of tapioca starch showed marked solubility, while those of potato starch exhibited no symptoms of gelatinisation. This proves that the potato starch undergoes extensive structural changes. The behaviour of potato starch subjected to microwave irradiation, in the presence of an adequate amount of water, resembled that of cereal starches which, when heated at 75°C, showed an insignificant amylose escape (pictures of this not shown). In other words, at 75°C cereal starches remained insoluble, while at 90°C (pictures not shown) they exhibited a marked solubility. The samples of microwaved potato starch heated at 90°C showed a marked drop in solubility, much more pronounced than corn or wheat starches.

Some changes occurring in the samples of microwaved starches were followed by scanning electron microscopy (Fig. 6). In the case of dry starches superficial cracks were detected, which could also have resulted from an electron bombardment. Of interest was the occurrence of centrally indented granules. Centrally indented granules were found to contribute essentially to the overall granule deformation occurring in starches of high moisture contents subjected to microwave processing. The above observations were close to those reported by Kawabata for heat-moisture treated starches



**Fig. 5.** Light microscopy microphotographs of the starch samples of moisture content 35% irradiated in a sealed beaker as compared to native starches. Native potato starch heated at 68°C (A) and 90°C (B), microwaved potato starch heated at 68°C (C), 75°C (D) and 90°C (E), microwaved tapioca heated at 68°C (F), 75°C (G) and 90°C (H).

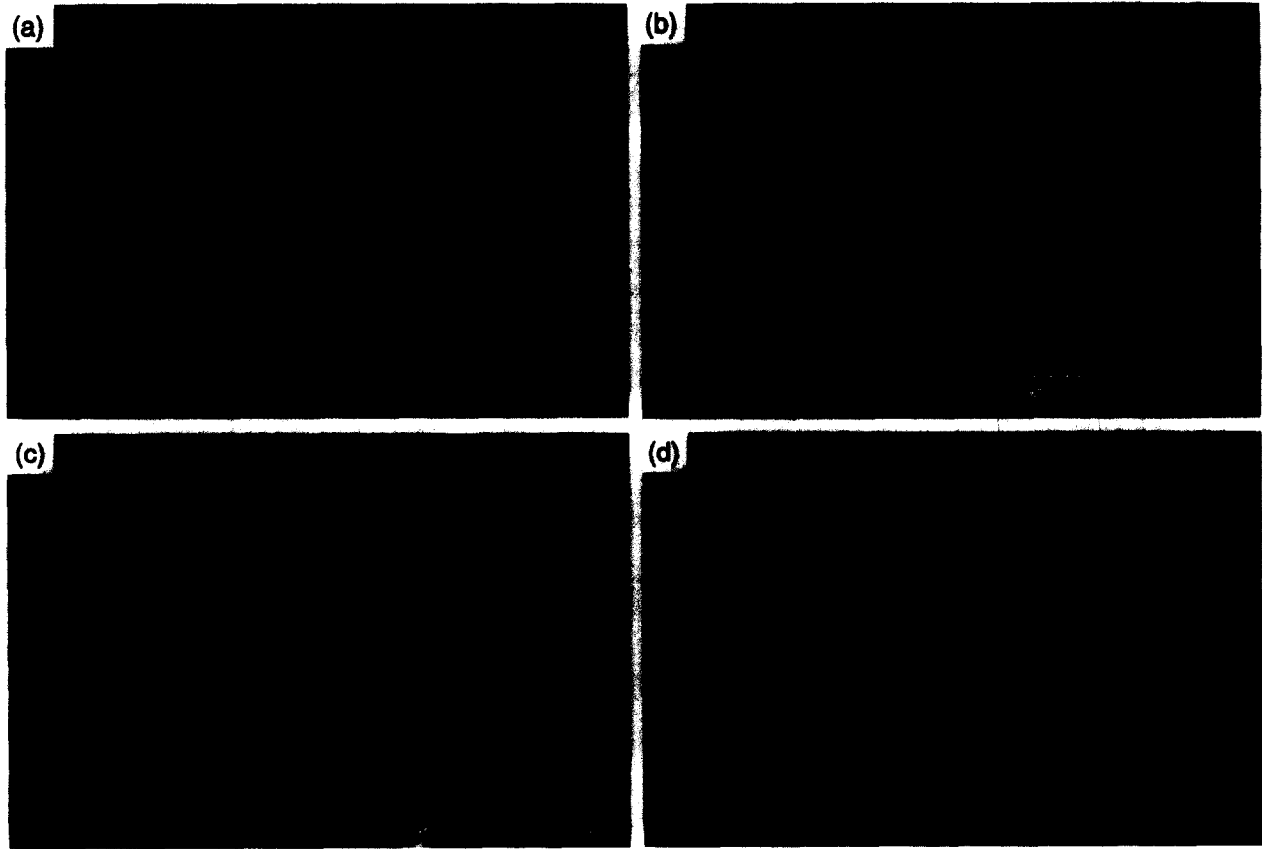


Fig. 6. SEM microphotographs of microwaved starches. Potato starch of moisture content 2% (A), potato starch of moisture content 35% (B), tapioca starch of moisture content 1% (C) and tapioca starch of moisture content 35% (D).

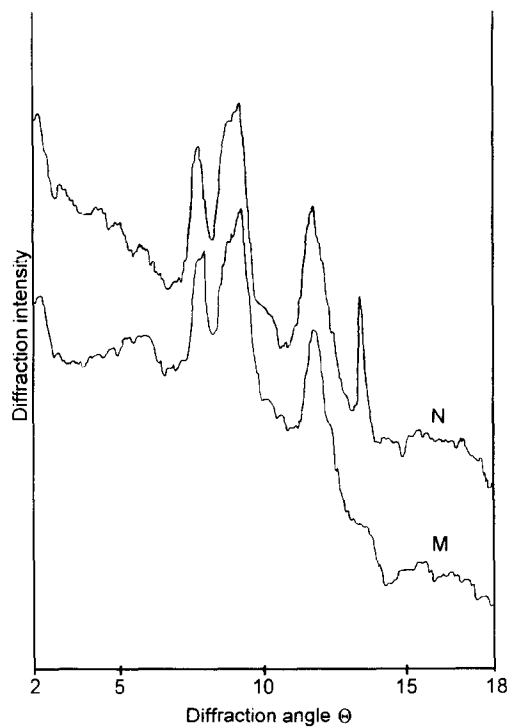


Fig. 7. X-ray diffraction patterns of potato starch of moisture content 35% irradiated in a sealed beaker as compared to native starch. N = native starch; M = microwaved starch.

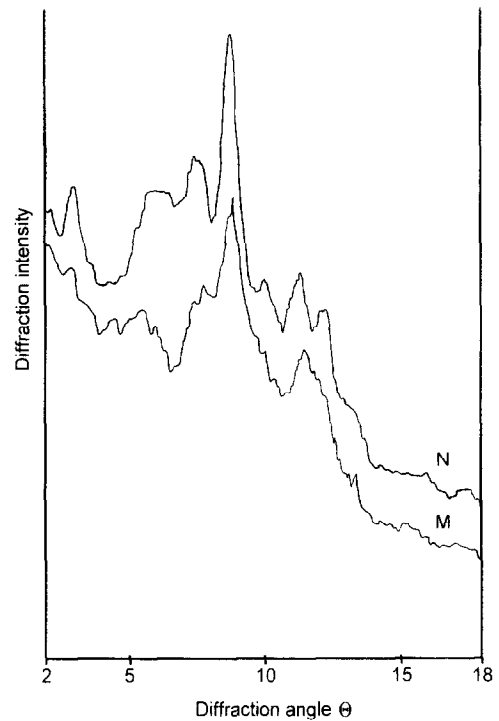


Fig. 8. X-ray diffraction patterns of tapioca starch of moisture content 35% irradiated in a sealed beaker as compared to native starch. N = native starch; M = microwaved starch.

(Kawabata *et al.*, 1994). Heat-moisture treatment of starches was evidenced to change their sorption properties, gelatinisation temperature, translucency and pasting characteristics. The most extensive changes were observed in tuber starches. In the case of potato starch, the type B X-ray diffraction pattern was found to change to type A (Sair, 1967; Lorenz & Kulp, 1981; Lorenz & Kulp, 1982a,b; Lorenz & Kulp, 1983; Donovan *et al.*, 1983). The classical heat moisture treatment of starch is effected by heating it in a pressure cooker at 100% relative humidity at 95–110°C for a period of up to 18 h. In our microwave irradiation experiments the temperature of isothermal processing ranged from 80 to 90°C and the time was up to 2.5 h; these are milder conditions than used in the classical procedure.

### X-ray diffractometry

Native potato starch gave a type B X-ray diffraction pattern typical of tuber starches (Fig. 7). On microwave irradiation it changed to a type A, typical of cereal starches. In the case of tapioca starch the interpretation is not as simple. Native tapioca starch gave an intermediate type of X-ray diffraction pattern (Fig. 8). There were marked differences between the diffraction patterns of microwaved and native starches, but the graph obtained is not typical of any kind of starch. None of the above differences were observed by Abraham in his experiments with cassava starch (Abraham, 1993). This divergence of results was due to the fact that the native starch used by Abraham gave a different X-ray diffraction pattern, one identical with that for our microwave-processed starch. The differentiation of X-ray diffraction patterns confirms our earlier conclusions, which we arrived at while analysing the course of commercial modification of tapioca starch differentiated physico-chemical properties and X-ray diffraction patterns, and demonstrates the necessity of thorough control of the production process and current adjustments of the technological parameters.

X-ray diffractometry analysis showed that under isothermal conditions the crystal structure of potato and tapioca starches changed. The changes that occurred in moist tuber starches on microwave irradiation resembled those brought about by heat moisture treatment.

### CONCLUSIONS

Under the conditions applied microwave radiation was found to affect the properties, structure and behaviour of potato and tapioca starches.

Microwave radiation was evidenced to affect the temperature and moisture contents of the experimental starches, with a strong correlation between the moisture content and the rate of temperature rise. The samples of low moisture content showed a rapid rise in temperature, while in those of higher moisture content the rise was less pronounced.

An isothermal transformation was discovered with the samples of moisture contents over 20%. This transformation was followed by:

- a rise in the gelatinisation temperature of the experimental starches and a change in their pasting properties from that typical of tuber to that typical of cereal starches
- a drop in the solubility of starch granules observed in light microscopy pictures as limited amylose leakage
- a change in the X-ray diffraction patterns; in the case of potato starch from type B to type A.

The above changes were similar to changes caused by the heat-moisture treatment process.

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