Journal of Thermal Analysis, Vol. 35 (1989) 1399–1404

DETERMINATION OF SUCROSE CONTENT OF CARIOGENIC DIETS BY THERMAL ANALYSIS

*Z. Gintner, *A. Végh* and *B. Ritlop*

CLINIC OF CONSERVATIVE DENTISTRY, SEMMELWEIS UNIVERSITY MEDICAL SCHOOL, BUDAPEST
*RESEARCH GROUP OF ORAL BIOLOGY, BUDAPEST, HUNGARY

(Received April, 1988)

The sucrose content of cariogenic diets was determined by thermal analysis. The thermal characteristics of various cariogenic diets were examined up to 700 °C. A linear correlation was found between the sucrose content of the sample and the mass loss in the range 180–240 °C.

The values determined by thermogravimetry were compared with those obtained by photometry as reference values.

Thermal analysis has been widely used in both medical and food chemical research [1–6]. It has frequently been applied to study the caramelization of mono- and oligosaccharides [7–10], and it has been successfully utilized to monitor the kinetics of the Maillard reaction, which occurs in food containing proteins and carbohydrates [10, 11].

These two reaction types are primarily responsible for the thermal properties of the cariogenic (i.e. caries-inducing) diets used in experimental cariology. These diets consist of three main components: sucrose, polysaccharides and proteins. An exact knowledge and continuous control of the sucrose content of cariogenic diets are indispensable with regard to their cariogenic effect. The quantitative determination of sucrose with no reducing property is rather troublesome by conventional techniques, especially in the presence of other carbohydrate components in the diet, due to the difficulties in separation and purification.

The aims of this study were to examine whether the sucrose content of cariogenic diets can be determined directly by thermal methods, and to establish the correlation between the results of thermal analysis and those obtained by photometry.

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Materials and methods

1 Materials

Cariogenic diet types SSP [12] and OBB [13], produced by the Laboratory Animal Breeding Institute of Gödöllő, Hungary, were tested (Table 1). For calibration, a model diet was prepared containing the components listed below:

sucrose (Reanal, Hungary);
wheat starch (Reanal, Hungary);
casein;
Al_2O_3 (Reanal, Hungary).

Table 1 Compositions of model and OBB and SSP diet groups

Compound	Model diet, %	OBB group, %	SPP group, %
Sucrose	0–70	0–70	0–50
Starch	70– 0	70– 0	50– 0
Protein	20	20	22
Others (vit., oils, minerals etc.)	10 (Al_2O_3)	10	28

Table 1 shows that the model diet contains the same three main components as the cariogenic diets. The OBB and model diets contained solely casein as protein source, whereas the protein content of the SSP diet group was composed of 18.2% casein and 4.5% soya protein.

The physiologically important components (e.g. vitamins, minerals, etc.) of the cariogenic diets were replaced by Al_2O_3 in the model diet.

2 Methods

2.1 Thermal analysis

Prior to the thermoanalytical measurements, each sample was rehomogenized for 10 minutes in a porcelain mortar to ensure uniform mixing of the components.

The equipment used was a Paulik–Paulik–Erdey Derivatograph, manufactured by the Hungarian Optical Works (MOM), with the technical conditions of the tests as follows: sample weight 50 mg; max. temperature (T) 700°; heating rate 2.5 deg/min; reference material Al_2O_3; atmosphere air; crucible Al_2O_3.

2.2 Photometric determination of sucrose

0.5 mg samples of thoroughly homogenized diet were suspended in 100 cm^3 of distilled water. After 10 minutes of thorough mixing, the samples were filtered through a glass filter. To 0.5 cm^3 of the filtrate, 2.5 cm^3 of freshly prepared antron reagent was added (50 mg of antron dissolved in 100 cm^3 of 72% sulphuric acid). The solution was incubated in a water-bath at 90–100° for 15 minutes. After cooling, the absorbance was read at 620 nm.

Results and conclusion

Figure 1 shows the thermal curves of a OBB diet containing 50% sucrose. The first peak in the DTG curve is caused by the evolution of mechanically bound

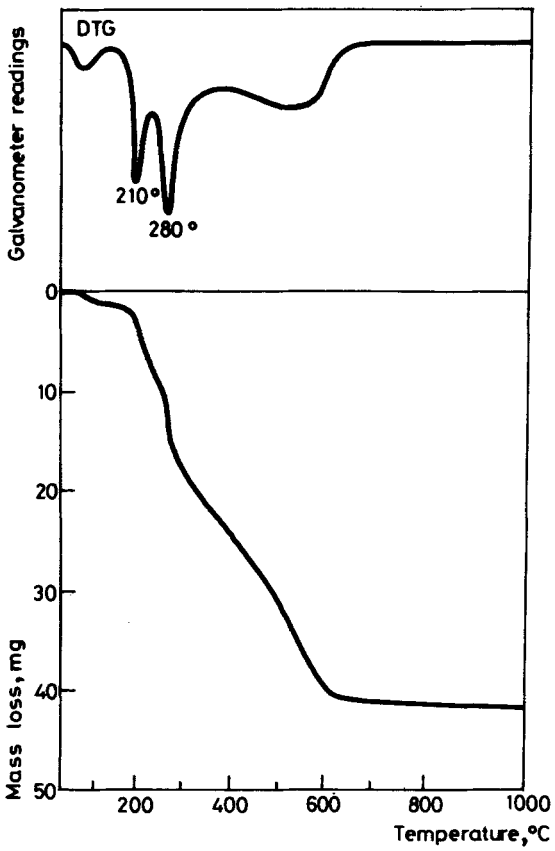

Fig. 1 Thermal curves of OBB diet containing 50% sucrose

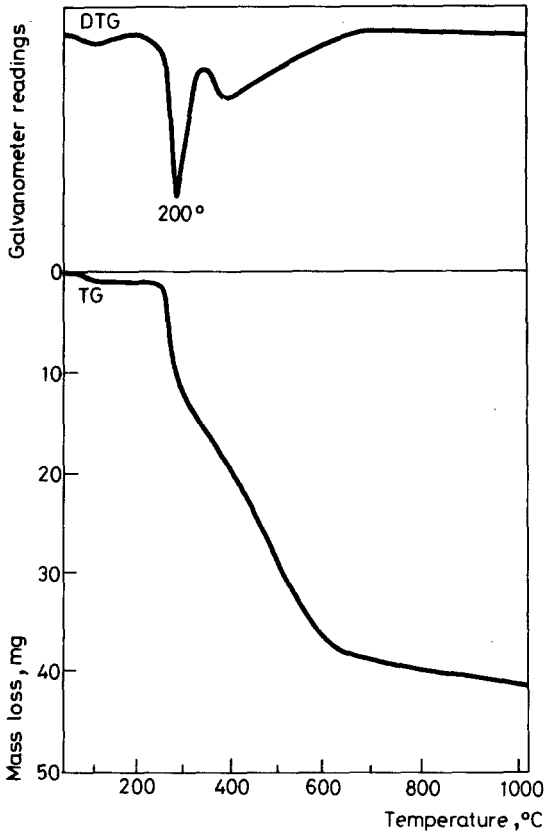

Fig. 2 Thermal curves of OBB diet containing no sucrose

water at 40–110°. Due to the biological properties of the diets, the previous dehydration of samples was not performed. The thermal reaction at 180–240° relates to the first caramelization phase of the sucrose. With no sucrose present in the sample, this peak is missing from the DTG curve, i.e. the thermal decomposition of the polysaccharides and the proteins is not significant at this temperature (Fig. 2).

On the basis of the linear correlation found between the sucrose content of the model diet (20, 30, 40, 60 or 70% sucrose) and the mass loss in the thermal reaction at 180–240°, a plot can be recorded (Fig. 3). The sucrose content of the diet samples was calculated via this calibration curve. Results of thermal and photometric tests are listed in Table 2.

Investigations of the thermal decomposition of sucrose indicated that glucose and fructose can be detected in the sample in the initial phase of the reaction. These

Table 2 Sucrose contents of cariogenic diet groups OBB and SSP measured by different methods

OBB group	Sucrose content, %			SSP group	Sucrose content, %		
	nominal	derivatograph	photometry		nominal	derivatograph	photometry
0/0	0	0	0	0/0	0	0	0
35/0	35	35	32	16/0	16	19	16
50/0	50	54	54	33/0	33	35	34
70/0	70	71	70	50/0	50	48	50

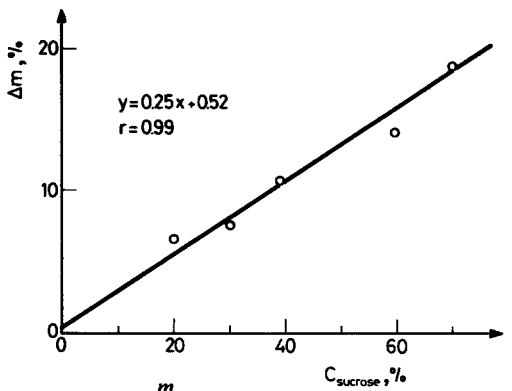

Fig. 3 Calibration curve. $m(\%) = \frac{m_x}{m_0 - m_{H_2O}} \times 100$; m_x: mass loss of sample at 180–240°; m_0: sample mass; m_{H_2O}: mass of water evaporated at 40–110°; $C_{sucrose}$, %: sucrose content of model diet

components undergo partial polymerization, accompanied by water release, to form oligo- and polysaccharides, together with oxo and other, as yet not identified coloured compounds [10, 11]. Glucose and fructose can also induce the Maillard reaction with proteins present in the sample at this temperature.

The decompositions of polysaccharides and proteins begin at 240° and continue up to about 650°. In this phase, the Maillard reaction is again of importance. This phase of decomposition can be divided into two stages, but we have not dealt with them in detail.

In our experience, the physiological components with very heterogeneous chemical structures (i.e. vitamins, minerals, etc.) do not appear to interfere significantly with the thermal reactions recorded. In contrast, the protein content of the model diet used for calibration and those of the samples to be determined must be identical (Maillard reaction) in order for exact results to be obtained. The reproducibilities of the two methods were compared. Nine parallel tests each were performed on different days with both methods, using samples containing 50%

sucrose. A CV value of 5.3% obtained with the thermoanalytical method, while photometry yielded 4.6%.

The results listed in Table 2 suggest that the chosen thermoanalytical method is suitable for direct and simple determination and also for routine tests of the sucrose content of cariogenic diets.

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Zusammenfassung — Mittels Thermoanalyse wurde der Saccharosegehalt von Karies verursachenden Diäten bestimmt. Die thermischen Eigenschaften verschiedener kariogener Diäten wurden bis 700 °C untersucht. Zwischen Saccharosegehalt der Probe und Massenverlust bei 180–240 °C konnte ein linearer Zusammenhang festgestellt werden. Die thermogravimetrisch erhaltenen Werte wurden mit den durch Fotometrie erhaltenen Werten als Referenzwerte vergliche.

Резюме — С помощью термического анализа было определено содержание сахарозы в кариогенных диетах. Термические характеристики различных кариогенных диет были исследованы до температуры 700°. Установлена линейная корреляция между содержанием сахарозы в образцах и потерей веса в температурном интервале 180–240°. Значения, определенные с помощью термогравиметрии, были сопоставлены со стандартными значениями, полученными фотометрическим методом.