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The potential role of isothermal calorimetry in studies of the stability of fresh-cut fruits

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ABSTRACT

Attention is drawn to the feasibility of using high sensitivity isothermal heat conduction calorimetry to study metabolic responses of differently processed and stored fresh-cut fruit. The heat production of endogenous (tissue metabolism during 12 h of analysis at 10 °C for kiwifruit and strawberry) and exogenous (microbial growth during 18 d of analysis at 10 °C for cantaloupe) biological processes was investigated.

Osmotic dehydration of fresh kiwifruit in sucrose solution (61.5 g/L) at different treatment times (30, 60 and 180 min), resulted in metabolic heat production decrease, confirming the progressive cell death induced by osmotic dehydration.

Analysis on strawberry slices under two atmospheric conditions (air and innovative modified atmosphere) seemed to confirm the inhibitory effect of N₂O on metabolic activity.

Cantaloupe samples immersed in three different syrups (SS: sucrose syrup (20 °Brix); SS₁: SS + 0.5 g/L ascorbic acid + 0.5 g/L citric acid; SS₂: SS₁ + 0.1 g/L potassium sorbate) showed a stability increase when additives with antimicrobial properties were included.

Our findings confirm that isothermal calorimetry provides a versatile and high sensitive tool for conducting fundamental metabolic studies on the effect of different processing operations on the quality and shelf life of fresh-cut fruit and vegetables.

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1. Introduction

The fundamental principle to consider for the stability of fresh-cut fruits is that they are metabolic active tissues and show physiological responses to preparation procedures as well as to the environment created in the package in which they are enclosed (Ahvenainen, 2000).

Isothermal calorimetry, the measurement of thermal power and heat at constant temperature, is a general measurement technique as nearly all processes (physical, chemical, biological, etc.) produce heat. A calorimeter measures the sum of all the heat produced in the sample, permitting the overall study of a biological system without going into detail (Wadsö & Gómez Galindo, 2009). Calorimetric measurements of the heat production rate have been used

to provide a direct indication of the gross metabolic activity of fruit and vegetables, as well as metabolic responses to stress provoked by processing such as wounding and pre-treatments with antioxidants (Gómez Galindo, Rocculi, Wadsö, & Sjöholm, 2005; Wadsö & Gómez Galindo, 2009). For microbial growth assessment, the measured heat is produced by the respiration of microbes, originating from both growth and maintenance. During exponential growth, the measured thermal power is proportional to the rate of increase in the biomass, but the thermal power can also—in later growth phases—come mainly from maintenance processes (Li & Wadsö, 2011; Wadsö & Gómez Galindo, 2009).

In this study, we have pioneered the use of isothermal calorimetry to monitor metabolic responses of different fresh-cut tissues subjected to different processing (osmotic treatment) and storage conditions (modified atmosphere - MA - and storage in syrup). The measurements allowed the heat production determination of endogenous (tissue metabolism) and exogenous (microbes growth) biological processes.

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2. Materials and methods

2.1. Raw material and sample preparation

2.1.1. Osmo-dehydrated kiwifruit

Medium size kiwifruits (*Actinidia deliciosa* var. Hayward) grown in the south of Italy were purchased in the local market and stored at 4 ± 1 °C until used. The fruits had a refractive index of 9.1 ± 1.0 °Brix. Hand peeled fruits were cut into 10 mm thick slices and immersed in a 61.5 g/L sucrose solution at 25 °C for 0 (control), 30, 60 and 180 min. The product:solution ratio was 1:4 (w/w). Four slices of 1 cm each from the central part of each kiwifruit (about 180 g) were placed in meshed baskets and immersed in the osmotic solution. The baskets were continuously stirred. The rotational speed was experimentally determined to assure negligible resistance to mass transfer. After osmotic treatment, the slices were taken from the solution, rinsed with distilled water and placed on blotting paper.

Eight kiwifruit cores of 0.5 cm in diameter were excised longitudinally from the pericarp tissue of each slice with a core-borer. The cores (about 2.5 g) were placed in a glass ampoule for isothermal calorimetry measurements, which was subsequently sealed with a teflon-coated rubber seal and an aluminium cap. Four replicates from four kiwifruit slices for each treatment condition were prepared.

2.1.2. Fresh-cut strawberries under modified atmosphere

Medium size strawberries (*Fragaria x ananassa* var. Candonga) grown in the south of Italy were purchased in the local market and stored at 4 ± 1 °C until used. The fruits had a refractive index of 9.1 ± 0.3 °Brix. Two fruits were cut longitudinally in eight pieces. To perform the isothermal calorimetry measurement, two strawberry pieces (of about 0.5 g each) were placed in each of the eight ampoules. Synthetic air (79% N₂ and 21% O₂) was flashed in four ampoules before they were sealed and a gas mixture of 79% N₂O and 21% O₂ was flashed in the other four ampoules before they were sealed. The sealed ampoules were immediately placed in the calorimeters.

2.1.3. Fresh-cut cantaloupe in syrup

Cantaloupe (*Cucumis melo* var. Cantalupensis) grown in the north of Italy with a refractive index of 8.3 ± 0.1 °Brix, was purchased in the local market and stored at 4 ± 1 °C until used. The fruits were cut in two halves. Forty eight cantaloupe cores 1.5 cm long and 0.5 cm in diameter were obtained using a core borer. Each core was transversally cut in four equal pieces. Twelve glass ampoules were prepared, placing 24 fruit pieces in each ampoule together with syrup in a 1:1 (w:w) product:solution ratio, simulating industrial conditions. The amount of sample used for cantaloupe experiments (about 9 g of fruit in 9 g of syrup) was chosen to simulate real packaging conditions in terms of product:syrup ratio and headspace volume.

Three different syrup formulations were prepared (4 ampoules for each syrup), with the following composition:

- SS: sucrose syrup (20 g/L);
- SS₁: SS + 0.5 g/L ascorbic acid + 0.5 g/L citric acid;
- SS₂: SS₁ + 0.1 g/L potassium sorbate.

All the chemicals used were of analytical grade (Sigma–Aldrich, St. Louis, MO, USA).

2.2. Isothermal calorimetry measurements

The rate of heat production was continuously measured in a TAM Air isothermal calorimeter (TA Instruments, New Castle,

USA), as described by Gómez Galindo, Wadsö, Vicente, and Dejmek (2008). This calorimeter has a sensitivity (precision) of ± 10 μ W. The instrument contains eight twin calorimeters. In each calorimeter, heat is allowed to flow between the reaction vessel containing the sample and a heat sink, the temperature of which is kept essentially constant. The heat transfer takes place through a heat flow sensor that is located between the vessel and the heat sink.

Twenty mL sealed ampoules containing the samples were placed in the calorimeter at 10 °C to simulate commercial storage temperature. Measurements were performed for 12 h (kiwifruit and strawberries) or 18 d (cantaloupe). Each calorimeter had its own reference that consisted of a sealed 20 mL glass ampoule containing a specific amount of distilled water. Obtained thermograms have been normalized for fruit sample weight.

After each calorimetric experiment, on kiwifruit and strawberries sample ampoules, gas measurements were performed (gas analyser mod. MFA III S/L, Witt- Gasetechnik, Witten, Germany) to verify the prevalence of aerobic conditions.

3. Results and discussion

Preliminary experiments (not shown) were performed to choose the right amount of sample for both kiwifruit and strawberry experiments in order to avoid the incidence of anaerobic conditions during the 12 h of analysis. This is a key issue as biological samples with aerobic respiration produce about 455 kJ per mol oxygen consumed (or carbon dioxide produced). Different types of anaerobic respiration produce much less heat; typically, much less than 200 kJ per mol carbon dioxide are produced under anoxic conditions (Wadsö & Gómez Galindo, 2009).

3.1. Metabolic heat of kiwifruit tissue after osmotic dehydration

Fig. 1 reports the thermal power, proportional to the metabolic heat, produced by kiwifruit pericarp tissue during the 12 h of analysis. After this period, in the ampoule headspace of fresh sample, O₂ and CO₂ values were respectively $19.7 \pm 0.3\%$ and $1.4 \pm 0.3\%$, confirming the maintenance of aerobic conditions until the end of the experiment.

The thermal power produced per gram of kiwifruit sample progressively decreased by increasing the time of the osmotic treatment. Osmotic dehydration, which is a partial dewatering

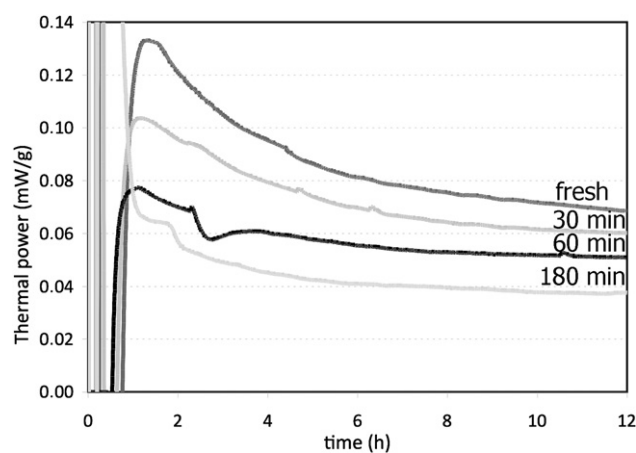


Fig. 1. Heat production profiles of pericarp tissue cylinders of control and osmo-dehydrated kiwifruit during 12 h of analysis at 10 °C. Osmotic dehydration was performed in sucrose solution (61.5 g/L) for different times (30, 60 and 180 min). Each thermogram is an average of four replicates. The initial signal disturbance is a consequence of sample insertion.

process caused by the immersion of cellular tissues in an hypertonic solution (Tylewicz et al., 2011), provokes osmotic stress to cells which may lead to cell death. The severity of the osmotic stress provokes the death of the first layer of cells allowing the diffusion of the osmotic medium inside the tissue, causing a progressive death of the cells that are in contact with the osmotic solution for enough amount of time (Mavroudis, Dejmek, & Sjöholm, 2004). The results presented here support this theory as the death of cells should provoke a decrease in the produced metabolic heat. In all curves, there is an initial decrease of thermal power from the initial stage suggesting that cell death in the sample cross section as well as mass transfer within damaged cells and the internalized sugar continues to take place hours after the sample was removed from the osmotic medium.

3.2. Metabolic heat of strawberry tissue under different atmospheric conditions

There is a great interest in the potential benefits of nitrous oxide (N_2O) in modified atmosphere packaging applications for fresh-cut fruits. This gas has shown a significant ripening inhibition effect for different fruit species caused by the repression of ethylene production (Rocculi, Romani, & Dalla Rosa, 2005).

The metabolic heat of strawberry samples during the 12 h of analysis is reported in Fig. 2. The sample analysed under MA showed a lower metabolic heat production after the fourth hour, confirming the inhibitory effect of N_2O as previously shown on fresh-cut apples, kiwifruit and pineapple (Rocculi, Cocci, Romani, Sacchetti, & Dalla Rosa, 2009; Rocculi, Romani, & Dalla Rosa, 2004, 2005). Strawberry slices stored in synthetic air had higher respiration as shown by the determination of O_2 and CO_2 levels on the ampoule headspace at the end of the experiment. The sample exposed to MA ($20.2 \pm 0.3\% O_2$ and $0.5 \pm 0.1\% CO_2$) showed more changes in the headspace composition than the sample stored in synthetic air ($19.1 \pm 0.2\% O_2$ and $1.2 \pm 0.3\% CO_2$). However, in the ampoule headspace, anaerobic conditions were never reached for all investigated samples.

3.3. Metabolic heat of microbial growth on cantaloupe under different syrup formulations

Fresh-cut cantaloupe is one of the most perishable fruits due to its neutral pH. With the aim of evaluating syrup formulations suitable for prolonging the product shelf-life and fresh quality characteristics, calorimetric analysis were performed for 18 d on

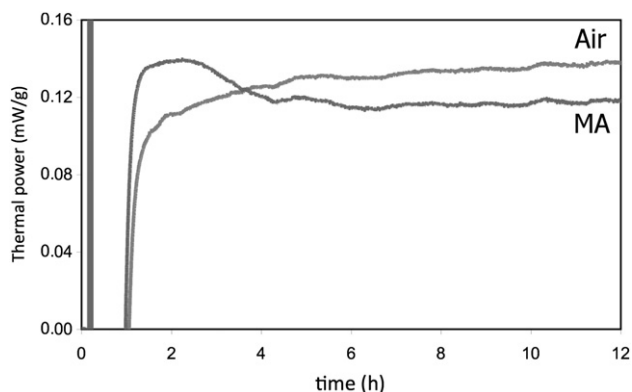


Fig. 2. Heat production profiles of strawberry tissue slices exposed to synthetic air (79% N_2 and 21% O_2) or modified atmosphere (79% N_2O and 21% O_2) during 12 h of analysis at 10 °C. Each thermogram is an average of four replicates. The initial signal disturbance is a consequence of sample insertion.

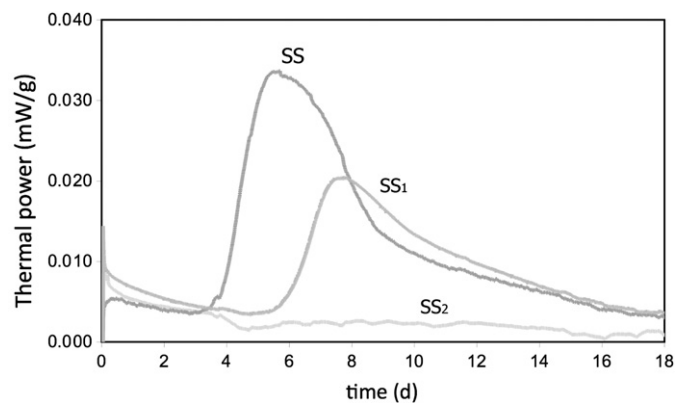


Fig. 3. Heat production profiles of cantaloupe tissue cylinders in different syrups (SS: sugar solution 20 g/L; SS₁: SS + 0.5 g/L ascorbic acid + 0.5 g/L citric acid; SS₂: SS₁ + 0.1 g/L potassium sorbate) during 18 d of analysis at 10 °C. Each thermogram is an average of four replicates.

cantaloupe sample cylinders immersed in three different syrups (Fig. 3).

Cantaloupe in sugar solution (SS) showed an intense peak of metabolic heat produced by microbial growth, starting after about 3 d of analysis. When ascorbic acid was included in the syrup's formulation (SS₁), the metabolic heat decreased and was delayed in about 2 days. In the sample where 0.1 g/L potassium sorbate was added (SS₂), heat production was absent. The effect of decreased pH and an antimicrobial additive on microbial contamination and the consequent increase in shelf life was, therefore, clearly shown. The same study can be done at any temperature wanted, changing any ingredient or additive in the syrup to evaluate the results on shelf life, illustrating the versatility and potential of the technique.

4. Conclusions

The main problem with isothermal calorimetry is related with its generality: it measures heat that comes from a large number of different sources or metabolic processes. However, this paper evidenced that isothermal calorimetry is a versatile and high sensitive tool to conduct fundamental metabolic studies on the effect of different processing steps and storage conditions on the stability of fresh-cut fruit tissue, by investigating both endogenous and exogenous biological phenomena. The progressive loss of tissue viability caused by processing such as dehydration opens interesting possibilities for processing optimization. Our results show metabolic consequences of the fruits exposure to N_2O , providing new insights for potential applications such as packaging. Metabolic heat evaluation of fresh-cut fruit in syrup showed that calorimetric measurements can be used to assess the product shelf-life according to the syrup composition which should be optimized for different fruits.

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