Potential of infrared heating as a method for decontaminating food powder

Process development and impact on product quality

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Zusammenfassung

Diese Dissertation erforscht die Möglichkeiten der Entkeimung von pulvrigen Lebensmitteln mittels Infrarotwärmestrahlung (IR). Paprikapulver wurde als Modelmaterial gewählt, angefeuchtet und beimpft mit Sporen eines psychrotrophen *Bacillus cereus* Stammes. Der Einfluss von IR-wärmestrom und -wellenlänge, und der Wasseraktivität (a_w) von Paprika wurde hinsichtlich Farb- und a_w-veränderungen, und der mikrobielle Entkeimung untersucht.

Paprikapulver wurde in drei verschiedene Pulverbehältern im Labormaßstab behandelt: ein (1) offener Pulverbehälter; und zwei geschlossene Pulverbehälter unter Benutzung von transparentem kurzwelligem (2) IR-glas und (3) IR-plastikmaterial. Die IR Erhitzung wurde nach dem HTST-prinzip durchgeführt und bestand aus einer Aufwärmphase und einer nachgeschalteten Halteperiode bei gewünschter Produkttemperatur.

Die Pulveroberfläche war der kritische Teil während der Erhitzung. Dort zeigten sich ungewünschte Bräunungserscheinungen und Produkttrocknung durch Temperaturen über 100°C. Dies wurde vor allem beobachtet beim offenen Pulverbehälter, wo Wasserdampf ungehindert entweichen konnte und durch Verwendung eines konstanten Wärmestromes ein unkontrollierbarer Temperaturanstieg stattfand. Durch Produkttemperaturkontrolle, mittels variablem Wärmestrom, und Verwendung eines geschlossenen Pulverbehälters wurden diese negativen Erscheinungen begrenzt. Oberflächliche Farb- und a_w-veränderungen hatten keine Auswirkungen auf den Gesamteindruck des Pulvers. Farbveränderungen im Produkt waren ein Ergebnis des Befeuchtens und von Produkttemperaturen während des Erhitzens über 60°C.

Je höher der Wärmestrom, desto höher auch die Oberflächentemperatur in Paprika. Bei gleichem Wärmestrom und $a_w < 0.8$, eine höhere Oberflächentemperatur wurde beobachtet für mittelwelliges-IR, während kurzwelliges-IR eine größere Eindringtiefe hatte. Diese unterschiedlichen Eigenschaften verschwanden aber bei $a_w > 0.8$.

Mikrobielle Abtötung hing von dem anfänglichen a_w und seinen Veränderungen während des Erhitzens, der Produkttemperatur und der IR Haltezeit ab. Produkttemperaturen von 95-100°C zeigten höhere Abtötungsraten als 90°C, wobei eine Erniedrigung des pHs von 4.5 auf 4.0 keinen bedeutenden Effekt hatte. Abtötungsraten von mehr als 4 $\log_{10}$ *B. cereus*/g wurden für den offenen (a_w 0.96), und geschlossenen IR glas (a_w 0.88) und IR-plastik (a_w 0.76) Pulverbehälter nach 6 min erreicht. Die gleiche Abtötung wurde unter Verwendung des IR-plastik Pulverbehälters (a_w 0.84-0-80) allerdings auch schon nach 2-3 min erzielt.

IR Entkeimung ist empfohlen für portioniertem Pulver eingeschweißt im IR-plastikbeutel, da hier kein nennenswerter Wasserverlust beim Erhitzen auftrat, das flexible Plastikmaterial einfach zu handhaben ist und eine mikrobielle Verunreinigung nach der Entkeimung ausgeschlossen werden kann. Der geschlossene IR-glas Pulverbehälter ist empfohlen, wenn das Pulver nach der Entkeimung noch getrocknet werden soll, da sich das Glas ganz einfach entfernen lässt.

Entkeimtes Pulver (a_w 0.76 und 0.84) wurde gelagert entweder für Pulver eingeschweißt im Plastikbeutel, oder nach Zugabe zu rohem Fleisch und Crème fraîche (Lagerung bei 7°C). Paprikapulver, gelagert bis zu 4 Monaten, zeigte mikrobiell stabile Werte von 2-3 $\log_{10}$ *B. cereus*/g, bei ebenso konstanten Messwerten von Farbe und a_w. Paprikapulver vermischt mit Fleisch zeigte einen Anstieg von 2 auf 5 $\log_{10}$ *B. cereus*/g, nach 12-20 Tagen, wobei die Werte unter dem Vergleichswert der unbehandelten Probe von 6 $\log_{10}$ *B. cereus*/g blieben. Paprika gewürzte Crème fraiche zeigte konstante Werte von 3 $\log_{10}$ *B. cereus*/g sogar nach 60 Tagen.

Entkeimung, Lagerung und Zugabe zu Lebensmittel wurde erfolgreich getestet für Paprika. Eine industrielle Umsetzung erfordert die genaue Auswahl von IR Wärmestrom und Produkt a_w um eine signifikante Abtötungsrate bei Beibehaltung der Produktqualität zu erzielen.

Schlagwörter: Infrarotwärme, Sporen, Entkeimung, Wasseraktivität, Farbe, Paprikapulver

Abstract

Dried powders, such as spices, may contain high microbial counts, particularly of bacterial spores, which are known for their high heat resistance and good survival ability. Although spores do not germinate in the powders themselves, adding the powders to high-moisture foods provides a suitable environment for microbial growth of spores. This thesis explored the potential of infrared (IR) heating for decontaminating food powders. Paprika powder was used as a model material, wetted to different water activities (a_w) and spiked with 7 $\log_{10}$ spores/g of a psychrotropic *Bacillus cereus*, SIK 340. IR heat flux and wavelength, and the a_w of paprika, were assessed for their effects on product qualities and microbial inactivation.

IR heating of paprika powder was studied using a pilot plant near- and medium-IR tunnel oven. The paprika powder was placed in three different lab-scale heating units: (1) an open heating unit, and two closed heating units using near-IR-transparent (2) glass and (3) plastic. The IR heating of paprika powder consisted of a warm-up period and a holding period at the desired product temperature. Heating was based on HTST treatment, i.e. rapid heating to a high temperature, which preserves product quality due to a reduced holding time.

During IR heating, the surface was the most critical part of the powder mass, displaying undesired browning and drying at temperatures over 100°C. This was predominantly seen with the open system, due to water evaporation and uncontrolled temperature increase when applying constant IR heat fluxes. Such product degradation was limited when the closed units were used, as the closure prevented water evaporation and the temperature was controlled using variable IR heat fluxes. In general, colour changes were a result of initial powder wetting and product temperature over 60°C during heating. However, *overall* colour and a_w were not significantly affected by the surface colour or a_w. The higher the IR heat flux, the higher the surface temperature in the paprika powder. At the same heat flux and at $a_w < 0.8$, higher surface temperatures were observed with medium-IR, while near-IR produced a greater heat penetration depth; however, those differences disappeared at $a_w \geq 0.8$.

Microbial reduction was dependent on initial a_w and its change during heating, product temperature, and IR holding time. Powder heated to a product temperature of 95–100°C displayed a higher microbial inactivation than did powder heated to 90°C, while the impact of lowering pH from 4.5 to 4.0 was negligible. A microbial reduction of more than 4 $\log_{10}$ *B. cereus*/g was achieved after 6 min at a_w 0.96, 0.88, and 0.76, for the open, closed-glass, and -pouch heating units, respectively. However, in the plastic pouch, the same reduction was achieved after 2–3 min at a_w 0.84–0.80. The IR decontamination of portioned paprika placed in sealed plastic pouches is recommended due to the negligible water evaporation, easy handling of flexible plastic material, and avoidance of powder recontamination subsequent to IR treatment. The use of an IR-glass heating unit is recommended when powder is dried to a_w 0.50 after IR decontamination due to the easy removal of the top lid.

Decontaminated paprika powder was either (a) stored in a sealed plastic pouch at a_w 0.76 or 0.84 or (b) mixed with meat or crème fraîche, and then stored at 7°C. Stored paprika powder displayed constant microbial values of 2–3 $\log_{10}$ *B. cereus*/g for the duration of the 4-month test period, while retaining a_w and an acceptable red colour. Paprika-spiced meat displayed maximal 5 $\log_{10}$ *B. cereus*/g after 20 d, remaining under the contamination level of 6 $\log_{10}$ *B. cereus*/g found in the untreated paprika powder. Paprika-spiked crème fraîche displayed constant microbial values of 3 $\log_{10}$ *B. cereus*/g for the duration of the 60 d test period.

The IR decontamination of paprika powder, and the subsequent storage and addition of the powder to high-water foods were successfully tested. Any industrial implementation would require the selection of an appropriate IR heat flux and product a_w to obtain significant microbial reduction while maintaining high product quality.

Keywords: Infrared heating, spores, decontamination, water activity, colour, paprika powder

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1 Introduction and objective of work

The need to increase food safety while maintaining high quality during the storage of food products have increased interest in processing methods that efficiently eliminate hazardous and unwanted micro-organisms without negatively affecting overall quality. The use of spice mixes, aroma components, and functional ingredients have increased in the food industry, especially in ready-to-eat meals and in highly spiced cuisines. Herbs and spices frequently harbour high amounts of micro-organisms, up to 6–8 $\log_{10}$ colony-forming units (CFU)/g, mostly due to poor sanitary conditions during growth, harvest, and/or drying. That level of contamination is often unsatisfactory for industrial purchasers or suppliers. The need for higher-quality spice mixes is especially important when the mixes are to be added to high-water food products, such as meat and dairy products, which will not be heat treated after the addition of spices. In the case of functional ingredients, which are often heat sensitive, better product quality will be obtained if mild heat treatment is used.

To meet customer requirements, such as microbial contamination below 4 $\log_{10}$ CFU/g and an absence of pathogens such as *Salmonella*, the spice processing industry is forced to conduct sanitation treatments. Most preservation techniques have been designed for high-water-content foods. Spices and herbs, however, represent a large group of powders. Commonly used treatments for these dried products are currently electromagnetic radiation, chemical agents, or steam, as the formerly standard ethylene fumigation has been restricted in parts of the world, such as the EU. Gamma radiation is another treatment method, but, apart from some exceptions, is also restricted in application. Heating is an effective method for decontaminating powders. However, the low a_w of powders limits heat transfer inside the powder mass. In the case of heat-sensitive spices, heating can impair the quality, by causing the loss of essential oils or changes in taste and appearance.

Infrared radiation (IR) is part of the electromagnetic spectrum and has advantages over conventional heating, as it heats the product directly, without heating the air around the powder, in a fast and effective thermal process. IR heating is used industrially in food processing for baking (roasting), drying, thawing, frying, and surface pasteurization. There is a lack of knowledge regarding the heating of powdered food by IR, and of the effects of IR heat flux and wavelength and product a_w on microbial inactivation and product quality.

Accordingly, this thesis investigated IR heating as a new technique for decontaminating food powders. Sweet paprika powder spiked with spores of *Bacillus cereus* was selected as the model material. Systematic studies were performed to identify the optimal a_w and temperature for the decontamination process while minimizing product quality changes during IR heating. The specific goals of this investigation were:

- To determine the temperature development and distribution in paprika powder during heating at different IR wavelengths and heat fluxes
- To evaluate the effect of IR heating on paprika powder with different a_w and pH levels and its effect on microbial reduction
- To assess the product changes in terms of a_w, colour, and water loss due to IR heating
- Based on the knowledge gained, to design an IR process for decontaminating paprika powder
- To assess the quality of the decontaminated powder during storage and after addition to two high-water foods, in this study, meat and crème fraîche

2 Literature review

2.1 Food powders

Many food ingredients are supplied in powdered form, such as herbs and spices, grain and potato flours, instant soups, salt, sugar, cacao, and vitamins. The major reason for supplying products in powdered form is simply to prolong the shelf life of the ingredients by reducing the water content; otherwise, in their natural state, the ingredients would become degraded in quality. The main function of the powdered form is to maintain the functional stability of the ingredient until it is used, which is usually in some wet formulation (Fitzpatrick & Ahrné, 2005). Besides, dry powders are lighter to transport, take less storage space, and offer opportunities to develop new and unique products.

Food powders can be produced by mechanical operations, such as grinding, or thermal treatments, such as spray or drum drying. Granular or powdered foods are two-phase systems, comprising a solid and a gas. At higher water contents, when liquid water may be present, three-phase systems (i.e. powder–water–air) can be found. Powders are mixtures of particles with characteristic size, shape, density, porosity, specific surface area, and flow properties. It should be remembered that the composition and properties of many food powders may be variable to different degrees and may also change with time.

Powders can be classified differently depending on the purpose of the classification, for example, for convenience or according to any particular relevant characterization or application (Peleg, 1983), as follows:

(1) by usage, e.g. flours, instant soups, spices, vitamin additives
(2) by major chemical component, e.g. starch, crystalline sugar, fatty, protein
(3) by production process, e.g. ground, spray dried, agglomerated, mixed, freeze dried
(4) by particle size, e.g. fine or coarse
(5) by moisture sorption pattern, e.g. extremely or moderately hygroscopic
(6) by flowability, e.g. free flowing, moderately cohesive, very cohesive

Powders are usually characterized at two levels: the individual particles and the powder in bulk. Although it is self-evident that a powder's bulk properties are primarily influenced by the particle properties, the relationship between the two is by no means simple and involves external factors, such as the system geometry and the mechanical and thermal histories of the powder. It is therefore practically impossible, in most cases, to predict a powder's bulk properties directly from those of the particles (Peleg, 1983).

The quality of a dry powdered product is determined by various product characteristics, such as colour, texture, porosity, density, and the rehydration capacity of the product (Krokida & Maroulis, 1997), and their reference standards depend on the type of product and its application. Other quality issues such as nutritive value and microbiological status are also important, although not related to consumer perceptions. The conditions under which the food powder is processed also often degrade product quality.

2.1.1 Particle size

Many properties of particulate systems depend on the *particle size*, which is the integral form of the solids of which the particles are composed. The mean particle size of a food powder is in a range of several orders of magnitude, that is, between single microns (e.g. individual starch granulates) to several hundreds or even thousands of microns (e.g. instant coffee). The classification of powders as a function of the particle size is shown in Fig. 2.1.

A ground solid can be characterized by the *particle size distribution* of its constituent particles. The best known techniques for the particle size analysis of food powders are dry sieving, microscopy and image analysis, liquid sedimentation and enumeration using a Coulter counter, and laser diffraction or near-IR spectroscopy.

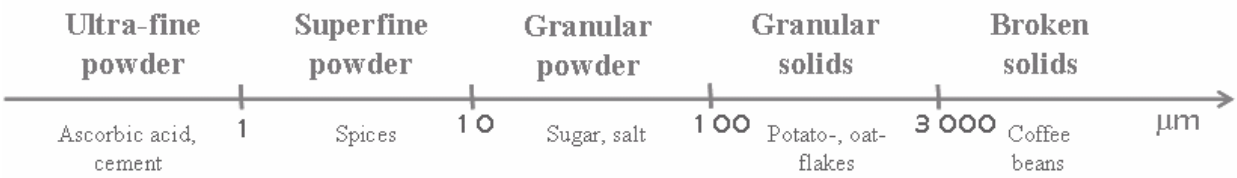

Figure 2.1: Classification of powder as a function of the particle size (Brown & Richards, 1970; Nedderman, 1992).

2.1.2 Powder density and porosity

The structure of a powder is complex, and can be characterized by various parameters, such as density (i.e. bulk, solid, or particle density), total (bulk) porosity, pore size, and specific volume.

The *bulk density* is the most emphasized quality factor of food powders or porous products, and is expressed as:

$$Bulk\ density = \frac{Weight\ of\ \ powder}{Volume\ of\ \ powder\ bed}$$

The powder weight includes the weight of all solid particles and any water contained in the powder bulk, including in the space between particles and in the void fraction inside the pores of individual particles. Bulk density strongly depends on the treatment or handling of the powder. For this reason, the bulk density of powders is usually reported as "freely settled" or "loose" bulk density (i.e. after the powder has only been poured), "tapped" density (i.e. after vibration), or compact density (i.e. after compression).

Most food particles have a similar *solid density*, i.e. the density of the solid material from which they are made disregarding any internal pores, of approximately 1.4–1.5 g/cm^3 depending on the moisture content (Peleg, 1983). This is mainly due to the similar density of the main ingredients, except for salt-based and fat-rich powders (Table 2.1).

Table 2.1: Main ingredients of food powders and their solid densities

Ingredient	Solid density (g/cm^3)
Glucose	1.56
Starch	1.50
Cellulose	1.27–1.61
Protein (globular)	~1.4
Fat	0.9–0.95
Salt	2.16
Water	1.00

Another type of density is the *particle density*, which is defined as:

$$Particle\ density = \frac{Particle\ actual\ mass}{Particle\ actual\ volume}$$

This parameter does account for the existence of internal pores and therefore can be considered as a measure of the true density of the particles without considering the shape of any internal pores or their position in the particle structure (Peleg, 1983).

Considering a bed of powder, spaces exist between the individual powder particles; in addition, most particles are themselves porous, i.e. having internal voids and pores. Thus *total porosity* is the fraction of volume not occupied by a particle or solid material and can be expressed as the ratio of void volume to total volume (bulk density) of the powder:

$$Total\ porosity = 1 - \frac{Bulk\ density}{Solid\ density}$$

The total porosity can be subdivided into intra- and interparticle porosity, depending on the intraparticle voids and interparticle spaces, respectively.

Food powders vary considerably in porosity, by 40–80%, depending on (1) water content and (2) geometric considerations. A packed bed is often defined as an arrangement of particles in which the porosity or cavity space is low at approximately 40%. Most food powders are known to be cohesive, which means that their interparticle attractive forces are significantly high relative to the individual particle's weight (Peleg, 1983).

(1) In general, moisture sorption is associated with increased cohesiveness, mainly due to the formation of interparticle liquid bridges, i.e. agglomeration. Therefore, especially in hygroscopic food powders, higher moisture content ought to result in a lower bulk density (i.e. higher porosity) or even in caking. At higher water contents, the amount of small particles (fines) is negligible.

(2) The variation in the shape of food powder particles is enormous, and the shapes range from extreme irregularity (in ground material) to approximate sphericity (starch) or well-defined crystalline shapes (granulated sugar, salt). Fines exclude air from the powder bed, as they fill the spaces between larger particles and increase the contact area between particles, resulting in higher density and lower porosity.

2.1.3 Water relationships and sorption isotherms

A food product is divided in two main parts: the *water content* and the solid content. The solid content involves all kinds of components except water, such as carbonates, proteins, fat, and minerals. The water content of powders is limited, commonly in the range from 5 to 20%. Water in a solid may be either free (unbound) or bound. Bound water is linked to other chemical substances within the material and it is unable to move; free water exists in cellular structures or capillaries and is able to diffuse throughout the material.

Over the past few decades, *water activity* (a_w) has become a very useful parameter in dealing with water relationships involved in food processing. Per definition, a_w is the moisture pressure above the food material divided by the moisture pressure above pure water (Marechal et al., 1999):

$$a_w = \frac{P}{P_0}$$

The a_w value represents the degree of free water within the material and its availability to participate in physical, chemical, and microbiological reactions. Only the free water has an impact on the product spoilage. Furthermore, achieving optimal a_w not only helps reduce undesirable microbiological growth, but also reduces enzyme activity and facilitates structural and textural stabilization (Fig. 2.2).

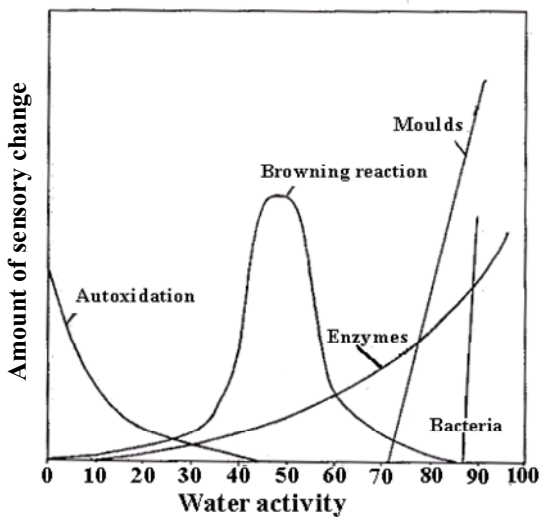

Figure 2.2: Characteristics of different causes of decay as related to the ambient equilibrium a_w of the food (at constant temperature and time) (Gerhardt, 1974).

The moisture or *sorption isotherm* is a useful thermodynamic tool for determining interactions between water and food substances, and provides information with which to assess food processing operations, such as drying, mixing, packaging, and storage. Sorption isotherms can also be used for selecting appropriate storage conditions and for designing packaging systems that optimize or maximize retention of aroma, colour, texture, nutrients, and biological stability (Debnath et al., 2002). The moisture level at which certain dry foods have good storage stability has been found to agree closely with moisture as a calculated monolayer of absorbed water (Mohammad et al., 1986).

Sorption isotherms consisting of a graphic representation of a_w versus moisture content at constant temperature are a common way of presenting the relationship between these two parameters (McLaughlin & Magee, 1998; Rahman & Labuza, 1999). As all foods have a complex composition and structure, sorption isotherms actually describe the integrated hygroscopic properties of the various constituents. Typical sorption isotherms of selected food powders are presented in Fig. 2.3. The shape of the curve depends strongly on the structural, physicochemical, and chemical properties of the food components. In general, the sorption isotherms of food products are sigmoid in shape, a result of several basic interacting water binding mechanisms (Aguilera & Stanley, 1999). The equilibrium moisture content of a product may differ depending on whether the product is being wetted (adsorption) or dried (desorption), which can be observed in almost all hygroscopic products (Mujumdar & Menon, 1995). Thus a lower vapour pressure is needed to achieve a given moisture content by desorption than by adsorption; this phenomenon is called hysteresis.

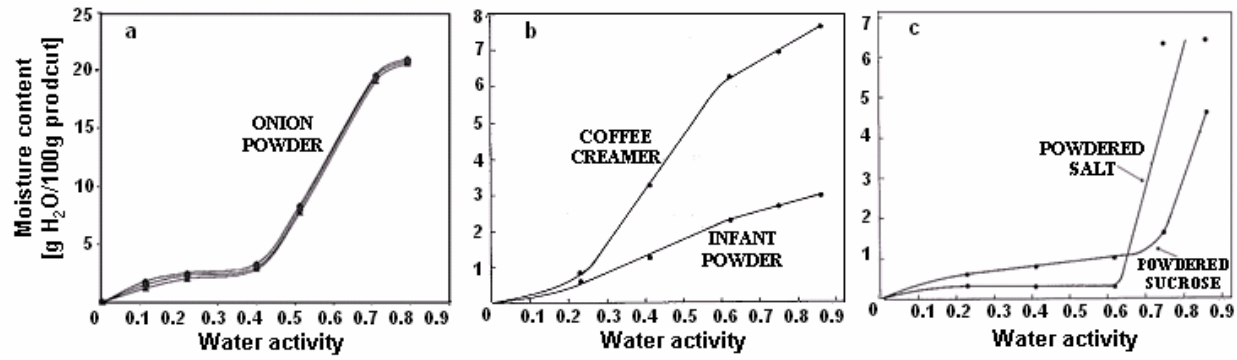

Figure 2.3: Sorption isotherms of a) onion powder (Debnath et al., 2002), b) coffee creamer and infant formula, and c) powdered salt and sucrose (Moreyra & Peleg, 1981).

2.1.4 Thermophysical properties

The thermal properties of foods need to be understood to be able to predict the heat transfer rates in foods during pasteurization, concentration, drying, heating, or cooling. The most commonly used thermal properties are (1) specific heat, (2) thermal conductivity, and (3) diffusivity. Regarding heat transfer by IR radiation, the absorptivity is of interest (Sweat, 1986). However, there is little reported research into the thermal conductivity of powdered foods (Muramatsu et al., 2005). Thermal properties of selected foods and powders are given in Table 2.2.

(1) *Specific heat* indicates how much heat is required to change the temperature of a material. Temperature and moisture content greatly influence the specific heat. Generally, the specific heat is expressed as a function of moisture content and/or temperature. The dynamic nature of differential scanning calorimetry (DSC) allows the determination of specific heat as a function of temperature (Singh & Goswami, 2000). Knowing the specific heat of each component of a mixture is usually sufficient to be able to predict the specific heat of a mixture. Specific heat is independent of mass density and can be written as:

$$c_p = \frac{Q}{m \cdot (T_2 - T_1)}$$

where c_p is the specific heat, Q the added heat, m the mass, and T_1 and T_2 the temperature before and after heating.

(2) The *thermal conductivity* (k) of a food material measures its ability to conduct heat. It is important not only for the process design, but also for the prediction and control of various changes occurring in food during thermal processing. Thermal conductivity depends mostly on composition of a powdered food material, but also on any factors that affect the heat flow paths through the powder, such as homogeneity, particle shape, particle size, and percentage and arrangement of void spaces. Granular or porous foods, such as powders, are solid–gas systems. Water has good thermal conductivity, but its amount is limited in powders. Air is present between particles but has a poor thermal conductivity. The thermal conductivity and optical properties of powders are affected by temperature, moisture content, and bulk density.

In granular and powdered foods, it is convenient to use the effective thermal conductivity (k_{eff}), an overall thermal transport property, assuming that heat is transferred by conduction through the particles and pores of the bulk material (Sweat, 1986; Drouzas & Saravacos, 1988). Thermal conductivity can be expressed as:

$$k = Q \cdot \frac{\ln\left(\frac{t_2 - t_0}{t_1 - t_0}\right)}{4\pi \cdot (T_2 - T_1)}$$

where k is the thermal conductivity of the sample (W/m°C), Q the energy generated by the probe heater (W/m^2), t_1 and t_2 the time (s) since the probe heater was energized, t_0 a time-correction factor(s), and T_1 and T_2 the temperature of probe thermocouple at t_1 and t_2, respectively. Determining the true thermal conductivity (k) of food powders is complex because of their heterogeneous composition and the random packing of their irregularly shaped particles; these characteristics affect the path of heat flow, chemical composition, temperature, and physical properties, such as bulk density, particle density, size, shape, arrangement, and contact area. Particle density was found to be strongly dependent on moisture content. For many powders, such as corn and potato starch, it was interesting to note that particle density increased with moisture content to a maximal value, after which it linearly decreased with further increase in moisture content (Sweat, 1986).

(3) The *thermal diffusivity* (α) of a food material is related to the thermal conductivity (k) by the following simple equation:

$$\alpha = \frac{k}{\rho \cdot c_P}$$

where ρ and c_P are the density and specific heat of the material, respectively. Thermal diffusivity is used in determining the heat transfer rates in solid food objects of any shape. Physically, it relates the ability of a material to conduct heat to its ability to store heat (Sweat, 1986).

Table 2.2: Thermal properties of selected food products

Food	Water content	Bulk density (kg/m^3)	Thermal conductivity (W/mK)	Specific heat of food (J/kg K)	Heat transfer coefficient (W/m^2 K)	Source
Water	100%		0.570	4168 (0°C) 4219 (100°C)		
Ice	100%		2.25	2050		
Freeze-dried foods	0%		0.01–0.04			(Fellow, 1988)
Fresh potato	80%		0.554	3517.6	30	(Ranjan, 2002)
Corn meal	7.5%		0.141	1691	1.32	(Jun, 2004)
Coffee powder	10%			1800 (50°C) 2050 (100°C)		(Singh, 1997)
Cumin seeds	9% 21%			1800 (50°C) 3090 (50°C)		(Singh, 2000)
Whole milk powder	1.4% 3.8%	650	0.076–0.081 (20–50°C) 0.082–0.092 (20–50°C)			(Muramatsu, 2005)
Rice flour	9% 18% 9% 18%	650 800	0.074–0.090 (20–50°C) 0.092–0.120 (20–50°C) 0.103–0.122 (20–50°C) 0.119–0.159 (20–50°C)			
Native corn starch	0% 12% 27%	1000	0.12–0.16 (30–150°C) 0.18–0.30 (30–150°C) 0.20–0.35 (30–150°C)			(Shah, 2000)
Corn	10% 40%		0.150 0.330			(Kustermann, 1981)

2.1.5 Heat transfer in food powders

Heat transfer in powders is an important process operation. Important factors affecting it in powders are bulk specific heat, bulk thermal conductivity, true and bulk density, and moisture content (Singh et al., 1997).

Heat transfer in powders by IR depends on the porosity and water content and is a combination of radiation, conduction, and – in negligible parts – convection as well.

2.1.5.1 Heat radiation

Radiative heat transfer occurs between two surfaces by the emission and absorption of electromagnetic waves. No physical medium is needed for the propagation of electromagnetic waves, which move at the speed of light. Gases are often transparent, while liquids and solids are strong absorbers of thermal radiation.

A body that absorbs all incident radiation, regardless of its wavelength or direction, is termed a black body. The emitted radiation is a function of wavelength and temperature but is independent of direction. Selected black bodies and their radiant characterizations are presented in Fig. 2.4.

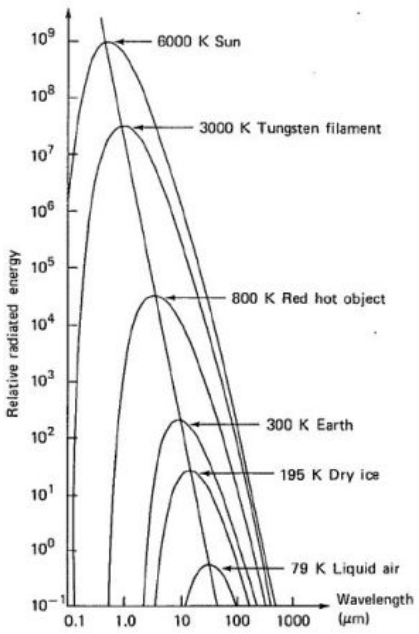

Figure 2.4: Spectral characterizations of selected black bodies (Skjöldebrand & Andersson, 1987).

The spectral distribution of a black body is described by Planck's law:

$$I_{\lambda,b}(\lambda,T) = \frac{2hc_0^2}{\lambda^5(\exp(hc_0/\lambda kT)-1)}$$

where $I_{\lambda,b}$ is the intensity of black-body radiation, λ the wavelength, h the Planck constant (6.63×10^{-34} m^2 kg s^{-1}), k the Boltzmann constant (5.67×10^{-8} Js^{-1} m^{-2} K^{-4}), c_0 the speed of light, and T the absolute temperature of the body. The spectral distribution has a maximum, and the corresponding wavelength, λ_{max}, depends on the temperature. Wien's displacement law describes how maximum intensity is shifted towards longer wavelengths at lower temperatures:

$$\lambda_{max} T = C$$

where C is a constant. The energy that radiates from a black body is described by the Stefan–Boltzmann law:

$$q = \varepsilon\sigma T^4$$

where ε is the emissivity and σ is the Stefan–Boltzmann constant.

2.1.5.2 Heat conduction

Conduction is heat transfer by molecular diffusion. Energy is transported in a medium due to a temperature gradient. In dense food products containing a substantial amount of water, conduction is the main means of heat transport. Heat transfer can be described by Fourier's law:

$$q = \frac{kA\Delta T}{x}$$

where q is the rate of heat transfer (heat flux), k the thermal conductivity, A the surface area, ΔT the temperature difference at a certain time, and x the thickness of the material; $\Delta T/x$ is

also known as the temperature gradient. As food is typically not homogeneous, the thermal conductivity of a food is usually not constant, but is dependent on the food's material properties. Conductive heat transfer is limited by the geometry and physical properties of a given food material.

2.1.5.3 Heat convection

When a fluid (liquid or gas) changes temperature, the resulting changes in density establish natural convection currents. Forced convection takes place when a stirrer or fan is used to agitate the fluid. Convective heat transfer through air is lower than through liquids. Condensing steam produces higher rates of heat transfer than does hot water at the same temperature, and the presence of air in the steam reduces the rate of heat transfer (Fellow, 1988). The rate of heat transfer can be expressed as:

$$q = hA(T_1 - T_2)$$

where h is the local heat transfer coefficient, A the surface area, and T_1 and T_2 are the surface and ambient temperatures, respectively. The heat transfer strongly depends on the velocity of the gas and the thermal boundary layers of the powder.

2.2 Herbs and spices

Herbs and spices comprise a large group of powdered foods used for their flavour, taste, colour, and aroma due to their distinctive seasoning and flavour-enhancing ingredients. They are mostly untreated, dried and/or mechanically processed plant material, such as roots, rhizomes, bulbs, barks, leaves, flowers, fruits, seeds, or the entire plant; most have their origin in tropical or subtropical environments (Gerhardt, 1974). Spices, herbs, and vegetable seasonings can be heavily contaminated with micro-organisms because of the environmental and processing conditions under which they are produced.

Spices can generally be stored for 6–9 months at 20°C at a relative humidity of 72–75% without any microbial spoilage. The higher the water content of the powder, the shorter the storage time. Fig. 2.5 shows some further rules of thumb regarding handling and storing spices (Gerhardt, 1974).

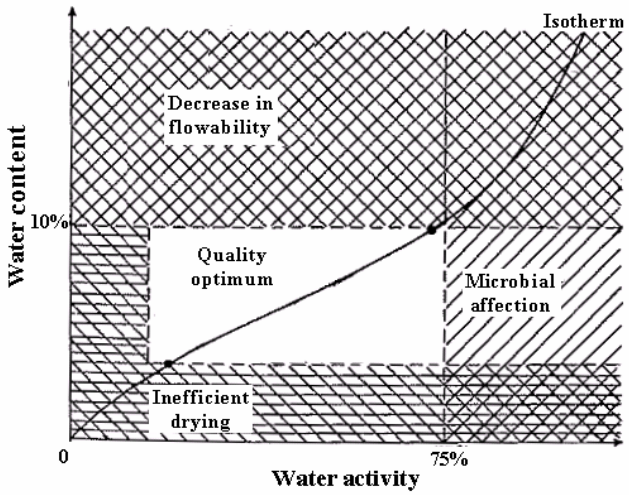

Figure 2.5: Schematic of optimal conditions for handling and storing spices (Gerhardt, 1974).

There are approximately 40 to 50 spices of *global* economic and culinary importance. However, many other species are used in traditional cooking in the regions of their natural occurrence. The annual quantity and value of the traded spices in 2002 are given in Table 2.3. Pepper (i.e., black or white pepper) topped the list accounting for 20% of the total value followed by capsicum (18%), vanilla (13%), nutmeg/mace/cardamom (9%), spice seeds (8%), and ginger (6%). The value of the global spice trade in 2002 was estimated at US$2–2.4 billion. However, the value of domestic spice consumption in many spice-producing countries is excluded and is itself substantial.

It is estimated that approximately 85% of internationally traded herbs and spices are dried and cleaned for use in a crude form without further processing. Traditionally, India and the Southeast Asia area are the biggest spice-exporting areas. A summary of most important spice-producing countries in 1998 and 2002 is given in Table 2.4. The largest spice importer is the EU, followed by the USA and Japan. In the EU countries, 55–60% of all spices and herbs are used by the food industry, 35–40% by the retail sector, and 10–15% by the catering sector (Douglas et al., 2005).

The increasing industrial sector use reflects the growing popularity of ready-to-use spice mixes. It also reflects the increasing consumption of processed foods and ready-to-eat dishes, which often rely on spices and herbs to enhance flavour. As well, the food industry has been actively promoting interest in exotic foods as a promising growth area. Advertising and media promotion by means of television cooking programmes, radio, and magazines has also stimulated spice demand. Ethnic groups have shops dedicated to their national foods, and supermarkets sell authentic ethnic products that are quick and easy to prepare. These developments have stimulated a wider range of food choice in home cooking and increased the demand for herbs and spices (Douglas et al., 2005).

Table 2.3: Total global imports of herbs and spices in 2002 (Douglas et al., 2005)

Item in 2002	Quantity (t)	Value (1000 US$)
Pepper, whole	236,999	403,136
Pepper, ground	25,079	73,943
Capsicum	323,688	451,855
Vanilla	5,015	308,086
Cinnamon, whole	76,981	104,052
Cinnamon, ground	13,567	20,306
Cloves, whole/stem	28,151	122,627
Nutmeg/mace/cardamom	42,33	229,452
Spice seeds	195,564	200,916
Ginger	230,744	141,536
Thyme/saffron/bay leaves	16,996	79,476
Spices, mixtures	180,491	313,806
World Total	**1,375,605**	**2,449,191**

Table 2.4: Main spice-exporting countries by value in 1998 and 2002 (Douglas et al., 2005)

Exporting Country	1998 (1000 US$)	2002 (1000 US$)
China	189,861	244,365
Madagascar	64,909	226,578
Indonesia	354,069	219,001
India	306,575	208,918
Guatemala	60,467	107,513
Brazil	119,161	105,801
Vietnam	85,142	103,316
Sri Lanka	80,117	83,876
Others	1,175,417	1,149,823
World Total	**2,435,718**	**2,449,191**

2.2.1 Sensory properties

The use of powdered components in spice mixes, aroma additives, and functional ingredients has increased in the food industry, especially in ready-to-eat meals and highly spiced cuisine (Sloan, 1993; Giese, 1994). According to Pruthi (1980), some spices impart a warm aroma with a tangy bitterness that gives them piquancy and a refreshingly clean taste; others have a smooth and mild flavour that intrigues the palate and stimulates the appetite. Some exceptional spices are very pungent, bitingly hot, racy, and strong smelling; others are crisp and succulent, with almost no pungency.

The *aroma and flavouring properties* of all spices are attributed to their essential oil and oleoresin contents. The volatile oils, called essential oils, are responsible for the characteristic aromas of spices. The content of oleoresins, which are non-volatile, can be extracted using alcohol or other chemical agents and are responsible for the typical taste and flavour peculiarities of spices. Essential oils are obtained from plants or their parts by water and/or steam distillation or by enzymatic treatment followed by steam distillation. Most essential oils are sensitive to high temperature, oxygen, and chemical agents. According to Pruthi (1980) essential oils consist of mixtures of:

(1) Hydrocarbons, e.g. terpenes, which oxidize easily under the influence of air and light or improper storage conditions due to their unsaturated character;
(2) Oxygenated compounds, e.g. alcohol, esters, aldehydes, and ketones, which are more soluble in dilute alcohol and more stable against oxidation;
(3) A small percentage of non-volatile residues, e.g. paraffin and waxes.

Heat treating spices influences their chemical and physical properties. Chemical properties affect sensory properties such as colour, taste, and aroma, whereas the physical properties influence handling properties such as swelling and rehydration capacity (Luning et al., 1995). Changes of flavour or taste in spices caused by heating have been extensively investigated for meat products. At temperatures of 90°C, some of the taste components weakened, and even more of these components weakened at temperatures exceeding 120°C. However, the degree of weakened also depended on the heating duration. On the other hand, the aroma of some spices increased by heating at 120°C. Taste stability decreased for white pepper – whether

natural spice or essence – between 70 and 90°C. At a higher temperature of 120°C, the natural spice displayed a larger decrease in flavour than was observed for the essence. For paprika, the taste stability was constant up to 130°C (Tändler et al., 1978).

The degree of *pungency* ("hotness", or more correctly, piquancy) can be measured in number of Scoville heat units (SHU) using the Scoville organoleptic test. Recently, however, this test has been replaced by high-performance liquid chromatography (HPLC) (Douglas et al., 2005), which identifies spice-specific piquant compounds, quantifies their levels, and compares them with the reference capsaicinoid content of the capsicum genus. Capsaicinoids stimulate chemoreceptor nerve endings in the skin, causing the burning sensation associated with chilli.

The combined character of pungency and taste vary markedly, however, depending on the chemical. In certain cases, chemicals having a taste sensation are also odour contributors. Piperine, responsible for the "bite" sensation in pepper, and capsaicinoid are odourless, while eugenol, which produces a familiar burning sensation, also has an odour (Pruthi, 1980).

Colour, or, more correctly, visual appearance in general, is a sensory attribute of freshness and taste (Setser, 1983). It plays an important role in evaluating the maturity, nutrition value, processing conditions, and storage life of spices and in setting federal and state grade specifications for foods (Little, 1964). It has been reported that many reactions, such as non-enzymatic browning (Maillard reaction), enzymatic browning, caramelization reactions, and oxidation, can affect colour during the thermal processing of foods (Maskan, 2001). The most common way of analyzing colour is the LAB method, in which the colour is analyzed in terms of three parameters, i.e., lightness – L, redness–greenness – a, and yellowness–blueness – b.

2.2.2 Antimicrobial activity

Many of the spices and herbs used today have been known for their preservative and medicinal powder since ancient times. The inhibition of micro-organism growth by spices and herbs is mainly due to the antimicrobial activity of their essential oils and other extracts. The antimicrobial activity varies depending on the particular micro-organism, spice or herb, and medium. According to Zaika (1988), the antimicrobial effects reported for some spices were:

Strong for cinnamon, cloves, and mustard,

Medium for allspice, coriander, cumin, rosemary, thyme, oregano, caraway, and sage, and

Weak for black pepper, paprika, and ginger.

Possible differences in the active component composition of spices should be considered. These differences may be due to geographic origin, climate, processing, or varietal differences. The sensitivity of micro-organisms to particular test substances may depend on the micro-organism strain (Zaika, 1988). Spices affect all stages of microbial growth: the lag phase is extended, the growth rate during the logarithmic phase is decreased, and total cell yield is reduced. Fungi are more sensitive to spices than bacteria are. Most spices are more active against Gram-positive than Gram-negative organisms. Little inhibition of bacterial growth in foods can be expected from a single spice component due to the small added amounts. However, since multiple seasonings, i.e. spice mixes, are frequently added to foods, the effects of combinations of several spices with other antimicrobials and processes must be considered. Seasoning meat and fish with high levels (i.e. 10% or higher spice content by weight) can be found in some parts of the world. In foods processed under sanitary conditions characterized by relatively low microbial populations, the antimicrobial effects of spices may offer substantial protection against fungi, Gram-positive bacteria, and, to some extend, also against Gram-negative bacteria (Shelef, 1983).

2.2.3 Characteristics of paprika

Paprika (*Capsicum annuum*) is a ground, dried fruit used to improve food taste and colour. Paprika belongs to the *Capsicum* genus, which has its origin in South America and was introduced to Europe by the Spaniards in the middle ages. In the 17th century, the cultivation and use of capsicum varieties as a condiment spread around the world. The most important producers of paprika powder are today Hungary, Spain, the USA, Morocco, and the Balkan area.

2.2.3.1 Nature and climate

Capsicum varieties are annual herbaceous plants (Fig. 2.6) belonging to the nightshade family (*Solanacea*). Beside paprika, other known varieties of capsicum are red and green chillies, cayenne peppers, Thai peppers, Korean red peppers, and the bell peppers (a non-pungent fruit vegetable). The pungency of capsicum varieties ranges from the mild-tasting bell peppers (used in salads), to the bland seasoning paprika or cayenne peppers, and strong-flavoured, more "burning" chilli peppers.

Figure 2.6: *Capsicum annuum* L. showing (1) flowering branch, (2) vertical section of flower, (3) unripe fruit (berry), (4) transverse section of fruit, and (5) ripe fruit (Rosengarten, 1969).

Capsicums vary tremendously in fruit and foliage size, shape, and colour, due to the different soils and climates where they are cultivated, and probably also due to human selection. The ripe, fleshy pericarp matures together with the round or elongated fruit, until it

losses its entire chlorophyll content and develops a high carotenoid concentration with different levels of esterification. Rainfall of 600–1250 mm is desirable over the growing season, but no rain is needed as the fruits ripen. Capsicums flourish in warm sunny conditions, and require 3–5 months of temperatures in the 18°C–30°C range; below 5°C, growth is retarded, and frost kills plants at any growth stage. A seedbed temperature of 20–28°C is the optimum for germination (Mínguez-Mosquera et al., 1994).

2.2.3.2 Powder production

Paprika powder is prepared by drying and then grinding the whole ripe fruit. Paprika has a thick waxy skin, which prevents rapid drying.

Sun drying the fruit is the most common drying method, and is done by spreading the crop in a dry area, either exposed to the sun or under cover. Fruits are spread in thin layers on a hard dry surface and regularly stirred to ensure uniform drying, so as to reduce discolouration and fungal growth. Drying takes up to 15 d depending on the sunshine hours and weather conditions.

Industrial drying can be done in hot-air dryers or drying chambers heated by burning oak logs, in which the smoke given off by the logs impregnates the fruits with an aroma and taste very highly prized by the consumer. The drying temperatures should remain below 60°C and the operation is performed until a water content below 10% is reached, whilst retaining colour and pungency. Approximately 25–35 kg of dried spice can be produced from 100 kg of fruit (Mínguez-Mosquera et al., 2000). The sorption isotherms of hot paprika powder are shown in Fig. 2.7.

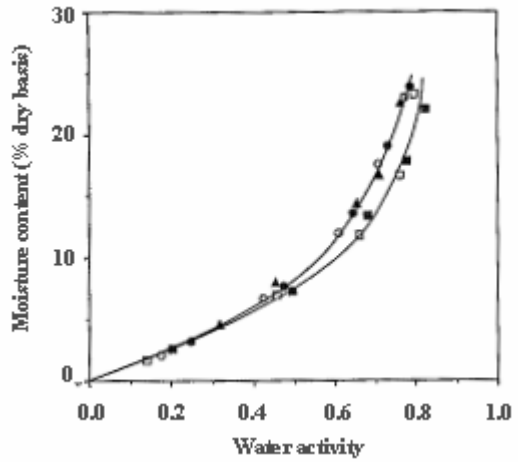

Figure 2.7: The sorption isotherm of hot paprika powder at 20°C comprising: (■) coarse powder with seeds, (□) fine powder with seeds, (●) coarse powder without seeds, (○) fine powder without seeds, and (▲) whole peppers (Lee et al., 1991).

2.2.3.3 Quality aspects and use

Product qualities are often negatively affected by drying, but also depend on other factors including the cultivar, moisture content, ripeness, and health status of the dry fruits before grinding and drying. High-quality paprika is deep red and has a minimum pungency. Since the highest concentration of carotenoid pigments occurs in the pericarp of the fruit, powders containing only pericarp will have the highest pigment content and thus be evaluated as being of the highest quality. Paprika powder has a carotenoid concentration lower than that of the whole fruit and fine powder has a lower concentration lower than coarse powder. More than thirty native paprika pigments have been isolated, consisting primarily of mono- and difatty acid esters of carotenoid alcohols (mainly capsanthin and capsorubin), plus smaller quantities of ß-carotene, zeaxanthin, and ß-cryptoxanthin. Other advantages of using paprika are related to its considerable volatile oil content and high amounts of carotenoids (which are natural antioxidants), ascorbic acid, and tocopherol (which are especially abundant in ripe fruits) (Simal et al., 2005). Other quality parameters of paprika powder, besides the carotenoid content, are particle size, water content, and microbiological quality. Paprika is known for its high microbial counts of 6–8 $\log_{10}$ CFU/g, typical contaminants being *E. coli*, Salmonella, *B. cereus*, and other spore formers (McKee, 1995).

Carotenoid loss in paprika powder is affected by a_w, storage temperature, atmosphere, light exposure, and treatment (Lee et al., 1992; Mínguez-Mosquera et al., 2000). Drying temperatures of 80 and 90°C produced more carotenoid degradation than did drying at 60 and 70°C (Malchev et al., 1982). The initial carotenoid concentration is strongly affected by the extent of powdering and whether or not seeds are included in the powder. The presence of seeds in paprika powder also markedly increases the concentration of lipids and lipogenase shown to be involved in the oxidation of the carotenoid pigments. Enzyme activity has been demonstrated in paprika spice, and it is likely that the characteristic paprika aroma arises in part from oxidative processes occurring during drying and storage.

It is recommended that paprika be stored in the form of coarse powder with seeds, at an a_w of 0.3–0.5 in a nitrogen atmosphere; longer-term storage at over a_w 0.70 is not recommended due to microbiological problems, for example, moulds can grow in paprika at relative humidities over 82% (Kim et al., 1984; Lee et al., 1991). No differences in colour loss have been noted between seed-containing and non-seed-containing samples for up to 5 months of storage, but by the end of the subsequent 7 months, seed-containing paprika lost less colour than did paprika without seeds. During storage, the content of ascorbic acid and tocopherol decreased by 90% after 5 and 2 months, respectively (Biacs et al., 1992).

2.3 Microbial contamination of spices

Herbs and spices are known to be a significant source of microbial contamination, due to their contact with soil, dust, excrement, insects, and other contaminants during growing, harvest, processing, storage, and transport. Such contamination is also related to the warmth and humidity of their tropical counties of origin, combined with unsophisticated traditional operations during processing, poor storage facilities, and often small-scale production units in these developing countries. Typically, for example, the spice may be harvested and sorted by hand, then dried by being spread on a sun-exposed surface outdoors, and finally packed when dried. Poor sanitary conditions during this processing commonly result in microbial contaminations of 6–8 $\log_{10}$ CFU/g (McKee, 1995).

The micro-organisms present in fresh spices have their origin in soil and air, and include pseudomonades, moulds, *Enterobacteriaceae*, bacilli, and clostridia. However, in dried herbs and spices it is primarily spore-forming micro-organisms that are able to survive, mainly the spores of *B. cereus*, *B. stearothermophilus*, and *B. coagulans* and the conidia of moulds of *Aspergillus* species (Modlich & Weber, 1993).

2.3.1 Characteristics of bacterial spores

One successful adaptation of micro-organisms by which they resist heat exposure is their ability to produce a heat-resistant form, i.e. the spore. The most important spore formers are members of the *Bacillus* and *Clostridium* genera (Earnshaw et al., 1995). Bacterial spores have a higher resistance to heat, radiation, desiccation, pressure, electric shock, and chemical agents than observed in vegetative cells of the same species (Smoot & Pierson, 1982). It is difficult, however, to explain exactly why micro-organisms are more resistant under dry conditions. One reason for the spore resistance is undoubtedly that water is very limited in spores, and proteins have been shown to be more stable at low a_w (Corry, 1973). Another reason is their metabolic dormancy; respiratory activity in spores can be as low as 10^{-4} of the maximum rate present in vegetative cells metabolizing substrate (Lewis, 1969). Although mature spores are metabolically dormant, they contain several substrates as well as enzymes that might be expected to act completely on these substrates within 10–20 min. The only two mechanisms likely to account for the almost absolute enzyme inhibition required for the enzymatic dormancy of spores are (1) the presence of inactive forms of enzymes, zymogens,

in the spore and (2) the inhibition of enzyme activity by the absence of water. The latter is likely the key mechanism, as dehydration could also inhibit zymogen activation (Setlow, 1994).

The spore itself is multi-layered, consisting of an inner core surrounded by the plasma membrane, primordial cell wall, cortex, and exterior coat (Fig. 2.8). In addition, spores of *B. cereus* are surrounded by a loosely attached exosporium with surface appendages possessing, for example, adhesion properties (Warth, 1978). The spore coat functions as a barrier protecting the interior parts of the spore from a wide range of deleterious substances, such as surfactants and enzymes. The next layer is the cortex, which plays a vital role in maintaining dormancy. The cortex is responsible for maintaining the dehydration of the spore core, which is essential for maintaining dormancy and the ability to resist heat damage (Earnshaw et al., 1995). The inner layer is called primordial cell wall because after germination it develops into the cell wall of the outgrowing vegetative cell. The inner part of the spore, the core, is quite similar to the vegetative cell; however, the inner part of the core has a very low water content and a high concentration of dipicolinic acid (DPA, Fig. 2.9) and Ca^{2+}. The fundamental transformation of a bacterial endospore into a vegetative cell involves at least three sequential processes: (1) activation, (2) germination, and (3) outgrowth (Berg & Sandine, 1970).

(1) Activation is a reversible process resulting in a spore that retains its typical heat resistance, non-stainability, refractility, and DPA content, but is no longer metabolically inactive (dormant). Activation is usually a required step in conditioning the spore for germination.

(2) Germination is an irreversible process resulting in a cell that has lost the typical characteristics of a bacterial spore. During germination, no macromolecule synthesis occurs, but spore components are broken down. Spores can be activated by sublethal heat, water, reducing agents (e.g. ethanol), low pH, Ca-DPA, ionizing radiation, various chemicals (e.g. D-cycloserine and urea), and aging (Smoot & Pierson, 1982; Kim & Foegeding, 1990).

(3) Outgrowth is a process in which new macromolecules are synthesized; new non-spore proteins and structures are formed, ultimately leading to the emergence of a new vegetative cell.

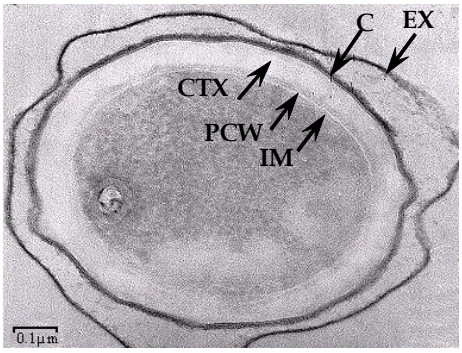

Figure 2.8: Section of free spore of *B. cereus* showing spore coat layer (C), exosporium (EX), cortex (CTX), primordial cell wall (PCW), and inner membrane (IM) (Gould & Hurst, 1969).

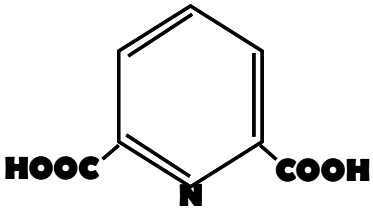

Figure 2.9: Chemical structure of dipicolinic acid.

2.3.2 Characteristics of *Bacillus cereus*

B. cereus is a spore-forming organism commonly found in soil, air, dust, water, and in many raw and processed foods, such as rice, cereals, corn flour, vegetables, spices, meat, milk, and dairy products. One of the earliest food poisoning outbreaks caused by *B. cereus* was reported in 1906 (Borch & Arinder, 2002), and up to 23% of reported food poisoning outbreaks in Europe and North America are caused by it. This micro-organism is of major concern for the food industry because it causes the deterioration of foods by producing lipases, proteases, and polysaccharides (Andersson, 1998). Hungarian meat and meat dishes, such as *goulash*, are highly seasoned with spices that often contain large numbers of aerobic spores. From 1960 to 1966 in Hungary, *B. cereus* ranked as the third most common cause of food poisoning (Murrell, 1989).

B. cereus is a Gram-positive, facultative anaerobic; it is endospore forming with mobile rods. Because the genus *Bacillus* encompasses such great genetic diversity, it has been subdivided into six groups, based on both phenotypic and genetic properties, and it has been suggested that *B. cereus* belongs to the *B. subtilis* group (Priest, 1993). *B. cereus* is characterized as large celled, being over 0.9 µm in diameter and between 3–5 µm in length, and it produces a central ellipsoidal endospore (Sneath, 1986).

This bacterium is widespread because of its ability to sporulate and adapt to various environments and is commonly found in a range of foods, including meat and milk products. Although pasteurization kills the vegetative bacteria, endospores of *B. cereus* that survive processing conditions can germinate and pose a risk of toxin production (Sinigaglia et al.,

2002). The bacteria are able to produce several enterotoxins and one emetic toxin causing diarrhoeal and emetic types of food poisoning, respectively. Toxin production depends on the cultural medium and bacterial growth conditions. The enterotoxins are produced in the exponential growth phase and the emetic toxin is probably produced through the modification of food components (Andersson, 1998). A population of at least 6–7 $\log_{10}$ *B. cereus*/g is thought to be necessary to cause illness in healthy adults. Infants, children, and the immunosuppressed are most likely to experience illness caused by lower populations in the range of 3–5 $\log_{10}$ CFU/g (Jaquette & Beuchat, 1998).

Factors affecting the growth and survival of *B. cereus* are the a_w, pH, and temperature of the ambient medium (Quintavalla & Parolari, 1993) (Table 2.5). Growth of vegetative cells typically occurs at 10–50°C, while the temperature range for germination is 5–60°C, with an optimum at 30°C (Johnson, 1984). Spores of *B. cereus* germinate at a_w values below 0.80 and outgrow at a_w values of 0.90 or greater (Hagen et al., 1967).

Table 2.5: Growth conditions for *B. cereus* (Kramer & Gilbert, 1989; Murrell, 1989)

Factor	Minimum	Optimum	Maximum
Temperature	5–15	28–37	35–50
pH	4.35–4.90		9.30–10.50
a_w	0.912–0.950		

The thermal inactivation of *B. cereus* is determined by the $D_{95°C}$ value – the decimal microbial reduction time at 95°C – which is reportedly 1.2–36.2 min (Anonymous, 2001), depending on the strain and the composition of the heating substrate. Cooling from 54 to 7°C, at linear cooling times of several hours. apparently halted growth. It is the psychotropic stains of *Bacillus* that are of concern for food stored at refrigeration temperatures; tests of some psychotropic *Bacillus* strains, mainly isolated from dairy products, indicated a probability of growth even at temperatures below 10°C (Fig. 2.10). Toxin production is reportedly higher at 12–15°C than at 30°C (Borch & Arinder, 2002). However, some strains are noted for their need for and response to heat activation, and after heat activation for 10–15 min at 80°C or 3–5 h at 65°C, they germinate and outgrow very rapidly in suitable media. Generation times of approximately 23–25 min have been observed at 30°C, which is remarkably rapid at that temperature compared with the growth rate of many organisms. These two properties perhaps explain why the consumption of foods, such as custard and sauces, which have been heated at

a sublethal temperature during cooking and then held overnight at room temperature, commonly results in food poisoning from *B. cereus* (Murrell, 1989).

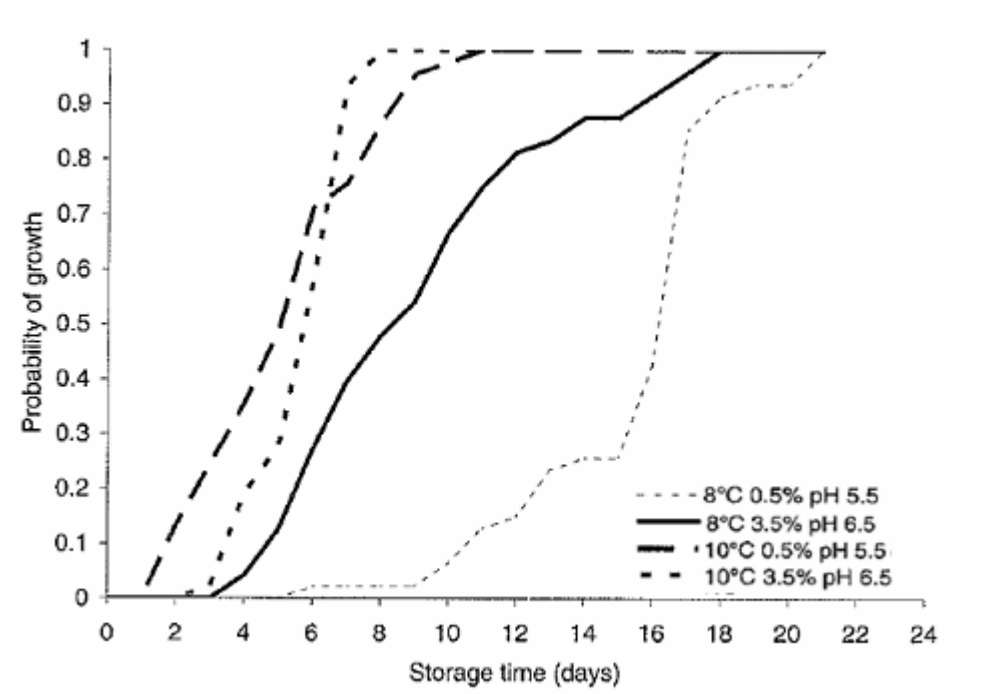

Figure 2.10: The probability of growth of psychotropic *B. cereus* (N_0 = 4 $\log_{10}$ spores/mL) in BHI broth of varying pH and salt levels at 8 and 10°C. Growth was recorded as change of absorbance (Borch & Arinder, 2002).

2.3.3 Microbial inactivation

The effect of heat treatment on micro-organisms is evaluated by exposing them to increasing doses of heat and determining the microbial numbers surviving before and after treatment. Viability is judged from the ability to multiply and can be determined conventionally using viable count techniques.

Sensitivity to heat varies greatly between different micro-organism species, and also between strains of the same species. Exposing microbial cells to high temperatures does not simply affect one specific target in the cell, as the thermal energy in the cell is integral to an entire complex system. This energy not only affects the cell as a whole, but also affects each individual constituent – the cell structure, molecules, and reactions. Cells contains several targets for the action of heat, so it can be proposed that the basal heat resistance of micro-organisms may be due to the intrinsic stability of macromolecules, ribosomes, nucleic acids, enzymes, and proteins inside the cell and the membrane. The prime cause of cell death from

thermal injury is still not clearly defined. Understanding the exact target and mechanisms of heat damage can give clues as to the opportunities for the combined application of heat and other preservation measures (Earnshaw et al., 1995).

In methods for thermally reducing bacterial numbers, such as pasteurization, the death rate is logarithmic, as is the rate of cell growth. The process is dependent on both the exposure temperature and the time required at this temperature to attain the desired rate of destruction. A direct relationship may be obtained by plotting time at a particular temperature against the $\log_{10}$ of the number of organisms, or more usually, the log surviving fraction, since this allows better comparison of the different survival curves. Thermal reduction calculations thus require knowledge of the concentration of micro-organisms to be destroyed, the acceptable concentration of micro-organisms that can remain after treatment, the thermal resistance of the target micro-organisms, and the temperature time relationship required for destruction of the target organisms (Goff, 1995). Fig. 2.11 presents the different types of heat-inactivation curves (Corry, 1973):

(A) This straight-line relationship has been interpreted as indicating that cell death is caused by the inactivation of only one site per cell, giving a first-order reaction relationship.

(B) Another common type of survival curve is that in which there is an initial "shoulder" before death becomes exponential.

(C) With spores, there may be an initial apparent increase before the numbers decrease, due to the activating effect of heat on spore germination.

(D) Occasionally, a two-phase curve may be obtained; this may indicate a mixed culture, or less commonly, a population containing a certain proportion of more resistant cells.

(E) This type of curve may represent the presence of cells with a wide range of resistances. The observed "tailing" may be due to a very small proportion of resistant cells, but is more often due to the protection of a few cells due to the drying effect, to lower temperatures because of evaporation from the surface of the liquid during heating, or to errors counting the cells given the small sample sizes involved. Clumping of cells can also result in "shoulders" on survival curves.

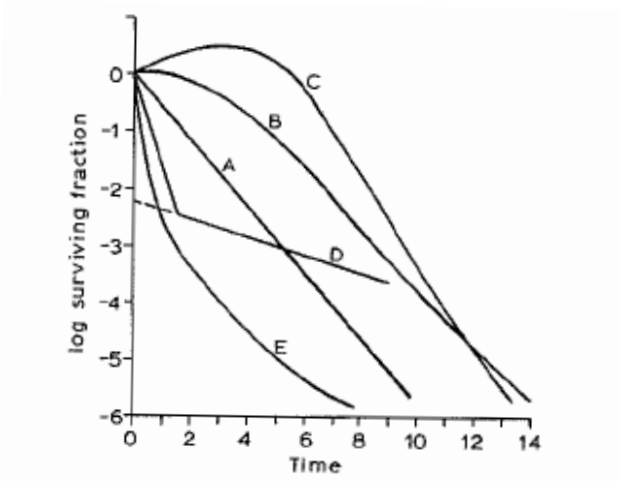

Figure 2.11: Types of microbial inactivation (Corry, 1973).

Death rates of a linear character (i.e. the straight part of an inactivation curve) can be described by the general formula:

$$\ln\left(\frac{N}{N_0}\right) = -k \cdot t$$

where slope k is obtained from the linear regression analysis of the survival count (N) relative to the initial number of micro-organisms (N_0), plotted logarithmically versus time (t).

By integrating, the following linear first-order differential equation is obtained:

$$\frac{dN}{dt} = -k \cdot N$$

The heat resistance of a micro-organism is usually expressed as the D-value, calculated as the treatment time required at a particular temperature to reduce the number of organisms by 90%, i.e. 1 $\log_{10}$ (Fig. 2.12). The D-value of an organism at any particular temperature is affected by the a_w and by various environmental factors, such as the presence of proteins, fats, salts, and amino acids as well as pH, age of culture (whether it is in the log phase, stationary phase, etc.), and even recovery medium.

If the $\log_{10}$ D-values for an organism are plotted against temperature, another straight line is obtained, called the z-value, which reflects the temperature dependence of a reaction (Fig. 2.13). The z-value indicates the increase of temperature required to effect a tenfold change in the D-value. The slope of this line is fairly constant for a given organism, regardless of

variables such as heating medium. Reactions that have low *z*-values are highly temperature dependent, whereas those with higher *z*-values require larger changes in temperature to reduce the needed treatment time. The general *z*-value for a spore-forming bacterium is approximately 10°C (Ohlsson, 1980).

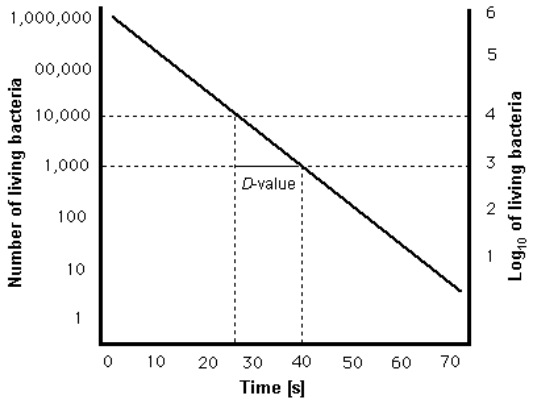

Figure 2.12: Determination of the *D*-value as a function of the survival rate of a micro-organism and treatment time, depicted for a *D*-value of 15 s (Goff, 1995).

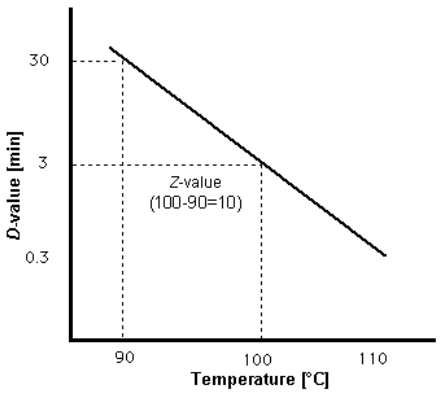

Figure 2.13: Determination of the *z*-value as a function of temperature and decimal reduction time, depicted for a *z*-value of 10°C (Goff, 1995).

2.4 Decontamination techniques for food powders

The micro-organisms present in dried spices cannot grow or multiply because of the small amount of available water. Nevertheless, these micro-organisms are still viable and retain the potential to multiply when the product is rehydrated. It is thus important to decontaminate food powders efficiently, especially if these powders are to be incorporated into a more complex preparation with a higher water content. The decontamination of dried powders is difficult, and the difficulty correlates with the presence of spores adapted to low water contents. The availability of water has a pronounced effect on the heat resistance of micro-organisms, and the resistance of dried micro-organisms is many times higher than the resistance of the same micro-organisms in an aqueous solution (Laroche & Gervais, 2003b; Fine & Gervais, 2005).

The first research treating the decontamination of spices is from the 1930s; at that time, the impact of spices on microbial decay had already been described. Jensen et al. (1934) pointed out that sterile ham could be contaminated by spore-forming bacteria from the added spices, causing swelling of the packed ham. Hall (1938) showed that shelf life of canned food was negatively affected by the addition of spices. He reported on experiments examining the reduction of the microbial population in spices by applying heat, formaldehyde, electricity, or a combination of heat, ethylene oxide, and carbon dioxide. The latter method was already commonly used at that time, and satisfactorily reduced microbe populations.

More current literature presents a considerable amount of data regarding the inactivation of micro-organisms in moist environments. However, there is less comprehensive information regarding the heat resistance determined in dry foods. Many studies have demonstrated that micro-organisms can be killed dramatically during treatment on standard media, such as agar. Much of the available data are derived from experiments using artificial matrixes, such as glass cover slips, stainless steel, or hydrophobic membranes; these data may not be representative of foods, which are complex substances involving distinctive interactions between food components and attached micro-organisms (Archer et al., 1998).

Most preserved foods have traditionally been thermally processed at a temperature of 60–140°C for a few seconds to a few minutes. High-temperature treatment can cause significant loss of flavour and aroma from a spice because the volatile oils are lost. Steam also results in a loss of volatile flavour and aroma components, colour changes, and increased moisture

levels. Thermal treatments have the problem of being dependent on the water content of the powder; powder can absorb as much energy as there is water available in the powder, i.e. temperature increase is unavoidable. Due to the large surface of the powder particle, water evaporates during heating, simultaneous with the possible release of volatile oils. For that reason, the optimal water content has to be found to achieve as high a microbial reduction as possible, accompanied by acceptable loss of volatiles and sensory quality (Modlich & Weber, 1993).

Thermal treatments are problematic for many powders, due to their sensitivity to heat; this has led to efforts to preserve foods using non-thermal methods that use less energy than do thermal processes (Barbosa-Canovas et al., 1998). For pasteurizing dried food/powders, the suggested non-thermal decontamination processes are ethylene oxide fumigation, gamma irradiation, UV-light irradiation, and treatment with various chemical substances. However, these processes have problems of their own. The use of ethylene oxide was prohibited by an EU directive in 1991 and has been banned in a number of other countries because of its carcinogenic by-products. Gamma irradiation effectively kills micro-organisms and can be used practically on a commercial scale, but this treatment is not always accepted by consumers and is not allowed in all countries (Dehne & Bögl, 1993). The efficiency of UV light is limited by its low degree of penetration and limited ability to treat “off-side” areas of food (e.g., portions in the shade, in pores, or in orifices) due to the “shadow effect” (Guerrero-Beltrán & Barbosa-Cánovas, 2004).

Alternative methods to decontaminate spices are widely described in the literature. Most of the novel decontamination techniques are mostly based on exposing the spice to a combination of moist environment (steam) and temperature (heat). To decontaminate dried food products, the suggested thermal decontamination processes are high-temperature short-time treatments (HTST) (Bueltermann, 1997; Laroche & Gervais, 2003a), dry heat (Baron et al., 2003) or steam (Schneider, 1993), and microwave (Gerhardt & Romer, 1985) or IR heating (Hamanaka et al., 2000). Innovative decontamination treatments using the HTST concept or pulsed UV light are promising and will be applied more frequently in the future, as they are highly effective and maintain high product quality (Kabelitz, 2007).

The following sub-chapters present a selection of possible thermal and non-thermal decontamination treatments for food powders. Radiative heating using infrared radiation, as a novel decontamination technology, is separately discussed in section 2.5.

2.4.1 Gamma irradiation

For the irradiation of food material, cobalt (Co^{60}) and caesium (Cs^{137}) are used as the source of radiation with energy bands of 1.17–1.33 and 0.66 MeV, respectively. Gamma rays (γ-rays, Fig. 2.14) penetrate up to several meters into material, and can even be used to decontaminate packed and bulk material. During treatment, security measures, such as the wearing of thick lead coats and the use of ray-proof locks and water-immersible radiation sources, must be taken in the vicinity of the irradiation chamber to protect workers. Spores are more resistant to γ-rays than vegetative cells are (Modlich & Weber, 1993).

The accepted gamma radiation dose, according to WHO/FAO recommendations, is 10 kGy for food, resulting in a dramatic reduction of the germ population. Rates of 5 and 10 kGr have been found to be sufficient to decrease the population of spore-forming flora in spices, and to inhibit the fungal flora and coliforms that contaminate black pepper powder (Emam et al., 1995). In that dose range, the character and chemical composition do not change noticeably, i.e. enzymes remain active, unlike in thermal treated powders (Müller & Theobald, 1995). Applying γ-rays is restricted in many countries, but exceptions are made for certain spices; due to customer suspicion and resistance, however, and the required labelling of irradiated foods, industry does not use γ-radiation on a larger scale on spices.

2.4.2 Fumigation

For quick, economical, and effective disinfection, fumigation with a suitable toxic gas or vapour is a recommended treatment, particularly for contaminants in the form of insects or other small animals. As the fumigant can move both into and out of the commodity, the chances of chemical residues in the commodity are greatly reduced.

The most effective fumigants are: methyl bromide, ethylene dibromide, or a mixture of the two; ethylene oxide alone or in admixture with CO_2 or methyl formate (1:1); and aluminium phosphatide or phosphine. Formerly, the most commonly used fumigant was ethylene oxide – i.e. $(CH_2)_2O$, or Etox – which was a reliable method for spice decontamination. Its use is now prohibited in the EU, but is still allowed in the US and Australia. The drawback of Etox is that it leaves a residue after treatment, which rapidly decreases, however, after two weeks of storage. In contrast, the concentrations of certain other fumigants, i.e. ethylene chlorohydrin, ethylene glycol, and chloro-2-ethanol, increase and remain for up to six months. However,

Etox is not very effective against spores (Pruthi, 1980). Decontamination tests found different microbial reduction rates, depending on the selected spice; for example, Etox effected total bacterial reduction in paprika and oregano powder, a 3 $\log_{10}$ reduction in black pepper, and no reduction in garlic (Vajdi & Pereira, 1973).

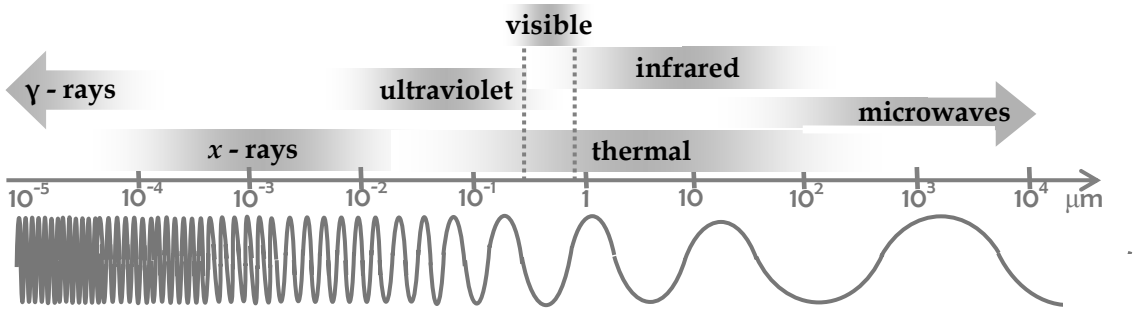

Figure 2.14: The electromagnetic spectrum.

2.4.3 UV light irradiation

Ultraviolet (UV) light occupies wavelengths in the non-ionizing region from 200 to 400 nm (Fig. 2.14) and can be subdivided into three regions according to the wavelength: short-wave UV (200–280 nm), medium-wave UV (280–320 nm), and long-wave UV (320–400 nm).

For microbial inactivation, short-wave UV light in the 250–260-nm range is lethal to most micro-organisms, such as bacteria, viruses, protozoa, moulds and yeasts, and algae. Monochromatic UV light (254 nm) is obtained by using low-pressure mercury vapour germicidal lamps. UV light acts as a physical method of microbial disinfection, and micro-organisms exposed to UV light are damaged at the deoxyribonucleic acid (DNA) level, by alteration of the microbial DNA. Once the DNA has been damaged, the micro-organisms can no longer reproduce and the risk of disease arising from them is eliminated.

The applications of the germicidal effects of UV light fall into three broad categories: (1) inhibition of micro-organisms on surfaces; (2) destruction of micro-organisms in air; and (3) sterilization of liquids (Bintsis et al., 2000). This method does not produce undesirable by-products that could change the sensory characteristics of a final product; it is a dry, cold process that can be simple, effective, and lower in cost than other sterilization methods (Bintsis et al., 2000; Guerrero-Beltrán & Barbosa-Cánovas, 2004).

However, the efficiency of UV light is limited by its low degree of penetration and limited ability to treat "off-side" areas of food ("shadow effect"), such as portions in the shade, in pores, or in orifices. Thus, the germicidal effect is obtained only by applying UV light *directly* to the target (Guerrero-Beltrán & Barbosa-Cánovas, 2004). Microbial cells can be even protected from UV light by moisture, proteins, fat, or other substances that absorb UV radiation or by being enclosed in fruits, leaves, seeds, roots, or cell agglomerates. The achieved reduction of cell numbers is also greatly dependent on the level of microbial contamination: lower microbial loads are harder to reduce, due to the lower probability of a germ coming into contact with a UV ray. The method can thus only be used on smooth surfaces or UV-transparent substances (e.g. thin layers of water), for sterilizing the surfaces of packaging materials (e.g. bottles, plastic cups, and aluminium foil), and for disinfecting media used in the pharmaceutical, electronic, and aquaculture industries. On the other hand, if the UV treatment is not performed properly, the opposite effect can occur, and micro-organisms can be activated, especially in the case of moulds (Modlich & Weber, 1993). Micro-organisms suspended in air displayed a higher reduction rate than did those in liquids, due to the low penetration of UV light through various physical media (Bintsis et al., 2000).

There are two modes of UV-light treatment: continuous (i.e. a constant light cycle) or pulsed (i.e. alternating light and dark cycles). Pulsed UV light is more effective and rapid at micro-organism inactivation than continuous UV light, because the energy released is multiplied many times. The power dissipation from a continuous UV-light system ranges from 100 to 1000 W, whereas a pulsed UV-light system can produce a peak power output as high as 35 MW. In pulsed UV-light treatment, the energy is stored in a high-power capacitor and released intermittently, producing several high-energy bursts in a short period of time (Krishnamurthy et al., 2004).

Pulsed UV light was tested for its ability to inactivate *E. coli* on the surfaces of alfalfa seeds and for its effect on subsequent germination. The parameters having substantial impact on decontamination were distance from the UV strobe, number of pulses (i.e. treatment time), and thickness of the seed layer. Faster inactivation times were achieved by using thinner seed layers and shorter distances from the UV strobe, but a greater than 4 $\log_{10}$ CFU/g reduction in microbial load could be achieved with all tested thicknesses simply by adjusting the number of UV pulses. Tests moreover indicated a satisfactory germination rate (Sharma & Demirci, 2003). The destruction of *Saccharomyces cerevisiae* dried onto glass beads was tested in a fluidized bed in order to avoid shadowed areas; the ambient water activity had no strong impact on microbial load reduction, but 58 J/cm^2 were needed to reduce the *S. cerevisiae* load

by 7 $\log_{10}$ (Fine & Gervais, 2004). Kuo et al. (1997) studied the surface sanitation of eggshells by reducing the *Salmonella typhimurium* load and the counts of aerobes, moulds, and yeasts. Pulsed UV light of 620 $\mu W/cm^2$ was applied in 1-min alternating light and dark cycles for 5 min and compared with continuous UV light of the same intensity. After 1 min of UV irradiation, a decrease of approximately 3 $\log_{10}$ CFU/g in S. *typhimurium* load was achieved. A population of approximately 2 $\log_{10}$ CFU/g was observed to remain, as the roughness of the eggshell surface shielded the bacteria from the radiation. The loads of aerobic microbes, mould, and yeast were reduced by 2 $\log_{10}$ CFU/g after 15 min of UV treatment (Jun et al., 2003).

2.4.4 Chemical sanitizing

Dormant bacterial endospores are not only resistant to heat, irradiation, and desiccation but also to various chemical substances. The use of chemical agents is a common non-thermal pre-treatment for inactivating spores in the processing environment or on surfaces (Kharde & Yousef, 2001; Setlow et al., 2002). The most commonly used sanitizers for eliminating spore contaminants are hydrogen peroxide (H_2O_2), chlorine (Cl_2), ethanol (C_2H_5OH), sodium hypochlorite (NaOCl) or calcium hypochlorite ($Ca(OCl)_2$), and ozone (O_3). However, much of the food material treated with those agents is not used for direct human consumption, but rather for crop seeding, due to the toxic, carcinogen, or mutagen residues remaining after the sanitation process (Piernas & Guiraud, 1997). At the time of the preceding study, the precise mechanisms of spore resistance or killing caused by acid, alkali, and ethanol were not fully understood. However, it is now known that *Bacillus subtilis* is killed by ethanol and strong acids due to the disruption of the spore permeability barrier, while spores are killed by strong alkali due to the inactivation of the spore cortex lytic enzymes (Setlow et al., 2002). Spore susceptibility to hydrogen peroxide is mostly related to enzyme inactivation in the spore, after which the core is unable to swell during germination (Melly et al., 2002).

Cl_2 and H_2O_2 have been successfully used to decontaminate processing environments, equipment surfaces, and occasionally even to disinfect the surfaces of solid foods, such as grains or vegetable seeds (Kharde & Yousef, 2001). An alternative to Cl_2 is the more effective chlorine dioxide (ClO_2), which has been successfully tested to reduce loads of the food-borne pathogens *E. coli* and *Listeria monocytogenes* on the surfaces of green peppers (Han et al., 2000, 2001).

Ozone, in both the gaseous and aqueous states, is another powerful antimicrobial agent that is suitable for use on food. However, in dry powders with water contents lower than 10% it has exhibited no effect; at least 14% residual moisture must remain for its use to be effective. Molecular ozone inactivates micro-organisms rapidly by reacting with intracellular enzymes, nucleic material, and components of the cell envelope, spore coat, or viral capsid. Ozone is generated on-site and decomposes quickly, leaving no residues, after application to food. As a sanitizer, ozone is active against all forms of micro-organisms at relatively low concentrations, but acts more rapidly when the targeted micro-organisms are suspended and treated in pure water or simple buffers than in complex systems such as food (Kharde et al, 2001). No carcinogenic or mutagenic products build up after treatment, but the oxidation of volatiles commonly occurs, so sensory changes can occur.

A common problem is the growth of *Aspergillus* moulds on the surfaces of nuts, seeds, and flour; this produces aflatoxin, which poses serious health risks. Various sanitizers, such chlorine gas and sodium hypochlorite, have been tested and found to produce a rather poor reduction of aflatoxin in peanuts, corn, and copra meal (Samarajeewa et al., 1991). Many seed sanitation treatments have been studied as a means to reduce the risk of illness associated with sprouts, such as alfalfa sprouts. Several different seed treatments have been investigated for their potential to inactivate food-borne micro-organisms by inhibiting their ability to germinate/sprout. Chemical treatment of alfalfa seeds prior to sprouting reduced *E. coli* loads by 90% after 5 min using aqueous ClO_2 and 50% after 3 min using ozonated water (Singh et al., 2003), by 2 $\log_{10}$ CFU/g after 3 min using acidified ClO_2, and to under the detection limit of 0.3 $\log_{10}$ CFU/g using 1% H_2O_2 (Taormina & Beuchart, 1999). Besides *Escherichia*, *Salmonella* populations have also been linked to outbreaks of human illness associated with seed sprouts. Results have indicated a reduction of *Salmonella* populations of approximately 3 $\log_{10}$ CFU/g achieved using several aqueous chemical treatments, such as 8% H_2O_2, 1% calcium hydroxide, and 1% calcinated calcium (Weissinger & Beuchart, 2000). Other disinfection treatments for reducing the counts of aerobic microbes on rice seeds prior to sprouting include applying hydrogen peroxide, which produced a 2–3 $\log_{10}$ CFU/g reduction in load, and soaking in a sodium hypochlorite, which produced a load reduction of up to 5 $\log_{10}$ CFU/g. Methyl and ethyl alcohol (70–75%), as liquid or vapour, have also been suggested for disinfecting spores in spices, wheat, and vegetable seeds, but their use is prohibited due to residues and the potential to inhibit germination (Piernas & Guiraud, 1997)

The literature review found no published research describing a method able to remove pathogens from seeds effectively enough to ensure complete removal in a commercial facility.

Even if pathogen loads on seeds are reduced to low levels, the sprouting process itself allows pathogens to multiply to dangerous levels (Montville & Schaffner, 2004). Even the different surface structures of different seed types affect the efficacy of seed sanitation, smooth seeds being easier to clean than wrinkled ones due to less shelter in the surface structure (Charkowski et al., 2001). For rough material, gaseous rather than liquid treatments are preferred, as gases have a greater ability to penetrate into pores and voids (Han et al., 2001).

2.4.5 Microwave irradiation

Microwaves are a portion of the electromagnetic spectrum with wavelengths ranging from 1 mm to 1 m, characterized by frequencies of 300 MHz to 300 GHz (Fig. 2.14). For common household applications and industrial purposes, only 2.45 GHz is permitted. Microwave irradiative energy has been used in food processing applications mainly due to its ability to cause fast internal heating. Microwave energy itself is not thermal; rather, heating is a consequence of the interaction between microwave energy and the dielectric food material, mainly affected by water. It can be said that the material itself converts the microwave energy into heat and thus heats itself (Singh & Heldman, 2001). Food properties, such as moisture content, density, temperature, and dielectric properties, determine whether a material can be successfully heated by a microwave field and determine the ability of the dielectric food material to store electrical energy.

Few studies have examined the use of microwaves to decontaminate spices or food powders. In general, a minimum water content of 10%, preferably equally distributed in the food powder, is required to treat a powdered food using microwaves (Modlich & Weber, 1993). In application, however, microwave heating has shown itself to be rather non-uniform in its results, due to the low water content of powders and the non-homogeneity of food materials. Microwaves could not adequately decontaminate spices in terms of reducing bacterial load. A microbial load reduction of only 2 $\log_{10}$ CFU/g can be realistically achieved, which is insufficient for highly contaminated spices (Vajdi & Pereira, 1973; Modlich & Weber, 1993). However, some moulds and yeasts could be reduced to values below 2 $\log_{10}$ CFU/g, even in spices that were not moist (Gerhardt & Romer, 1985). Heating with microwaves can even induce spore germination in inadequately heated food, resulting in increased microbial content (Rosenberg & Bögl, 1982).

2.4.6 Steam and dry heating

There are several different industrial steam treatments on the market, which are used to decontaminate herbs or spices by means of either the high-temperature short-time (HTST) or low-temperature long-time (LTLT) principle. Steam treatment is most recommended for unground spices, as is can protect sensitive sensory components better than other methods (Modlich & Weber, 1993).

The classical approach to overcoming or at least minimizing undesirable quality changes occurring during thermal processing is to use HTST treatment (Fig. 2.15). The inactivation of bacterial spores having a *z*-value of 10°C is generally the benchmark for the duration of sterilization. Differences in temperature dependence mean that if a given product is sterilized at two different temperatures, but to the same bacteriological inactivation level, the negative effects on product quality will be less pronounced in the product sterilized at the higher temperature (Ohlsson, 1980). The different *Z*-values of different micro-organisms and product ingredients mean that with treatment at higher temperatures for shorter times, a region exists (the shaded area in Fig. 2.15) in which pathogens can be destroyed while the vitamin content, for example, can be maintained (Goff, 1995).

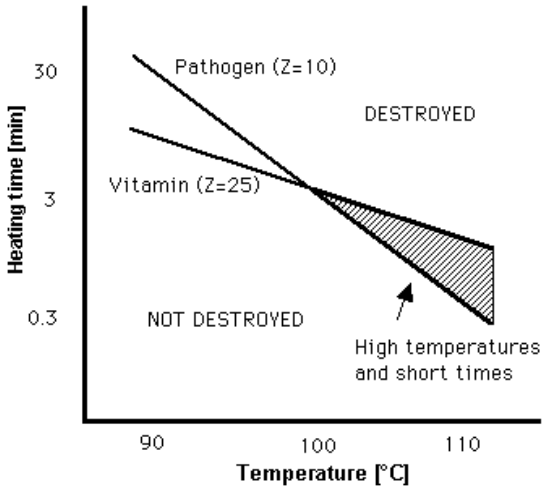

Figure 2.15: Relative changes in time–temperature profiles for the destruction of micro-organisms. Above and to the right/below and to the left of each line micro-organisms and quality factors would be/not be destroyed (Goff, 1995).

High temperature rapidly inactivates micro-organisms and enzymes – the goals of pasteurization or sterilization – and short times will produce fewer undesirable quality changes. For example, an increase of inactivation temperature of 20°C results in an increase

of product degradation by a factor of 4–6, while the inactivation rate for micro-organisms increases by a factor of 100 (Völker et al., 1998). The problem in applying this principle to solid foods is that parts of the food in contact with the hot surface will become overheated in the time needed for the heat to transfer to the interior or coldest part of the food. In severe cases, this surface overheating will produce quality losses due to the low heat diffusivity of foods (Ohlsson, 1994).

Using HTST, overpressure is required to obtain treatment temperatures of 120°C and above with treatment durations of seconds to a few minutes. This method is favourable for treating spices as it maintains quality better than other methods, but cooling or drying afterwards is strongly recommended. Bueltermann (1997) treated black pepper with a hot stream of nitrogen gas at a temperature of 560°C for 2 s; due to the short treatment time, the colour of the pepper grains did not change significantly, and water and volatile oil evaporation from the surface was minimal. An industrial application for sanitizing spices with steam is described by Schneider (1993), as follows: spices are pre-heated to 50–55°C, then autoclaved by steam injection at 120°C for 20–60 s, followed by a fast cool-down and final drying. A final microbial reduction of 3 $\log_{10}$ CFU/g could be obtained, while for paprika the decrease was 2.6 $\log_{10}$ CFU/g and colour changes were unavoidable.

Using LTLT, temperatures below 100°C are applied for durations of hours up to days, the main focus being to maintain sensory quality. In general, microbial reduction rates of only 1–3 $\log_{10}$ CFU/g are commonly achieved, so this method is not always recommended for highly contaminated spices. Degradation or loss of heat-sensitive substances, such as sugars, aroma components, and colour components, is unavoidable; however, the pungency of spices seems not to be significantly affected (Modlich & Weber, 1993). Dry-heat LTLT can be used, for example, in drying egg whites to keep their functional properties, such as gel formation, water-holding capacity, and whipping capacity, together with their flavour and emulsifying ability (Baron et al., 2003). Dry heat can also be used to inactivate eight strains of *Salmonella* spp. inoculated in corn flour; after 24 h of treatment at 49°C, the cell population was effectively reduced from 5 to 2 $\log_{10}$ CFU/g, while a moisture level of 15% was slightly more effective than 10% (Cauwenberge et al., 1981).

2.5 Infrared radiation

Infrared radiation (IR) is that part of the electromagnetic spectrum with wavelengths between those of ultraviolet and of microwave radiation, ranging between 0.76 μm and 1 mm. IR is distinguished as near- (0.76–2 μm), medium- (2–4 μm), and far-IR (4–1000 μm); IR produces the greatest heat transfer effect of all portions of the electromagnetic spectrum and is emitted by all warm bodies (Molin, 1976).

When penetrating the food, the IR light hits a molecule causing changes in the vibration and rotation of the molecules. When the state of the molecule returns to normal, the absorbed energy is transformed into heat. In food materials, water is the substance most affected by IR radiation, but also other biopolymers, such as proteins, lipids, and carbohydrates. Water is a triatomic molecule, and there are three vibration modes for the –OH bonds, namely, symmetric stretching, asymmetric stretching, and deformation vibrations. Other polar groups affected by IR are –NH, –CO, -OH, and C=C. The main approximated absorption range for common food components are expressed in Fig. 2.16. Water is predominant over a wide range of wavelengths. Selective heating is possible (by eliminating water from the food) using the distinct absorptivity of a target food ingredient.

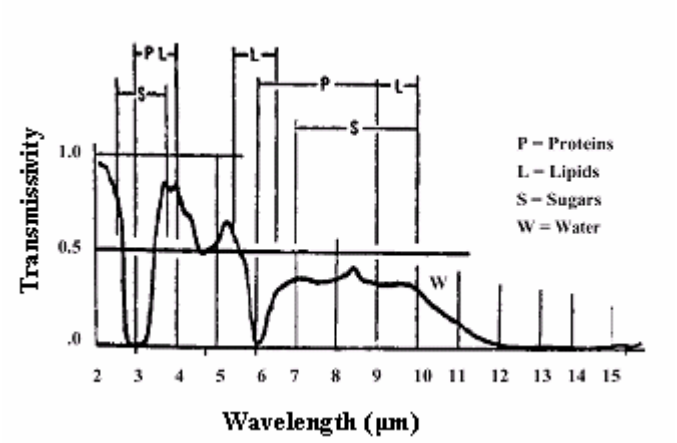

Figure 2.16: Principal absorption bands of the main food components compared with water (Sandu, 1986).

2.5.1 Optical properties

The IR absorption properties (i.e. optical properties) of a foodstuff are difficult to describe but important to know. IR energy emitted from the IR heat source, or radiator, usually passes through air, which is largely transparent to IR, and is absorbed by the food material; there, the energy is converted into heat by interacting (e.g. by reflection, absorption, transmission, and scattering) with molecules in the food (Fig. 2.17). The sum of all interactions is 1. The dissipation of radiative energy, i.e. heat absorption, results in product-specific surface temperatures and penetration depths, depending on the IR wavelength, food composition, a_w, and product thickness.

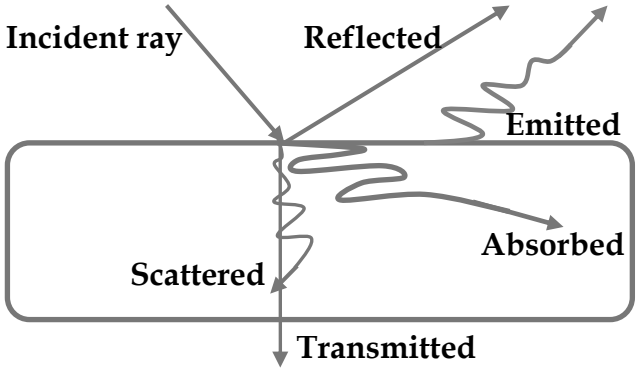

Figure 2.17: Incident radiation on a surface.

When IR meets a food material, reflection takes place on the surface. Surface reflection produces the visible impression (colour) and generally accounts for approximately 50% of the incoming energy at IR wavelengths <1.25 μm and for less than 10% at greater wavelengths (Skjöldebrand & Andersson, 1987). There are two types of reflectance, regular and diffuse. Regular reflection refers to the energy reflected from the "outside" of the surface, without any penetration, producing only the gloss or shine of polished surfaces. Such reflection accounts for only approximately 4% of the incoming energy in the case of most organic materials and is thus negligible in foods; it accounts for more than 90%, however, in the case of metals such as gold or aluminium. Diffuse reflection refers to the reflected energy that escapes from the surface of a sample after having penetrated the sample. The amount of energy entering and leaving the sample is affected by the particle size and shape, and hence by the bulk density and packing performance of a powdered material (Ginzburg, 1969; Williams, 1987).

When penetrating a material, some of the radiation is widely absorbed over the outer surface, expressing itself as the surface temperature. When the absorptivity of the surface layer of the food is high, its transmissivity is low, and vice versa. To ensure intensive heating of the food powder, it is desirable to have surface layers of good transmissivity, i.e. to have the zone of maximum temperature substantially deep inside the material. Scattering and transmissivity are dependent on the wavelength, product layer thickness, and moisture content; transmissivity thus increases with decreased layer thickness, lowered moisture content, and shorter IR wavelength. The total reflectivity of a material decreases with decreasing moisture content, i.e. absorption increases. Transmission energy can even be dissipated within a powder, due to scattering and absorption, which can be important in the case of thicker samples. The transmissivity of water peaks at 0.8–1 μm and decreases to zero at 1.3–1.4 μm, at which point the absorption reaches a maximum (Ginzburg, 1969). However, IR heating is limited by the penetration depth, which is the thickness corresponding to a transmission of 37% – i.e. when 63% of the radiative energy is absorbed by the food material. The IR penetration depth is expected to decrease with increased a_w. Dry powdered products, such as flour or salt, have a penetration depth of 2 mm compared with a penetration depth of 1 mm in a slurry product, such as tomato paste. The approximate penetration depths of selected materials are given in Table 2.6. Portions of the material deeper than the penetration depth are heated mostly by heat conduction. The relationship between IR wavelength, penetration depth, and surface temperature is as follows: the shorter the wavelength, the greater the penetration depth and surface temperature (Ginzburg, 1969).

Table 2.6: Approximate depth of penetration of near IR into various materials (Ginzburg, 1969)

Material	Penetration depth	λ_{max} (μm)
Grain, cellulose	a few mm	~ 1
Grains of wheat	2 mm	~ 1
Flour, salt	2 mm	~ 1
Raw potato	6 mm	~ 1
Dry potato	15–18 mm	~ 0.88
Tomato paste	1 mm	~ 1
Bread, rye	7 mm	~ 0.88–1
Dough, wheat	4–6 mm	~ 1
Carrots	1.5 mm	-
Apples	4.1 mm	1.16
Quartz sand	5 mm	1
Agar	15 mm	0.88
Ice (dist. water)	30 mm	0.88
Human skin	10 mm	1

Ginzburg (1969) reviewed data from other Soviet researchers who studied drying or thermal treatments of different powdered or granular foods, for example, the drying of flour, roots, and seeds and the roasting of cocoa beans and nut kernels. The smallest consumption of energy for drying flour was obtained with a 10-mm-thick flour layer. Flour has high reflectivity, due to its colour, and a relatively low transmissivity, due to the scattering effect occurring within the flour bed. For example, a finely ground and moist mixture of wheat and potato flour reflects 54% of incident IR radiation; the same, but dried, reflects 57% of the incident radiation (Ginzburg, 1969). For materials with rough surface, such as powders, both regular and body reflection can be observed. For instance, at near-IR of λ <1.25 μm approximately 50% of the radiation is reflected back, while less than 10% radiation is reflected back at the far-IR region (Skjöldebrand, 2001).

2.5.1.1 Measuring optical properties

Optical properties are usually measured using thermal sensors. One of the most common sensors is a pyroelectric detector made of lithium tantalum ($LiTaO_3$). To measure *transmittance*, a pyroelectric detector with a small temperature-sensitive area (1 mm^2) was located close (i.e. 2 mm) to the back of a much larger sample (15 mm deep). The food sample was put on a calcium fluorite glass during the measurements. The radiation from the IR source was pulsed at 90 Hz using a rotation chopper, to eliminate the influence of background radiation. The electrical signal from the detector was read with an oscilloscope (Dagerskog, 1979a).

Absorption refers to the temperature variation inside the product caused by radiant energy variations and is most frequently measured using a bolometer or a thermocouple. In the bolometer, the temperature rise due to radiation causes a change in electrical resistance, which is used to vary a voltage. The thermocouple uses the radiant energy to heat two metal junctions and set up an electromotive force between the junctions, the voltage of which is directly proportional to the amount of radiant energy. A pneumatic detector consists of a gas-filled chamber that undergoes a pressure rise when heated by radiant energy. Small pressure variations cause the deflection of one wall of the chamber; this moveable wall also functions as a mirror and reflects a light beam directed upon it to a photocell (Cross, 1960).

Reflection of the various food samples was recorded using a reflectance spectrophotometer, a monochromator, and an integrating sphere reflectance attachment. In this method, the

monochromatic light is diffusely reflected to the detector, due to reflection, refraction, and diffraction on the particles and droplet surfaces inside the sample. The equipment contains filter wheels, tilted filters or grating monochromators, and lead sulphide detectors. Diffuse reflectance instruments are often equipped with grating monochromators and silicon detectors (Dagerskog, 1979a; Isaksson, 1990).

2.5.1.2 Types of IR emitters

In practical application, two methods of IR heating are used, either electrical or gas. In the latter method, IR emission is due to the radiative energy of the flame of the burnt gas. Electrical IR generators include (1) reflector-type IR incandescent lamps, (2) radiators having quartz tubes, or (3) metallic or ceramic tubes containing resistance elements. Electrical IR-radiation generators are characterized by input voltage and power, working temperature, radiation density, construction, shape, dimensions, etc. (Ginzburg, 1969).

(1) Reflector-type IR incandescent lamps are parabolic in shape, the source of radiation being a spiral tungsten filament (Fig. 2.18). The inside surface is covered with a thin layer of silver that acts as a mirror reflector, directing the radiant stream onto the irradiated object. The lamp bulb is filled with a mixture of argon and nitrogen. The wavelength of maximal radiation of the lamp is between 1.05 and 2.25 μm; radiation of $\lambda > 2.6$ μm is partly absorbed by the lamp bulb, which leads to increased temperature (over 70°C) and a corresponding shortening of service life, which is usually approximately 2000 h. Only 2% of the input energy is transformed into light, approximately 65–70% into IR energy (up to $\lambda = 2.6$), while 33–28% comprises absorption inside the bulb and heat losses into the surrounding medium. A maximum heat density of 0.2–2 W/cm^2 can be obtained at distances of 400–150 mm.

To optimize a drying process, for example, so-called coiled-coil filament lamps are often used, containing two helical filaments with a special control. The control permits one to switch on either filament or both filaments at the same time, different degrees of lamp power being combined in one lamp. The reflectors of these lamps are often coated with a thin layer of gold.

(2) To produce a high-efficiency IR generator, quartz glass must be used to sheath the radiator. Quartz glass has a high thermal stability of over 1000°C and good transmissivity over the IR range up to 4 μm. Due to these properties, relatively small generators (tube diameter ~10 mm) can be produced with a maximum heat flux of 6.2–10 W/cm^2. A spiral of

chrome–nickel wire (nichrome, brand name “Kanthal”) is wound on to a quartz tube of small diameter, within a sheath tube approximately 20 mm in diameter; the length of the radiator can be up to 2 m and its power up to 7500 W.

Another material used is a coiled tungsten filament mounted inside a protective hermetically sealed tube containing a vacuum (Fig. 2.19); this arrangement permits heating up to 2000–3000°C. The delay from switching on the lamp until the rated energy stream is obtained is 0.6 s. To ensure the continuous removal of tungsten deposit from the inside tube surface and its return transfer onto the spiral, modern quartz radiators use the regenerative iodine cycle. These lamps should only be used horizontally, as having the radiator in a vertical position would affect the iodine cycle by increasing the iodine concentration in the lower part of the tube. When this cycle is not working properly, the service life of the lamp is shortened, and light stream decreases in intensity.

(3) These types of radiation generators utilize a filament or spiral of a certain resistance, pressed into filler, which is located inside a jacket. The spiral is usually made of nichrome having substantial heat stability up to 1100°C and anticorrosive properties. The filler consists of a fireproof material, usually aluminium or magnesium oxide, allowing for a highly conductive heat transfer from the spiral to the jacket. Various materials and structures are used in the manufacture of the jackets, which may be metallic tubes of circular or oval cross section, ceramic tubes, or panels. The time until working temperature is reached is considerable, and can be up to 4–5 min.

To concentrate the IR energy, the heater is fitted with a reflector, parabolic in shape and made of pure polished aluminium or an aluminium–magnesium alloy. It is important that the reflective surface remain clear during operation, as otherwise the coefficient of reflection (0.90–0.95) may be greatly reduced.

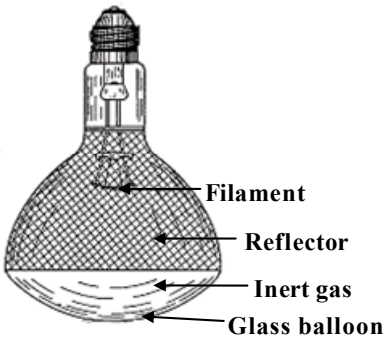

Figure 2.18: Schematic of a reflector-type IR incandescent lamp.

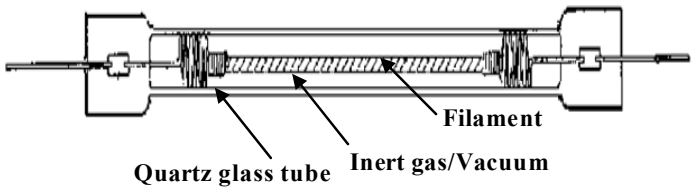

Figure 2.19: Schematic of an IR quartz tube emitter.

2.5.2 Processing advantages

Compared with conventional heating techniques in which the heat is transferred by conduction or convection, IR heating offers several processing advantages (Molin, 1976; Dagerskog, 1979a; Sakai & Hanzawa, 1994; Ranjan et al., 2002):

- Direct and efficient heat transfer to the food
- No heating of the ambient air
- Reduced processing time and energy costs
- Specific heat penetration into the product
- High heating power per unit of surface area, i.e. high heat fluxes possible
- Easy control of the energy flow by continuous regulation of the electrical power, i.e. quick thermal response
- Possibility of designing compact heating units with automatic features, offering high controllability and safety
- Decreased flavour loss and better preservation of vitamins
- Absence of solute migration from the inner to the outer regions
- Surface colour development

If these advantages are to be obtained, exact control of IR emission is required, due to the danger of overheating owing to the rapid heating rates experienced during IR treatment.

Near-IR is generally considered to be the most suitable wavelength for industrial heating applications due to the higher temperatures produced. This radiation is transmitted through most gases, the exceptions being carbon dioxide and water vapour. Certain solid materials, such as paper, glass, and plastics, are somewhat transparent to near-IR. When such a material is irradiated, direct heat transfer into the interior of the material occurs. Metals such as gold, copper, and aluminium provide high reflectivity (Molin, 1976).

2.5.3 Effect on food quality

IR is widely used in non-food industries, such as the automotive, electronics, and paper industries, for heating and drying purposes. In industrial food processing, IR heating is used for baking (roasting), drying, thawing, frying, and surface pasteurization (Fig. 2.20) (Sakai & Hanzawa, 1994; Ranjan et al., 2002). No food is a perfect black body; foods are usually assumed to be grey bodies. IR is located between the wavelengths of UV light and microwaves, which, compared with IR, have very low and high penetration depths, respectively, into food. Thus, surface pasteurization could be a challenge, due to the rapid heating of the surface and limited penetration into the food. However, the thermal conductivity of the food can be a limiting factor regarding heating with IR (Hamanaka et al., 2000).

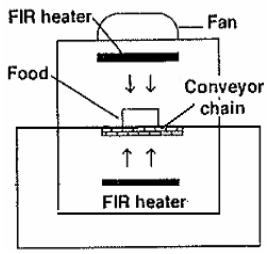

Figure 2.20: Schematic of a far-IR oven (Sakai & Hanzawa, 1994).

At the Swedish Institute for Food and Biotechnology (SIK), the first experiments using IR heating were done to examine the frying of meat or fish products (Fig. 2.21) (Dagerskog, 1979a,b); later experiments compared bread baking in conventional and IR ovens (Fig. 2.22; Skjöldebrand & Andersson, 1987) and explored the effects of a combination of impingement and IR on baking and colour development in bread (Olsson, 2005). For all tested foods, processing times could be reduced greatly by using IR, and fast crust formation had a positive effect on product quality by retaining a higher a_w in the product.

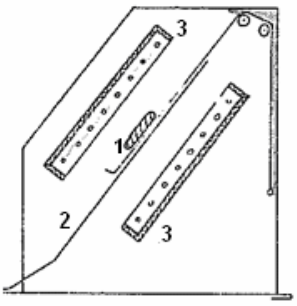

Figure 2.21: Schematic of meat frying using double-sided IR heating: (1) meat, (2) inclined stainless steel net, so drip losses do not fall on the (3) IR radiators (Dagerskog, 1979b).

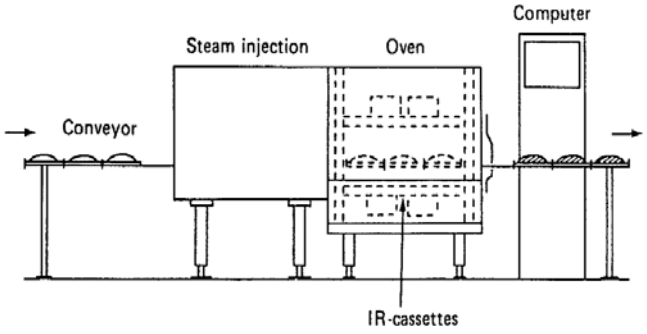

Figure 2.22: Schematic of semi-continuous bread baking using an IR heating oven (Skjöldebrand & Andersson, 1987).

Infrared heating causes thermal shock to the micro-organisms, due to the fast temperature increase, which micro-organisms cannot withstand (Kabelitz, 2007). In particular, the characteristics of IR absorption by water molecules inside the micro-organisms could be the most important factor in the microbiocidal process, since IR is easily absorbed by water. Some research has successfully used IR to inactivate micro-organisms on the surfaces of, for example, cottage cheese (Rosenthal et al., 1996), corn meal (Jun & Irudayaraj, 2003), and grains (Hamanaka et al., 2006b). Figs. 23–26 present different assemblies of IR equipment for drying and/or decontaminating powdered or granular food material.

Near-IR (950 nm) heaters displayed higher inactivation efficacy against the bacterial spores of *Bacillus* than did other IR heaters. The *D*-value was also affected by the initial a_w value, spores with a_w values of approximately 0.9, 0.7, and 0.6 being most resistant to near-IR heating at wavelengths of 950, 1100, and 1150 nm, respectively (Hamanaka et al., 2006a). Hamanaka et al. (2006b) developed a rotating grain sterilizer (Fig. 2.23) in which the heat resistance of spores was found to be markedly affected by the initial a_w, which had to be 0.93, corresponding to a moisture content of 24% (w.b.). Repeating the sterilization process three times induced an additional microbial effect; however, due to a high lethal temperature of over 100°C, the germination rate was markedly reduced.

Hamanaka et al. (2000, 2001) also applied far-IR (Fig. 2.24) to wheat and soy beans to lower bacterial counts and eliminate *Aspergillus niger* moulds. A decrease of 1 $\log_{10}$ CFU/g in the bacterial count was achieved after 60 s of treatment using 2 kW, while *A. niger* required 20 s at a power of 1 kW. Higher irradiation power produced a faster decrease in the microbial load for the same specie. For soybeans, a surface temperature of 100°C was measured after

just 7.2 s of irradiation at a power of 2 kW. Differences in temperature increase are related to different degrees of IR absorption depending on the surface reflection. IR heating inside a wheat bulk occurred due to conduction, and discoloration was confirmed after 50 s. The water content during IR heating remained constant for up to 60 s at powers of 0.5–1 kW but decreased significantly at powers of 1.5–2 kW when 30–40 s had passed; this caused internal enzyme degradation due to the rapidly reduced water content. In order not to overheat the surface during drying, IR treatment was done for 20–40 s and then stopped for four hours, to let the water redistribute itself throughout the sample.

Herbs were dried using near-IR (Fig. 2.25) in a gentle temperature range of 35–50°C, which resulted in a shorter processing time than that obtained with air drying. Up to 75% of the water content was removed without loss of volatiles while retaining natural colour. Low-temperature drying with IR radiation required a thin bed of herbs and a large drying area to achieve dried material of the desired quality. Except for reduced coliform counts, the microbial quality was not improved, due to low process temperature (Pääkkönen et al., 1999).

When drying diced potatoes, moisture was lost only from the top, causing a negligible total loss of moisture from the pieces. It was noted that, by applying the highest power level of 11 kW/m^2, the surface became burnt after 5 min of heating. This method could thus be used for rapid surface heating to achieve colorization or when moisture has to be sealed inside the food system, such as during frying (Ranjan et al., 2002).

Jun and Irudayaraj (2003, 2004) stated that far-IR has a pronounced effect on the inactivation rate, due to the absorptivity of proteins in that range. A selective far-IR system, using an optical 5.45–12.33-μm bandpass filter (Fig. 2.26), was developed to heat food components based on their different IR absorptions. Spores of *A. niger* and *Fusarium proliferatum* were inactivated due to selective protein denaturation in 5.88–6.66 μm range. The absorbed heat flux for *selective* far-IR heating was greater than for normal far-IR heating. After 5 min of heating, a 2 $\log_{10}$ CFU/g reduction was accomplished with selective far-IR heating, which was 40% more lethal to fungal spores than was normal for far-IR heating after the same treatment time.

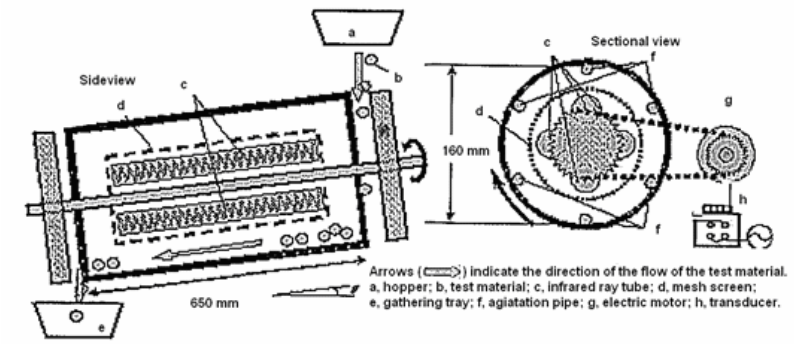

Figure 2.23: Schematic of the rotary drum grain sterilizer using IR heating (Hamanaka et al., 2006b).

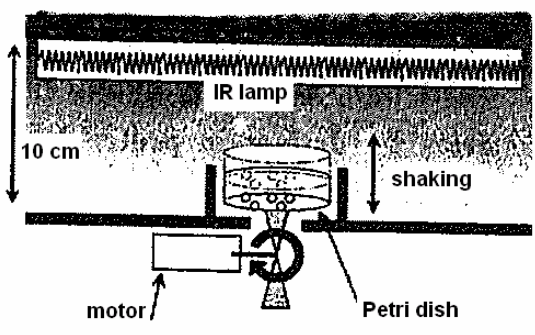

Figure 2.24: Experimental set-up for inactivating bacterial spores in wheat flour (Hamanaka et al., 2000).

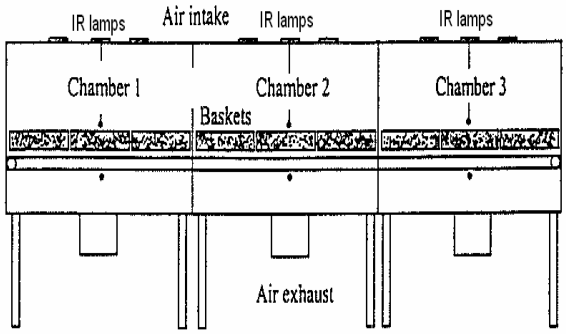

Figure 2.25: Schematic of IR heating set-up for drying herbs (Pääkkönen et al., 1999).

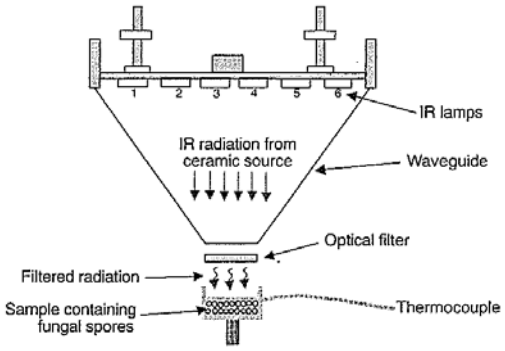

Figure 2.26: Schematic of set-up for selective IR heating of corn meal to inactivate fungal spores (Jun & Irudayaraj, 2003).

3 Materials and methodological considerations

This thesis focused on developing a decontamination system for paprika powder using IR heating. The research included the development of techniques for adjusting the product properties of paprika powder to allow the successful decontamination of *B. cereus* spores and the evaluation of the properties of the available IR heating equipment. Problems and phenomena occurring during IR heat treatment were identified and analysed, and solutions were sought by further enhancing the powder sample holder and IR heating equipment.

3.1 Paprika powder

Paprika powder (*Capsicum annuum*) imported from Israel and supplied by Nordfalks Industri AB (Mölndal, Sweden) was stored at 19°C and 51% relative humidity. The moisture content of the powder was 8.5 ± 0.5% dry base (d.b.) and the a_w was 0.50 ± 0.03.

3.1.1 Adjusting the a_w value

Paprika powder was wetted to different a_w values. Up to a_w 0.88, the powder was considered wetted powder, whereas powders with higher a_w value had the consistence of slurry.

For an a_w value of 0.50, the original paprika sample was used without adding water. Wetting the powder up to a_w 0.88 was done using an adapted version of the methods described by Coşkun et al. (2005) and Muramatsu et al. (2005), as follows. Water was spayed on thin layers of powder, which were then stored for 2–3 d in a refrigerator (8°C) to enable the moisture to distribute itself uniformly throughout the sample. Before using the wetted partly agglomerated powder, it was sieved again to maintain its status as powder. Samples with an a_w value of 0.96 were prepared by adding water to the paprika powder in a ratio of 1.7:1 while stirring the mixture, until a homogeneous slurry was achieved.

3.1.2 Adjusting the pH value

Paprika powder wetted with water had a pH of 4.5; to obtain a pH of 4.0, 1 N citric acid (Merck, Darmstadt, Germany) was added to the water before the wetting procedure.

3.1.3 Particle size

Particle size was analyzed by sieving 100 g of paprika powder, after which the particle fractions obtained were measured using a laboratory plansifter fitted with six square-mesh sieves of mesh sizes of 40, 125, 500, 710, 1000, and 2800 μm. The method was adapted from Chapelle et al. (1989).

3.1.4 Mixing with high-water foods

Crème fraîche and raw pork tenderloin are examples for high-water foods commonly used in combination with paprika powder, for example, paprika-spiced crème fraîche and paprika-marinated meat. Due to possible high microbial contamination, mixing paprika powder with crème fraîche and pork can significantly reduce their shelf life.

3.1.4.1 Crème fraîche

Crème fraîche (27% fat, dry matter) was bought at a local supermarket. Crème fraîche (30 g) and paprika powder (10 g), i.e. a 4:1 ratio, were put in a sterile glass and mixed until homogeneous using a sterile spatula (Fig. 3.1).

3.1.4.2 Meat

Vacuum-packaged pork tenderloin (n = 2) was bought at a local supermarket. Before using the meat for experiments, it was frozen for 1 d and then decontaminated on the surface. To reduce the surface contamination, each side of the tenderloin was treated using a near-IR heat flux of 23 kW/m^2 for 2.5 min, resulting in denaturation of the meat surface; the meat was then wrapped in aluminium foil, immediately cooled on ice, and refrozen. The pasteurized, i.e. denatured, meat surface was then removed, while the raw meat from the centre was used for the experiments.

The raw meat (pH 5.4–5.6) was cut into pieces of approximately 3 × 3 × 1 cm (approximately 30 cm^2 surface area, weight 14 ± 1.3 g), which were then dipped in paprika powder (Fig. 3.2), resulting in 1.45 ± 0.18 g of powder covering each meat piece; the pieces were then placed in a sterile Petri dish.

Figure 3.1: Crème fraîche (1), paprika powder of a_w 0.84 (2), and crème fraîche mixed with paprika powder (3).

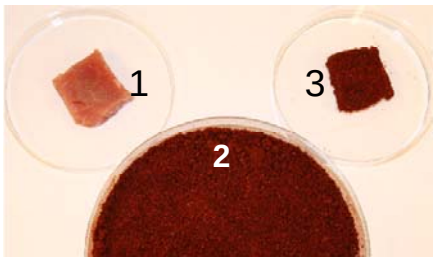

Figure 3.2: Raw pork piece (1), paprika powder of a_w 0.84 (2), and meat dipped in paprika powder (3).

3.2 Spores of *B. cereus*

The psychotropic strain of *B. cereus*, SIK 340, used in the experiments was isolated from a local dairy in 1998 and is maintained in the SIK culture collection. The strain was kept frozen at –20°C until used for this thesis research in 2005–2008.

3.2.1 Production of spores

B. cereus, SIK 340, was cultured overnight in broth containing 15 g/L of tryptone (Bacto), 2.5 g/L of yeast extract, 0.15 g/L of $CaCl_2$, and 0.25 g/L of $MgSO_4$, and then transferred to sporulation agar plates. The sporulation medium contained 8 g/L of Difco nutrient broth (BD Biosciences, Franklin Lakes, NJ, USA), 0.25 g/L of $MgSO_4$, 0.97 g/L of KCl, 0.15 g/L of $CaCl_2$, 2×10^{-3} g/L of $MnCl_2$, 0.3×10^{-3} g/L of $FeSO_4$, and 3% agar (S. Ståhl, Department of Microbiology, University of Lund, Sweden). After incubation for 5–7 d at 30°C, a spore yield of 90–100% was obtained. The spore crop was harvested, washed three times in 0.9% NaCl, and stored at –20°C (Andersson & Rönner, 1998).

3.2.2 Spiking of powder

The inoculum was prepared by thawing 1 mL of spore suspension and diluting it in 99 mL of 0.1% peptone water (1.0 g of bacteriological peptone, 8.5 g NaCl/L of Milli-Q water; Difco, BD Biosciences) to achieve a concentration of approximately 8 $\log_{10}$ spores/mL, after which the mixture was stored for up to 7 d at 4°C. Screening tests indicated that the storage time did not induce germination. Approximately 3 mL of spore solution was added to 27 g of pre-wetted paprika powder by spraying with an atomizer while mixing. The final spore concentration in the paprika powder after spiking was 7.23–7.48 $\log_{10}$ spores/g.

3.2.3 Determination of *D*-values

D-values were determined by means of linear regression on the straight part of the microbial reduction curves obtained when the $\log_{10}$ number of survivors was plotted against holding time. The *D*-values do not represent the absolute *D*-values at the tested product temperatures, due to the temperature gradient within the powder bed during IR heating and while taking microbial samples from the overall powder.

3.3 IR heat treatment

3.3.1 IR treatment chamber

Heating was executed in an IR drying oven (Ircon Drying Systems AB, Vänersborg, Sweden) composed of two independent sections (each 70 × 70 × 50 cm), one operating at near- IR and the other at medium-IR (Fig. 3.3). For near-IR, the radiator was a quartz tube filled with halogen gas and containing a tungsten filament, while for medium-IR, the radiator was a quartz tube filled with air and containing a Kanthal filament; their emission maxima (and corresponding temperatures) were 1.2 μm (2100°C) and 2.7 μm (800°C), respectively.

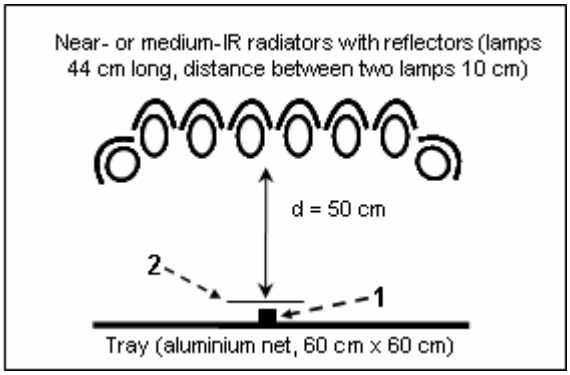

Figure 3.3: Schematic of near- and medium-IR heat chamber, showing position of the black body (1) and IR-transparent material (2).

3.3.2 Determining the IR heat flux

The heat flux emitted by the near- and medium-IR radiators was calculated from the time required for the temperature to increase from 50 to 100°C (ΔT = 50°C) using a black painted reference copper plate (5 × 5 × 0.6 cm) with a thermocouple in the centre. The placement of the black body in the IR chamber is shown in Fig. 3.3. The distance between the radiators and the black body was 50 cm.

To measure the IR heat flux distribution of the heating area, a device consisting of sixteen black bodies was placed on the aluminium tray as shown in Fig. 3.4. The heat flux for each position of the black body was calculated as described above. The heat flux distribution was graphically displayed using Matlab software program (Mathworks, Natick, MA, USA).

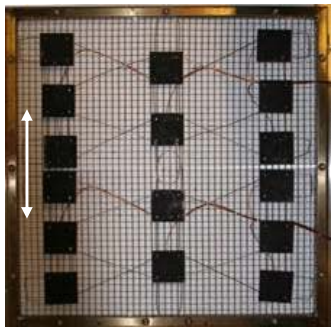

Figure 3.4: Assembly of black bodies used to measure temperature distribution in the IR oven (600 × 600 mm). The arrow indicates the movement of the IR heating area of ±100 mm from the zero position at 300 mm.

For some IR heating applications, IR had to pass through an IR-transparent material of either glass or plastic before coming into contact with the paprika powder. The IR transparency factor of the materials had to be determined by calculating the quotient of the

measured heat flux with and without the material. This was done by placing the material on top of the black body, i.e. between emitter and absorber, during IR heating (see Fig. 3.3). Paprika powder is considered a grey body, as it absorbs less energy than a black body.

3.3.3 Heating of paprika powder

A mass of 30 g of paprika powder (10 mm bed thickness, bulk density of 537 kg/m^3) was placed in the same position as the black body and heated by either near- or medium-IR (see Figs. 3.3 and 3.5 a), or in a boiling water bath as shown in Fig. 3.6.

3.3.4 Temperature profile measurement

During IR heating, the temperature was measured every second at four different locations in the powder bed: directly under the surface and at depths of 1, 3, and 8 mm (Fig. 3.5 a). During heating in a water bath, the temperature was measured at depths of 1, 3, and 8 mm (Fig. 3.6). Type T thermocouples (Pentronics, Sweden), a logger (Intab AB, Sweden), and computer software (Easyview, Sweden) were used to record the temperatures (Fig. 3.5 a). To position the thermocouples at their exact locations in the powder bed, they were fixed on a Teflon device (Fig. 3.5 b and c).

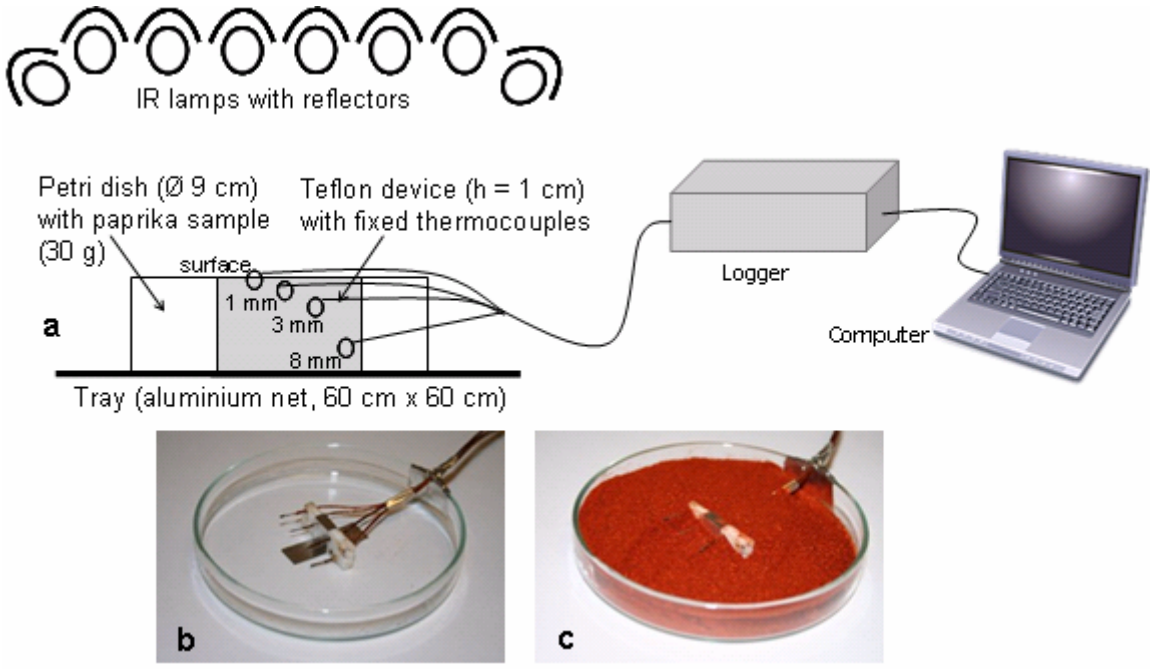

Figure 3.5: Assembly of the general system for heating paprika and measuring the temperature (a); image of Petri dish with thermocouples on the surface and at depths of 1, 3, and 8 mm, fixed on a Teflon device (b) and image of the same dish containing paprika powder and thermocouples (c).

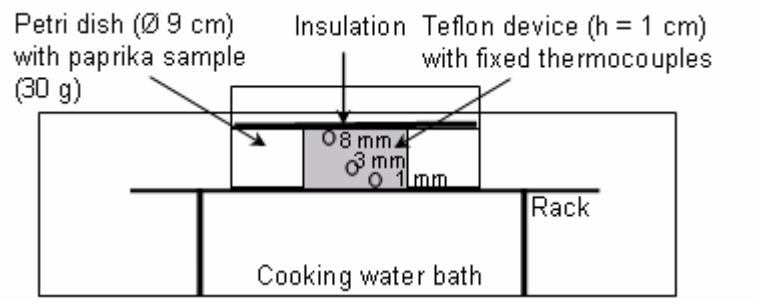

Figure 3.6: Assembly of water bath system for heating paprika powder; positions of fixed thermocouples shown.

3.4 Evaluation of product quality

The quality of paprika powder before and after the IR heat treatments was evaluated in terms of colour, a_w value, water content and total water loss, concentration of *B. cereus* spores, and level of natural background flora.

The effect of storage on decontaminated paprika powder was evaluated in terms of colour, a_w, volatiles, and concentration of *B. cereus* spores. Paprika powder was placed in a sealed plastic pouch (for a description of the plastic material used, see section 5.3.) during IR heat treatment and subsequent storage. Powder was stored at room temperature and each plastic pouch was wrapped in aluminium foil, to eliminate the effect of light on the colour of the stored powder.

The effect of storage in the refrigerator at 7°C on the concentration of *B. cereus* spores was evaluated for paprika powder added to crème fraîche and to the surface of pork tenderloin. In addition, the concentrations of background flora and lactic acid bacteria were determined for paprika added to the surface of pork tenderloin.

Paprika samples were taken from different locations in the powder bed, namely, the *surface* (to a depth of 2 mm), *inside* (from depths of 2–10 mm), and *overall* (mixture of entire sample after IR heating). The overall sample was prepared by mixing the entire powder bed with a spatula until homogeneous.

3.4.1 Colour

Colour measurements were made in triplicate, taking three measurements per trial (n = 9), using a Minolta CR-10 camera (Tokyo, Japan); the L^* and a^* colour parameters were

measured, L^* being the lightness and a^* the red–green chromaticity co-ordinate. The reference white ceramic plate had the co-ordinates $L = 89.4$ and $a = -3.0$.

3.4.2 Water activity and content

Water activity, *water content*, and *total water loss* in paprika powder were measured in triplicate. Water activity was measured by placing 1.5–2 g of powder in an a_w chamber (Aqualab®, Decagon Devices Inc., Pullman, WA, USA). The water content (in 2.5–3 g of powder) was measured using the gravimetric method, i.e. by drying the powder in a vacuum oven until constant weight was reached. The total water loss was measured by weighing the whole sample before (mass of approximately 30 g) and after IR treatment.

3.4.3 pH value

The *pH* was measured by diluting 1 g of powder in 9 mL of distilled water while monitoring the mixture using a pH meter (HI 9318, Hanna Instruments, Woonsocket, RI, USA) with a tip probe electrode.

3.4.4 Microbial concentration

The *microbial quality* was evaluated for paprika powder, crème fraîche spiced with paprika, and pork tenderloin spiked with paprika. The total and spore plate populations were counted and expressed as colony-forming units per gram (CFU/g) for paprika powder and crème fraîche, and per cm^2 (CFU/cm^2) for the surface of pork tenderloin.

After IR decontamination treatment, the spiked powder was put immediately on ice, cooled, and stored on ice until recovery from powder.

Microbiological analysis during storage was conducted for the decontaminated powder after 0, 1, 2, 4, 8, 12, and 16 weeks ($n = 4–6$ per sampling occasion), for pork meat samples after 0, 3, 6, 9, 12, 15, 20, and 30 d ($n = 4$ per sampling occasion), and for crème fraîche samples after 0, 10, 20, 30, 40, and 60 d ($n = 4$ per sampling occasion).

The bacterial concentration was determined by (1) diluting the material in 0.1% peptone water, (2) shaking or vortexing the dilution to recover micro-organisms from the material, (3) followed by plating appropriate tenfold dilutions (0.1% peptone water) on TGE agar plates (24 g of tryptone glucose extract agar/L distilled water; Difco, BD Biosciences, Franklin

Lake, NJ, USA), and (4) incubation for 24 h at 30°C. An appropriate dilution factor and duration and speed of shaking/vortexing were chosen for each material (Table 3.1), which comprised either only paprika powder or paprika powder mixed with high-water foods (crème fraîche and raw pork tenderloin). Vortexing was used for the latter two products, due to the higher forces required to separate powder and bacteria from the high-water product.

Spore counts were made by heating the first dilution of the powder in a water bath for 10 min at 80°C; the samples were then plated and incubated as described above.

The visual appearance of colonies on TGE agar was used to distinguish between *B. cereus*, lactic acid bacteria, and the background flora. Large white colonies were considered to be *B. cereus*, pin-point-sized transparent colonies were considered to be lactic acid bacteria, and the remaining colonies – mostly medium-sized, white–yellow in colour – were considered to be natural background flora.

Table 3.1: Conditions for spore recovery from paprika powder

Sample	Dilution factor of recovery medium	Speed (rpm)		Duration
		Shaker	Vortex	
Paprika powder	1:10, i.e. 1 g of powder in 9 mL of solution	250		20 min
Paprika + crème fraîche	1:5, i.e. 4 g of product in 18 mL of solution		2000	2 min
Paprika + pork	1:1, i.e. 1 cm^2 meat surface in 1 mL of solution		2000	2 min

3.4.5 Volatiles

Paprika powder was wetted to a_w 0.84 and placed in a plastic pouch, which was then sealed and IR heat treated. Volatile compounds were collected from paprika powder before and directly after IR heat treatment and from the same samples after storage for 5 weeks at 20°C without light exposure. Samples were analysed in triplicate.

For the collection of volatiles, each 30 g paprika sample was put into a 500-mL glass flask, which was then sealed using a plastic screw top equipped with a headspace adapter; the flasks were then placed in a climate chamber at 30°C for 30 min to allow the powder sample to equilibrate. Subsequently, 1 L of helium gas was fed through the bottle at a rate of 40 mL/min, and allowed to pass through a Tenax cartridge (Tenax TA 60–80 mesh, 150 mg); where the volatiles were trapped.

Volatiles were identified and quantified using gas chromatography-mass spectrometry (GC-MS). The gas chromatograph used was a ThermoQuest Trace GC 2000 (ThermoQuest–CE Instruments, Milan, Italy) equipped with a 30 m × 0.32 mm capillary column with a 1-µm-

thick film (DB-5MS; J&W Scientific Inc., Folsom, CA, USA). The mass spectrometer used was an Automass Solo (ThermoQuest). The initial temperature of the GC oven was 25°C, which was maintained for 2 min. Subsequently, the temperature was increased by 4°C/min until a final temperature of 220°C was reached, which was then held for 10 min. Helium was used as a carrier gas at a flow rate of 35 mL/min. The integration and identification of the GC peaks was carried out using Xcalibur^{TM} computer software (ThermoQuest). The compounds were identified on the basis of their mass spectra and comparison to the retention times obtained from the chromatographic runs for the corresponding reference chemicals. The compounds were quantified using an external standard and by performing GC-MS on the pure compounds in question.

3.5 Statistical analyses

The detection limit for total plate counts was 1 $\log_{10}$ CFU/g; samples containing below 1 $\log_{10}$ CFU/g were considered to have no detectable organisms. Mean values and their standard deviations (SD) were calculated for colour, a_w value, and microbial numbers and tested for their significant differences ($P < 0.05$) using Microsoft Excel 2003 (Microsoft, Redmond, WA, USA).

Results and discussion

This thesis explored the potential of IR heating as a method for decontaminating food powders. Paprika powder spiked with spores of *B. cereus* was used as the model material.

The studies focused on developing an IR heating system for decontaminating the paprika powder, by inactivating the spores, while maintaining its product quality. The parameters observed to negatively affect the product quality of paprika powder during IR heating were eliminated by improving the heating units.

The results and discussion section is divided into three chapters, **chapters 4–6**, according to their focus on different aspects of the investigation.

In the first part, **chapter 4**, the effects of wetting paprika powder on product quality are analysed, as are the heating characteristics of the IR energy sources.

The second part, **chapter 5**, presents the three IR decontamination prototypes – IR heating units I to III – developed at a laboratory scale. The effects of IR heating on paprika powder are evaluated in terms of (1) product temperature profile, (2) changes in a_w value and colour, (3) reduction of the spore concentration of *B. cereus* and of the natural background flora, and (4) final drying of the wetted decontaminated powder to a_w 0.50.

The third part, **chapter 6**, presents possible food applications of the decontaminated paprika powder, such as (1) storage of intermediate-wetted powder for 4 months, and (2) mixing with high-water foods and subsequent storage at 7°C. The effects of storage are evaluated in terms of colour, a_w, volatiles, and microbial counts of *B. cereus* and the natural background flora.

4 Properties of paprika powder and the IR heating method

4.1 Effect of wetting on paprika powder

4.1.1 Sorption isotherm

Table 4.1 shows the amounts of water to be added to achieve desired a_w values. The sorption isotherm curve obtained for paprika powder displayed typical sigmoid behaviour (Fig. 4.1). Dry paprika powder at a_w 0.50 had a water content of 8.5% (d.b.). Water content values and corresponding a_w values higher than 8.5% were obtained by wetting the powder, while a_w values below 8.5% were obtained from samples taken from the heated surface of the powder mass during IR treatments. A_w values of 0.3–0.6 were relatively constant for corresponding water contents of 8–12%. Moderately wetting the powder from a_w 0.6 to 0.76 resulted in a slight increase of water content to 17% d.b., while adding additional water up to a_w 0.88 resulted in a steeper increase of water content to approximately 30% d.b. At a_w values higher than 0.88, the powder became very sticky and finally a slurry, i.e. it lost the properties of a powder at water contents of 50% and higher.

Table 4.1: Water added to dry paprika powder (8.5 g d.b.) by spraying the powder to achieve the desired a_w

a_w	Added g water/100 g d.b.
0.50	0
0.72	6
0.76	8
0.80	12
0.84	15
0.88	22
0.96	52

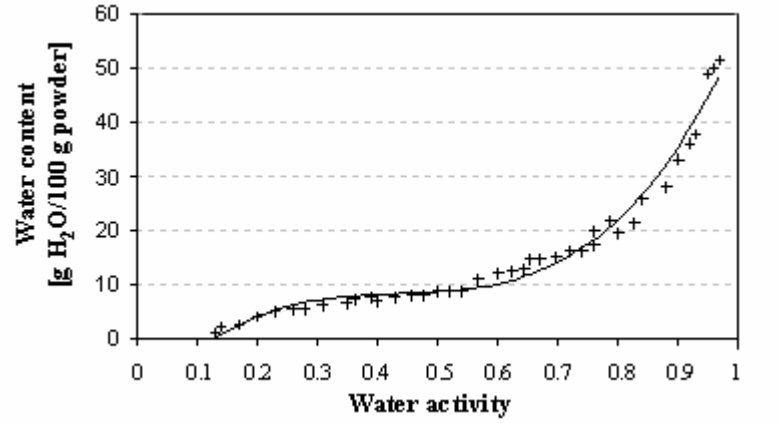

Figure 4.1: Measured water contents and associated a_w values of paprika powder.

4.1.2 Particle size distribution

The effect of wetting on the particle size of paprika powder is depicted in Fig. 4.2. With wetting, the mean particle size increased from 140 μm (range, 40–500 μm), through 300 μm (range, 40–1000 μm), to 600 (range, 125–2800 μm) at a_w values of 0.5, 0.8, and 0.90, respectively, due to particle agglomeration during wetting (Hogekamp & Schubert, 1993). At higher water contents, the amount of small particles (fines) is negligible (Peleg, 1983).

Particle size distribution at a_w values of 0.50, 0.60, and 0.70 increased steeply to 70–90% for particles up to 250 μm in size. Further wetting increased the particle size expressed in a more linear fashion, especially wetting to a_w 0.80 and 0.88. At a_w 0.90, the mean particle size was markedly larger than at 0.88. Thus a_w 0.88 was considered as the limit of paprika powder wetting.

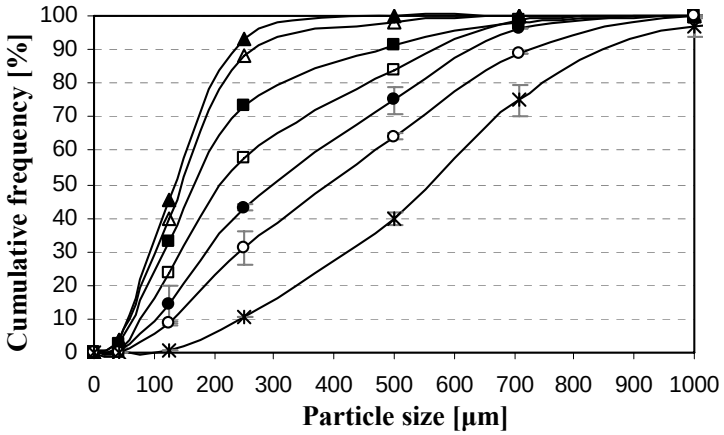

Figure 4.2: Particle size distribution of paprika powder at a_w values of 0.50 (▲), 0.60 (Δ), 0.70 (■), 0.75 (□), 0.80 (●), 0.88 (○), and 0.90 (Ж).

4.1.3 Colour

Besides temperature and the presence of carotenoids, a_w value also affects the colour of paprika powder (Carbonell et al., 1986). Fig. 4.3 shows the initial colour of paprika in relation to the ambient a_w. The colour of paprika powder was expressed as the product of the *L* (lightness) and *a* (redness) colour parameters (i.e. $a \times L$), values >500, 500–300, and <300 being rated as red, medium red, and dark, respectively (Ramakrishnan & Francis, 1973). Fig. 4.4 shows the corresponding visual impression of paprika powder wetted to different a_w values. At all a_w values, the colour was rated as red, but adding water reduced the brightness of paprika powder.

Dry paprika powder (i.e. a_w 0.50) had an initial colour value of 950, and wetting the powder significantly affected its colour. A moderate 10% decline in colour value was observed up to a_w 0.76; at a_w 0.80, wetting reduced the colour value by 15%, while at a_w 0.88 and 0.96 the colour value was reduced by 30% and 42%, respectively. However, according to the above rating, the colour at a_w 0.96 is still considered red ($a \times L = 550$).

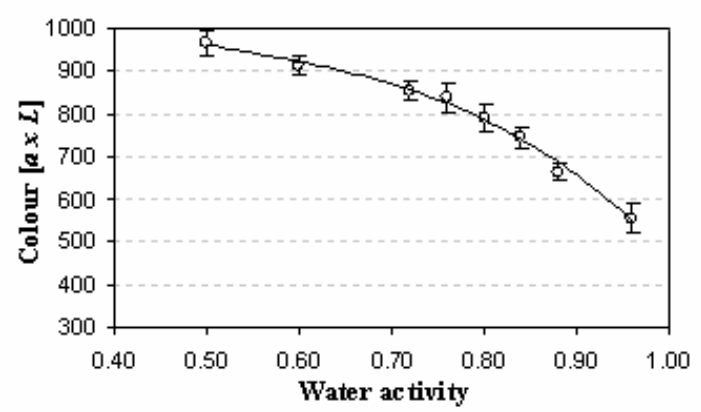

Figure 4.3: Colour value ($a \times L$) in relation to the ambient a_w in paprika powder.

Figure 4.4: Appearance of paprika powder at a_w values of 0.50 (1), 0.60 (2), 0.76 (3), 0.80 (4), 0.84 (5), and 0.96 (6).

4.2 Evaluation of IR energy sources

4.2.1 Measured heat fluxes

Fig. 4.5 shows the measured temperature increase of the black body during exposure to selected near- and medium-IR output power. The respective calculated near- and medium-IR heat fluxes are shown in Fig. 4.6. The higher the output power, the higher the temperature increase, and, consequently, the higher the emitted heat fluxes. There is a linear relationship

between output power and heat flux for near-IR heat fluxes of 5–23 kW/m^2 at 1–100% output power and for medium-IR heat fluxes of 1–11 kW/m^2 at 30–100%.

Considering the same output power, higher heat fluxes were obtained using near-IR radiators. In the 4–11 kW/m^2 range, near-IR and medium-IR produced the same heat fluxes, only differing in output power. This phenomenon is used later to study the effect of wavelength on IR heating. At IR heat fluxes of 5 kW/m^2 and lower, heating resulted in a slow temperature increase of the black body (especially observed for medium-IR at 25% output power) and thus were insufficient for heating. However, such heat fluxes could be used to maintain the desired product temperature after the warm-up period.

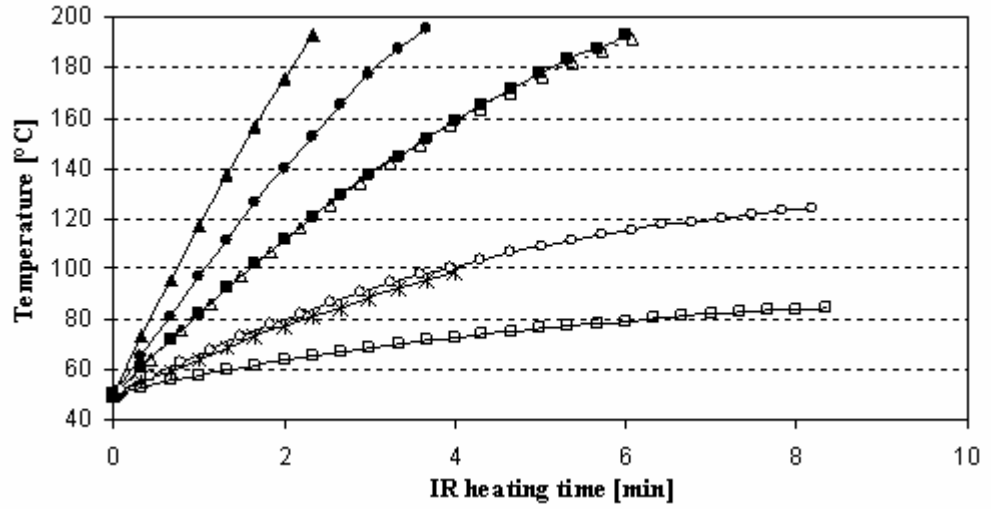

Figure 4.5: Measured temperature of the black body during exposure to near- and medium-IR at output powers of 100% (▲and Δ), 50% (● and ○), and 25% (■ and □, respectively) and to near-IR at 1% (ж), with a distance of 50 cm between the IR energy source and the black body.

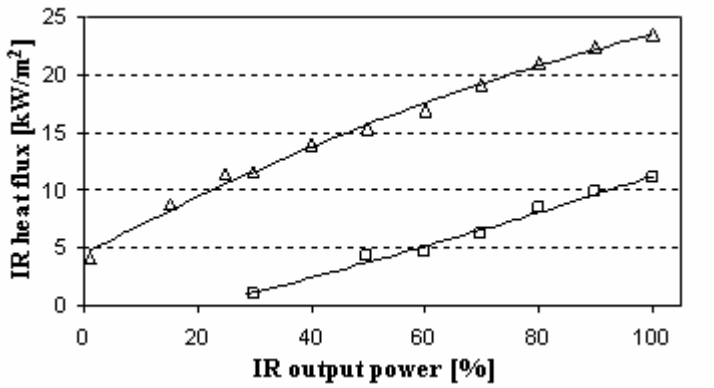

Figure 4.6: Output power versus calculated near- (Δ) and medium-IR heat flux (□), with a distance of 50 cm between the IR energy source and the black body.

4.2.2 Heat pattern in the IR chamber

The heat flux distribution in the IR oven was measured for each of near- and medium-IR at output powers of 50 and 100% (Fig. 4.7). The highest heat flux was measured in the centre of the heating area, with a slight shift from the centre towards the lower edge for medium-IR at a 50% output power. Heat fluxes decreased by 10–15% towards the outer edges of the heating area. The heat pattern was more horizontal for near-IR (Fig. 4.7 a, b), while a more radial pattern was observed for medium-IR (Fig. 4.7 c, d). Due to the non-homogeneous heat flux distribution of the heat pattern, the powder sample was always placed in the same position in the centre of the oven.

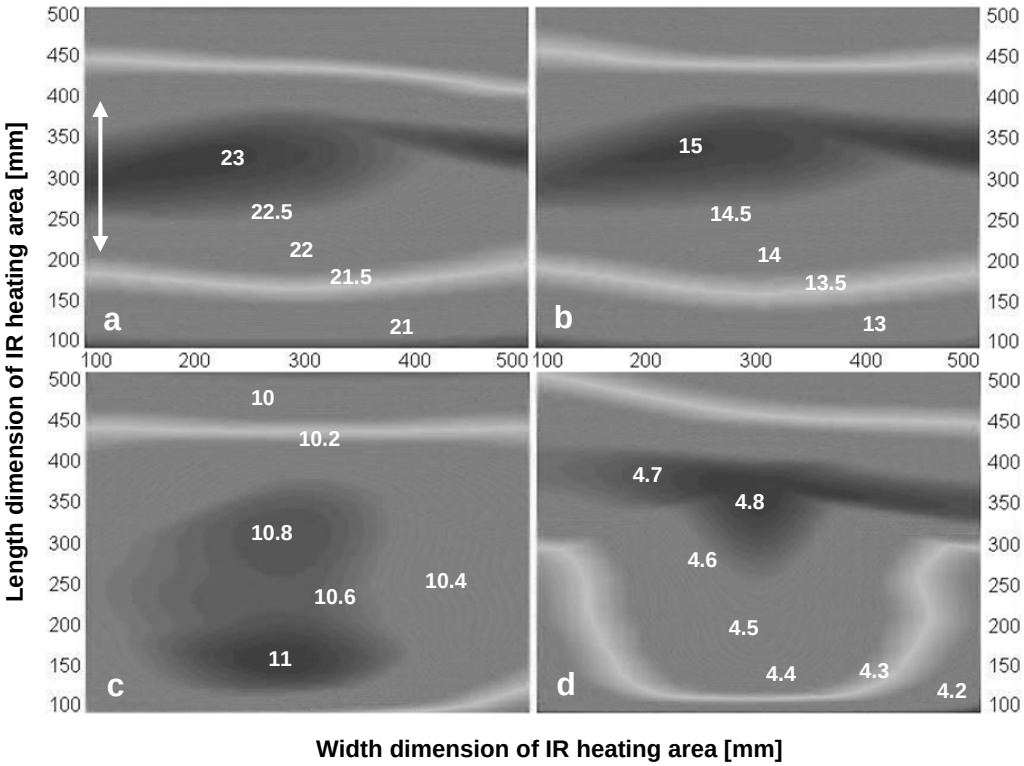

Figure 4.7: Heat flux distribution (kW/m^2) in an IR oven (600 × 600 mm) for near-IR at output powers of 100% (a) and 50% (b) and for medium-IR at output powers of 100% (c) and 50% (d), with a distance of 50 cm between the IR energy source and the black body. The arrow in (a) indicates movement of the IR heating area of ±100 mm from the zero position at 300 mm; IR energy sources were fixed.

5 Process development of IR heating of powders

5.1 Open IR heating unit 1

5.1.1 IR heating system

In industrial food processing, IR heating is used for baking (roasting), drying, thawing, frying, and surface pasteurization (Sakai & Hanzawa, 1994; Ranjan et al., 2002), IR being applied directly to the surface of the target. Heating unit 1 was used to heat paprika and offered no protection against the evaporation of water from the powder; it is therefore called open heating unit 1 (Fig. 5.1).

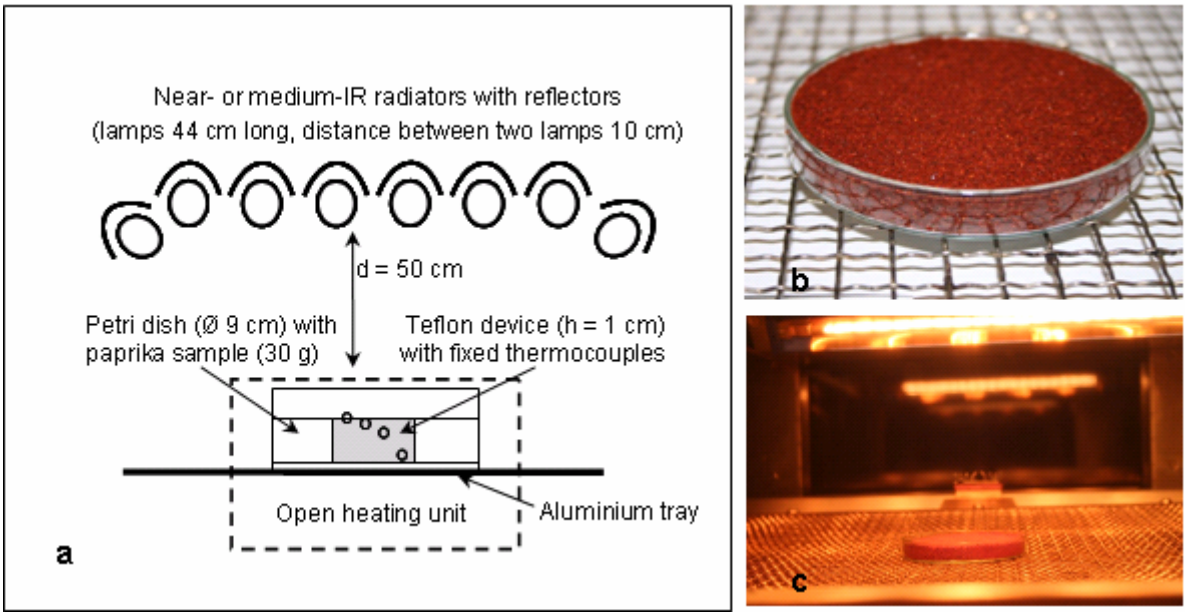

Figure 5.1: Schematic cross-section of open heating unit 1 (separate units for both near- and medium-IR wavelengths); the Teflon device has fixed thermocouples on the surface and at depths of 1, 3, and 8 mm (a). Image of paprika sample placed in open heating unit 1 (b); and side view during near-IR heating (c).

Selected constant heat fluxes of 23 and 11 kW/m^2 (near-IR) and 11 and 5 kW/m^2 (medium-IR) were used to heat paprika powder having a_w values of 0.50 (dry powder), 0.80 (wetted powder), and 0.96 (slurry). The effects of IR heating on product quality were evaluated for colour, a_w and water content, and reduction of the *B. cereus* and microbial loads of the natural background flora.

Furthermore, heating times and temperature profiles were compared for paprika powder at a_w values of 0.5 and 0.8 placed in the water bath and during exposure to a medium-IR heat flux of 5 kW/m^2, as well as during heating of paprika (a_w 0.96) and pure water using selective near- and medium-IR heat fluxes.

5.1.2 Heating of paprika powder in water bath and by IR

The heating of dry (a_w 0.50) and wetted powder (a_w 0.80) to 95°C in a 10-mm-deep powder bed using medium-IR at 5 kW/m^2 and a boiling water bath were compared (Fig. 5.2).

Paprika powder with an a_w value of 0.50 could be heated much faster using medium-IR than using a water bath, which requiring heating times of 3 and 22 min, respectively. At a_w 0.80, the heating time in the water bath was reduced to 18 min, due to better heat conduction. However, at a_w 0.80, medium-IR required a longer heating time – approximately 4 min – due to less surface heat absorption and reduced penetration depths at higher a_w values (Ginzburg, 1969).

The temperature gradient between depths of 1 and 8 mm in the powder bed is much higher with IR heating than with the water bath. At higher a_w values, this temperature gradient decreased, being negligible with water bath heating, but still being 40°C with medium-IR heating. Radiation is the dominant mechanism of heat transfer to the surface, causing a fast temperature increase on the surface and to a depth of 1 mm; at depths of 3 and 8 mm, however, most heat was transferred by conduction, due to the poor penetration depth of IR.

The poor thermal conductivity of powders and foods in general can explain the great temperature difference between the surface and inside of the powder bed. At higher a_w values, the temperature gradient between the surface and inside decreased, mainly due to more effective heat conduction and higher surface reflection. The penetration depth of IR is expected to decrease with increased a_w; accordingly, in dry powdered products, such as

flour or salt, IR had a penetration depth of 2 mm compared with only 1 mm in a slurry product, such as tomato paste (Ginzburg, 1969).

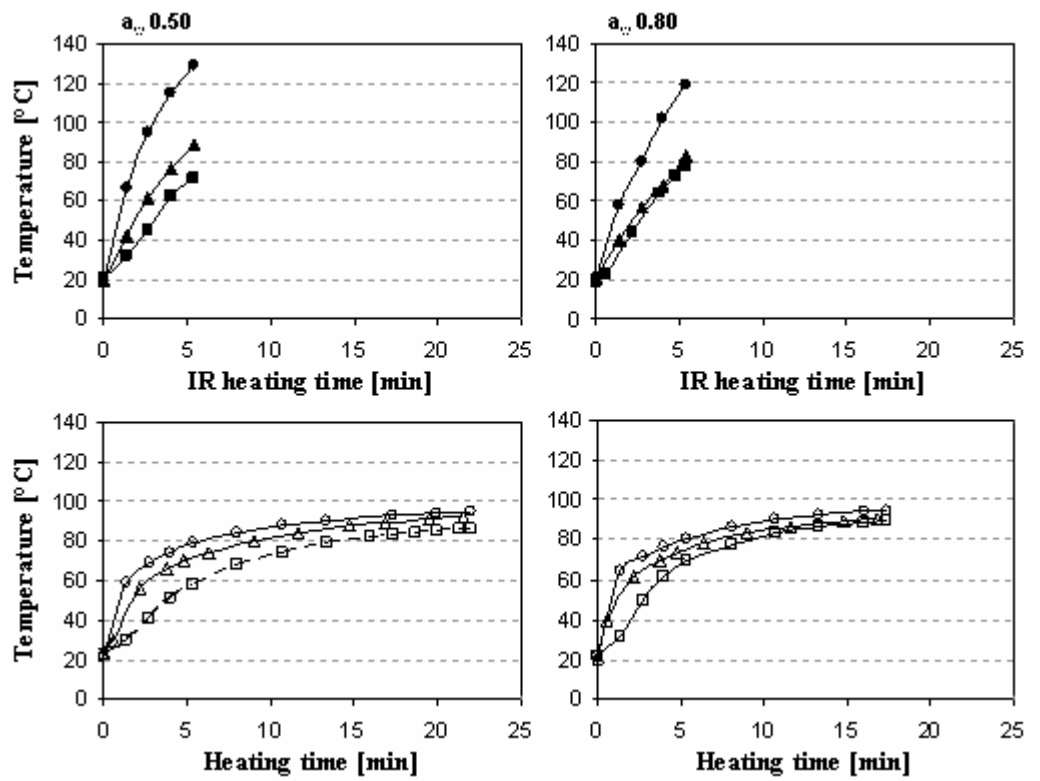

Figure 5.2: Temperature profile of paprika powder at a_w values of 0.50 and 0.80 measured at depths of 1 (● and ○), 3 (▲ and Δ), and 8 mm (■ and □) during heating with medium-IR (5 kW/m^2) and in a water bath (100°C), respectively.

5.1.3 IR heating of paprika slurry and water

Paprika powder with an a_w of 0.96 (slurry) and pure water were heated using selected constant IR heat fluxes (Fig. 5.3). Temperature was measured in 1-mm depth increments in a 10-mm-deep bed.

Using a near-IR heat flux of 23 kW/m^2, no differences were evident until the boiling point was reached. Heating at near- and medium-IR heat fluxes of 11 kW/m^2 produced the same behaviour in paprika powder until 100°C was reached after 8 min. With near- and medium-IR heating, the temperature of pure water reached approximately 90 and 85°C, respectively, after 8 min, while further heating caused a slight increase to 97 and 90°C, below the boiling point of water (particularly for medium-IR). At a medium-IR heat flux of 5 kW/m^2, both tested samples displayed the same heating behaviour up to 70°C; further

heating caused a faster temperature increase for paprika slurry than for pure water, reaching 93 and 80°C after 17 min heating, respectively.

These differences in the heating of paprika slurry and pure water can be explained by (1) the absorption behaviour of water at different wavelengths, which absorbs the maximum amount of IR at 0.8–1.0 μm, close to the near-IR wavelength of 1.2 μm, and (2) the fact that IR absorption behaviour is a matter of product colour and surface reflection, paprika displaying higher absorption than water (it is significant, in this regard, that water is somewhat transparent to radiation). Total reflection from materials is known to decrease with the decrease in moisture content (Ginzburg, 1969).

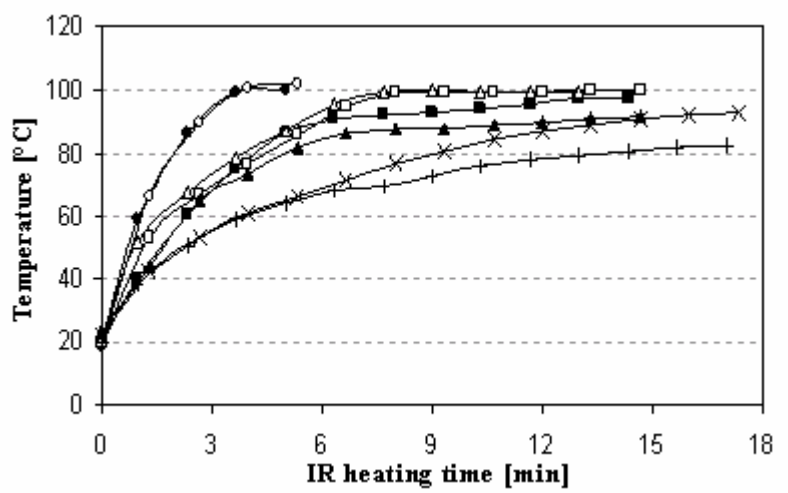

Figure 5.3: Heating of water and paprika powder (a_w 0.96) at constant near-IR heat fluxes of 23 (● and ○) and 11 (■ and □) kW/m^2 and constant medium-IR heat fluxes of 11 (▲ and Δ) and 5 (+ and ×) kW/m^2, respectively.

5.1.4 Temperature profile in paprika powder during IR heating

The effects of a_w values of 0.5, 0.8, and 0.96 on the heating of paprika powder at heat fluxes of 23 and 11 kW/m^2 (near-IR) and 11 and 5 kW/m^2 (medium-IR) are presented in Fig. 5.4.

5.1.4.1 Effect of heat flux and a_w

As expected, the higher the heat flux, the faster the temperature increase on the surface of the paprika powder bed. Lower a_w values resulted in faster temperature increases and greater temperature differences between the surface of and inside the powder bed. At lower

a_w values, powders have lower transmissivity and lower surface reflection levels, i.e. higher energy absorption on the surface (Ginzburg, 1969).

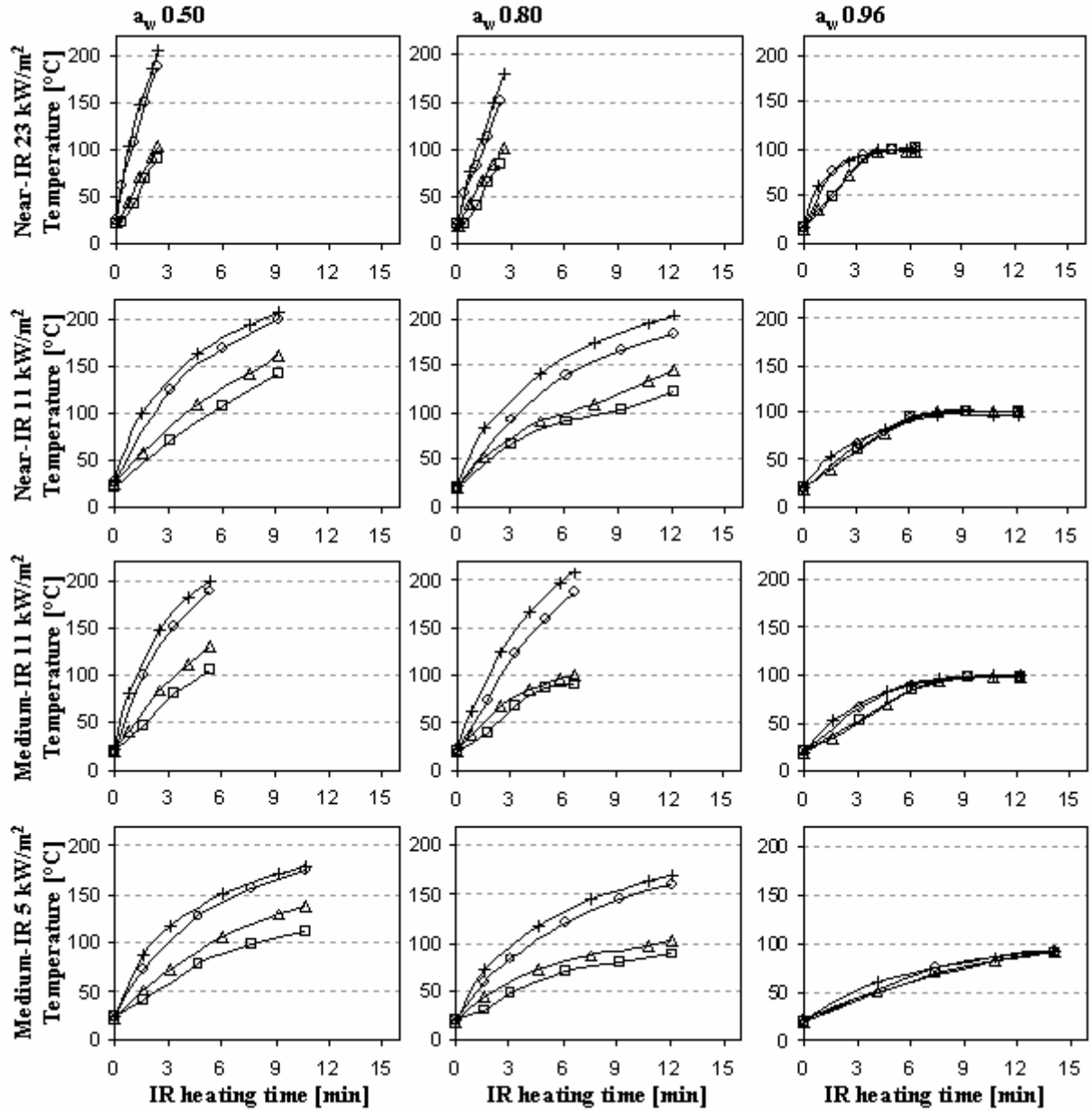

Figure 5.4: Temperature of paprika powder measured on the surface (+) and at depths of 1 (○), 3 (Δ), and 8 mm (□) at a_w values of 0.5, 0.8, and 0.96 during heating at near-IR heat fluxes of 23 and 11 kW/m^2 and medium-IR heat fluxes of 11 and 5 kW/m^2.

5.1.4.2 Effect of wavelength

Comparing the effects of near-IR and medium-IR at a heat flux of 11 kW/m^2, it was observed (Fig. 5.4) that at a_w values 0.5 and 0.8, higher surface temperatures were achieved with medium-IR, while the interior temperatures (at depths of 3 and 8 mm) differed by only a few degrees. For example, after 3 min of heating, the surface temperature of a_w 0.5

paprika powder was 160°C with medium-IR and 140°C with near-IR, while at a depth of 3 mm, the temperatures were 100 and 98°C, respectively. With near-IR heating at a_w values of 0.5 and 0.8, there was a slightly smaller temperature difference between depths of 3 and 8 mm, which may be explained by the greater penetration depth of near-IR. Longer wavelengths penetrate the powder only a little, so the energy is mostly converted to heat on the surface, whereas shorter wavelengths penetrate somewhat further into the material (Sakai & Hanzawa, 1994). Increasing the a_w decreased the differences in surface temperature between the near- and medium-IR treatments, resulting in quite similar temperature curves for both wavelengths at a_w 0.96.

Notably, for a_w 0.8 paprika powder, the same temperature curve was observed with near- and medium-IR heating up to 80°C; as soon as the water was removed, however, a higher surface temperature was observed with medium-IR (Fig. 5.5). This may be explained by the different behaviours of wet and dry particles when absorbing radiative energy.

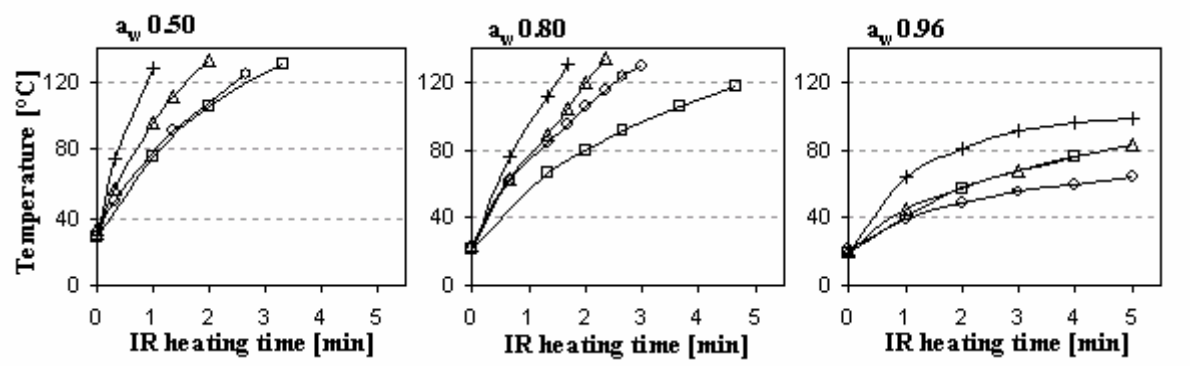

Figure 5.5: Effect of near-IR heat fluxes of 23 (+) and 11 (○) kW/m^2 and medium-IR heat fluxes of 11 (Δ) and 5 (□) kW/m^2 on the surface temperature of paprika powder at a_w values of 0.5, 0.8, and 0.96.

5.1.5 Changes of product quality during IR heating

5.1.5.1 Effect on colour

Changes of the surface, bottom and overall colour of wetted paprika powder during IR heating are presented in Fig. 5.6. Colour was expressed as the product of the *L* (lightness) and *a* (redness) colour parameters (i.e. $a \times L$), values >500, 500–300, and <300 being rated as red, medium red, and dark, respectively (Ramakrishnan & Francis, 1973). Dark samples having values of 300–200 were rated as brown and <200 as black or charred.

For near-IR heat fluxes of 23 kW/m^2 and medium-IR heat fluxes of 11 kW/m^2, the surface colour value decreased at all a_w values, becoming unacceptably dark and charred

within a few minutes. This property of IR could be used in processes aiming to create particular surface colorations; however, in our case such surface coloration is undesired, as it decreases product quality.

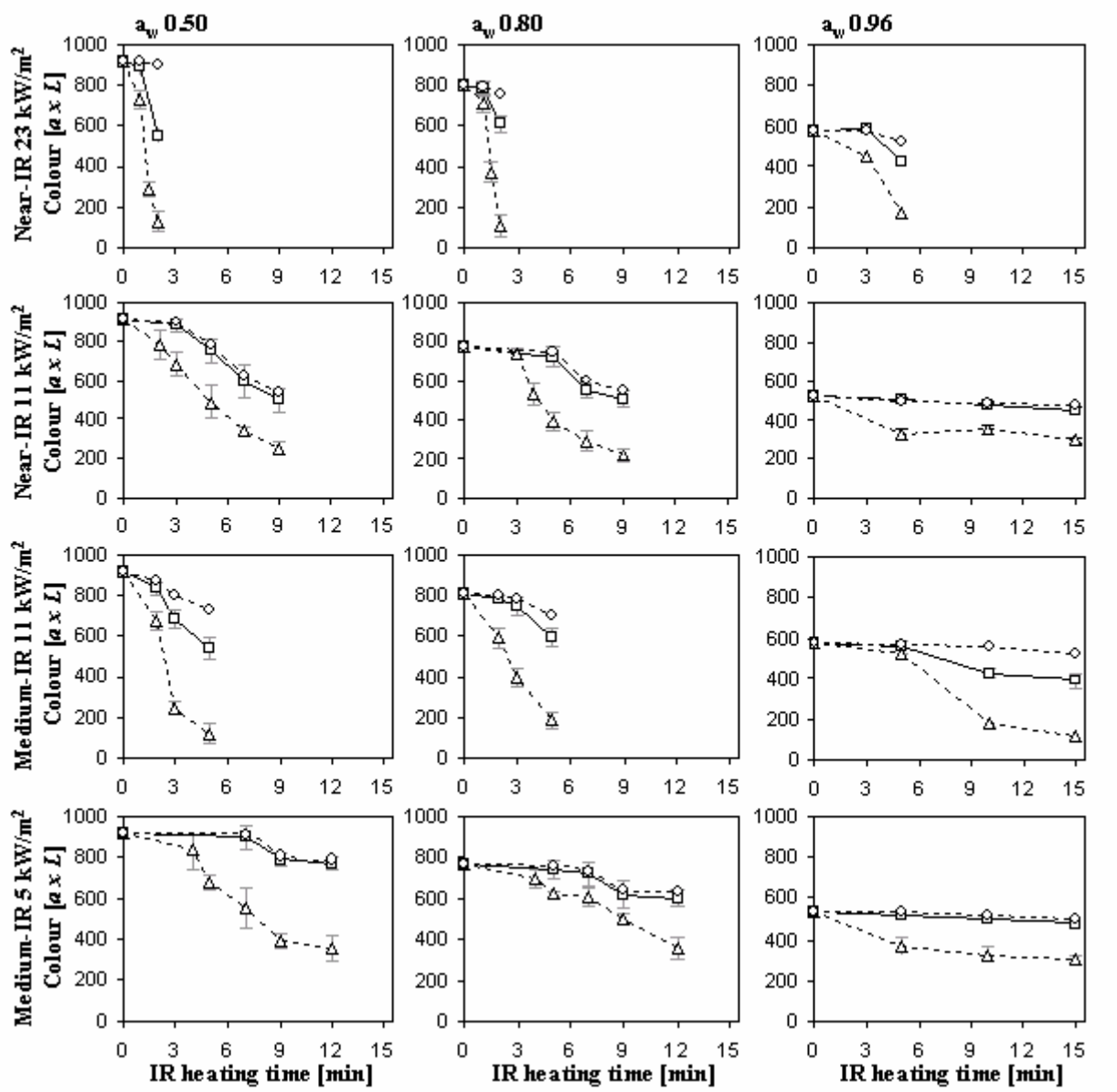

Figure 5.6: Surface (Δ), bottom (○) and overall (□) colour measurements (*a x L*) of paprika powder at a_w 0.50, 0.80, and 0.96 for near-IR of 23 and 11 kW/m^2 and medium-IR of 11 and 5 kW/m^2.

For treatments at near-IR of 11 kW/m^2 and medium-IR of 5 kW/m^2, surface browning occurred to values of the medium-red range, but bottom and overall colour stayed within acceptable red colour levels ($a \times L > 500$). Overall colour was a mixture of bottom and surface colour. Surface colour showed significantly lower values, while bottom and overall colour did not differ greatly. Thus for the overall colour impression, surface colour had no significantly negative effect.

The overall colour values for the cited heat fluxes after 9 min and 12 min were similar to the surface colour values after 4 min and 5 min, respectively. During the first 3 min of heating, no significant surface colour changes ($P < 0.05$) were observed, as the temperature was under 100°C. A significant reduction in colour value on the surface ($P < 0.05$) was observed after 5 min, however, as the temperature then exceeded 130°C. Further heating caused a rapid decrease of colour value, due to further surface temperature increase, in agreement with the results of Ramakrishnan (1973).

Water loss, a_w, and microbiology studies were done only with a near-IR heat flux of 11 kW/m^2 and a medium-IR heat flux of 5 kW/m^2, since these conditions result in better overall product colour.

5.1.5.2 Effect on a_w and total water loss

The total water loss and change of a_w during IR heating were studied for a medium-IR heat flux of 5 kW/m^2 and a near-IR heat flux of 11 kW/m^2 (Fig. 5.7). The total water loss and change in overall a_w were higher for a near-IR heat flux of 11 kW/m^2 and a_w values of 0.5 and 0.8, due to the higher surface temperatures. IR heating effectively removed water, especially at a_w 0.5 and 0.8, at which 40–95% of the total water could be evaporated within 9 min; however, at a_w 0.96 only 10–30% of the total water could be removed within the same heating period.

A moisture gradient developed between the sample surface (dry zone formation) and centre, similar to the temperature and colour gradients previously described. After 4 min of heating, paprika wetted to a_w values of 0.5 and 0.8 decreased significantly in surface wetness to a_w 0.3 and 0.4, respectively, due to surface heating. The bottom and overall a_w were not significantly ($P < 0.05$) affected in that time, though further heating did decrease the overall a_w as well, while the bottom a_w remained higher. At a_w 0.96, no changes were observed in the surface, bottom, or overall a_w values ($P < 0.05$) after 5 min heating.

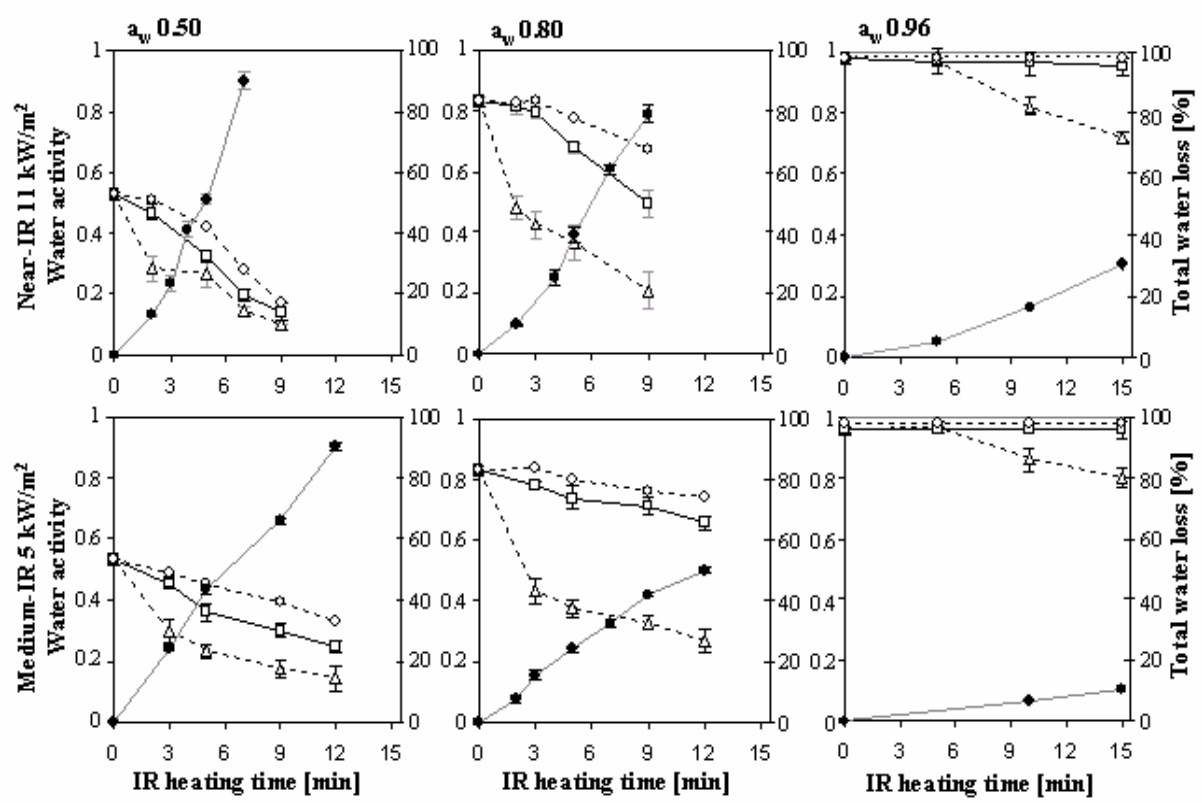

Figure 5.7: Effect of IR heating for near-IR heat flux of 11 kW/m^2 and medium-IR heat flux of 5 kW/m^2 on surface (Δ), bottom (○) and overall (□) a_w and on total water loss (●) in paprika powder at a_w 0.5, 0.8, and 0.96.

5.1.5.3 Effect on *B. cereus* spores and the natural background flora

The effect of IR heat treatment on microbial numbers was studied for a medium-IR heat flux of 5 kW/m^2 and a near-IR heat flux of 11 kW/m^2. The IR treatments tested and their effects on microbial counts of *B. cereus*, SIK 340, spores and natural background flora are presented in Table 5.1. The temperature history of the product during IR heating is shown in Fig. 5.4. The *total* IR treatment time was determined based on retaining acceptable overall colour ($a \times L \sim 500$) and on process time at a product temperature over 95°C (reached at a depth of 1 mm in the powder bed). For a heat flux of 11 kW/m^2, the limits of acceptable overall colour of 503, 501, and 456 were reached after 8, 9, and 15 min, corresponding to a_w values of 0.50, 0.80, and 0.96, respectively (see Fig. 5.6); the respective process times over 95°C were 6, 6, and 8 min. For a heat flux of 5 kW/m^2, the colour changes were not as pronounced as for 11 kW/m^2, so the total process time was then determined so as to achieve a similar process time over 95°C for both treatments. Thus, for a heat flux of 5 kW/m^2, the durations of the process time over 95°C were 6, 7, and 9 min, which required total IR treatment times of 9, 10, and 28 min, corresponding to a_w values of 0.50, 0.80, and 0.96, respectively.

The number of *B. cereus* spores could be reduced by a maximum of only 1 $\log_{10}$ spores/g in the surface sample and in the sample representing the overall powder bed (Table 5.1) at a_w values of 0.5 and 0.8. This low reduction is explained by the low a_w of the paprika sample, which increased the heat resistance of the spores. A similarly small reduction was also achieved on the surface of the sample with an initial a_w of 0.96. During heat treatment, the a_w decreased during the long warm-up period. In the centre parts of the a_w 0.96 sample (with a high remaining a_w), a significant reduction of 5 $\log_{10}$ spores/g was obtained at a heat flux of 5 kW/m^2 and of over 6 $\log_{10}$ spores/g at 11 kW/m^2.

No reduction was observed in the level of natural background flora at a_w 0.5 ($P > 0.05$). At a_w 0.8, however, overall reductions of 1.6 and 0.7 $\log_{10}$ CFU/g were obtained for heat fluxes of 11 and 5 kW/m^2, respectively, while no microbes were observed in the a_w 0.96 paprika.

Table 5.1: Concentration of *B. cereus*, SIK 340, spores and natural background flora ($n = 2 \times 3$) (±SD) in different locations in paprika powder with a_w values of 0.5, 0.8, and 0.96 after exposure to a medium-IR heat flux of 5 kW/m^2 and a near-IR heat flux of 11 kW/m^2. The initial population was 7.48 $\log_{10}$ *B. cereus* spores/g in inoculated samples and 4.90 $\log_{10}$ CFU/g of the background flora in uninoculated samples

			Microbial counts after IR heating ($\log_{10}$ CFU/g)		
			B. cereus		Natural flora
	Heat flux (kW/m^2)	Location in powder	Total plate count	Spore plate count	Total plate count
a_w 0.50	5 med-IR	surface	7.1		4.7
		overall	7.2 (±0.0)	7.2 (±0.1)	4.5 (±0.1)
	11 near-IR	surface	6.8		4.6
		overall	7.1 (±0.1)	7.0 (±0.1)	4.4 (±0.1)
a_w 0.80	5 med-IR	surface	6.9		4.0
		overall	7.2 (±0.1)	7.0 (±0.5)	4.1 (±0.1)
	11 near-IR	surface	6.8		3.7
		overall	6.3 (±0.1)	6.2 (±0.3)	3.2 (±0.2)
a_w 0.96	5 med-IR	surface	7.0 (±0.3)	7.0 (±0.2)	4.7 (±0.1)
		inside	2.2 (±0.6)	2.1 (±0.4)	2.0 (±0.2)
		overall	6.2 (±0.5)	6.1 (±0.3)	3.5 (±0.2)
	11 near-IR	surface	6.6 (±0.2)	6.5 (±0.4)	<1
		inside	<1	<1	<1
		overall	5.8 (±0.2)	5.7 (±0.2)	<1

5.1.6 Summarized process observations for open heating unit 1

Applying constant heat flux produced an uncontrollable increase in surface temperature, which, especially at higher heat fluxes (i.e. a near-IR heat flux of 23 kW/m^2 and a medium-IR heat flux of 11 kW/m^2), resulted in significantly degraded product quality in terms of surface colour. However, at lower heat fluxes, the colour remained in the acceptable range, but long warm-up times were required. At the same heat flux and at low a_w values, higher surface temperatures were observed for medium-IR, while near-IR produced a slightly greater penetration depth. However, at higher a_w values, no temperature differences were observed between the two wavelengths.

The most critical aspect of the IR heating of paprika powder using an open heating unit was that the surface displayed undesired drying and browning at temperatures over 100°C. However, observed surface drying and browning did not necessarily affect the overall a_w and colour values.

As water was able to evaporate from the open system, spores at the surface of the powder mass increased their heat resistance, displaying low inactivation rates. No significant reduction in *B. cereus* spores was obtained at a_w values of 0.5 and 0.8. The surface value of a_w value of 0.96 had already decreased during the long warm-up period, resulting in poor reduction of microbial numbers on the surface and consequently the overall sample. Nevertheless, areas with high remaining a_w values, i.e. the bottom area, displayed complete inactivation (>6.5 $\log_{10}$ CFU/g) at a detection limit of 1 $\log_{10}$ CFU/g, the natural background flora showing itself to be more sensitive to IR heating.

In further discussion in this thesis, only surface and overall values are measured, as the surface represents the sensitive part during heating, while the overall colour represents the overall impression of the IR-treated paprika powder.

5.2 Closed IR heating unit 2 using IR glass

5.2.1 IR heating system

Fig. 5.8 shows the improved heating unit, closed heating unit 2, developed for decontaminating paprika powder.

The major problem encountered when heating using the previously discussed open heating unit was that the decrease of a_w, especially at the powder surface, resulted in a low spore inactivation rate. To address this problem, a near-IR-transparent glass panel made of quartz glass (Neoceran 0; Ircon Drying Systems, Vänersborg, Sweden) was placed on the top of the heating unit. No headspace was left between powder and glass panel to prevent the undesired evaporation of water from the powder. In addition, the heating unit was surrounded by aluminium-covered insulation foam to reduce heat loss from the system.

When using the open heating unit, surface colour degradation was most significant when temperatures exceeded 130°C, so such temperatures should be avoided. Using a combination of different heat fluxes for heating, i.e. *variable IR heat fluxes*, could help preserve product quality; the powder could be rapidly heated to the desired product temperature using high heat fluxes, and then kept at that temperature using lower heat fluxes. Near-IR heat fluxes were used, as they facilitate both high and low heat fluxes.

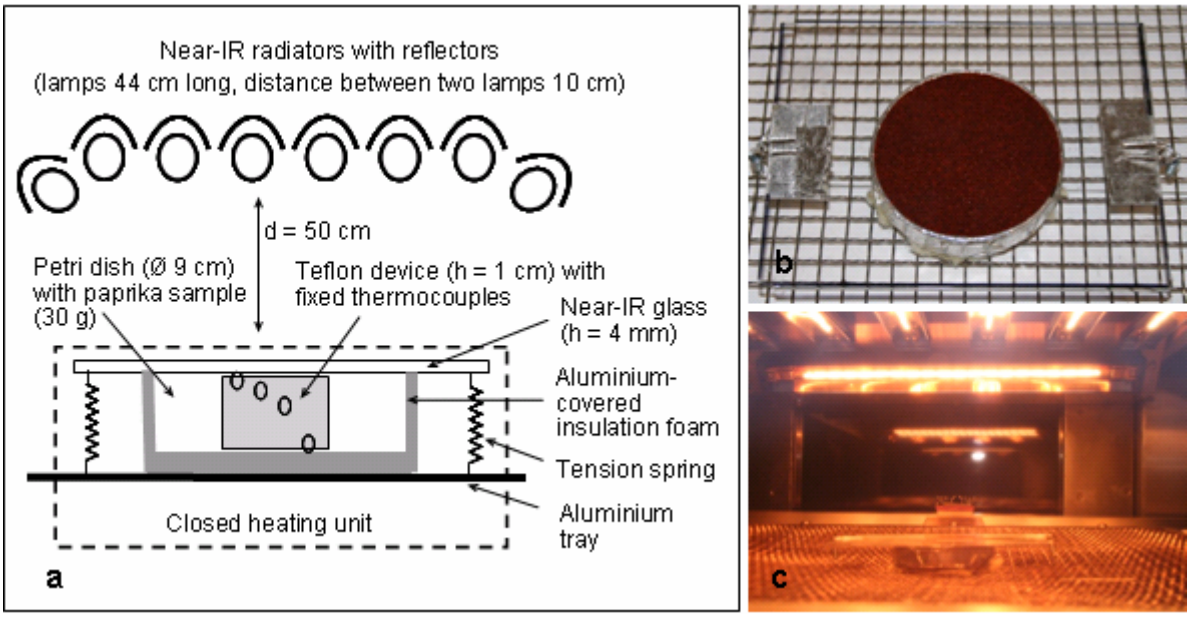

Figure 5.8: Schematic cross-section of the IR heating chamber and closed heating unit 2 (a). Top view of closed heating unit 2 with IR-transparent glass (b); and side-view of closed heating unit 2 during near-IR heating (c).

5.2.2 Heat fluxes through IR-transparent glass

The heat fluxes obtained in closed heating unit 2 (Table 5.2) indicated that the glass cover had a transparency factor of 90%, in agreement with the specifications provided by the supplier (Ircon Drying Systems AB, Vänersborg, Sweden).

Table 5.2: Selected IR output powers and their corresponding near-IR heat fluxes obtained with and without use of a near-IR-transparent glass between IR source and black body; with the IR transparency factor of the glass

Output power	Heat flux (kW/m^2)		IR transparency factor (%)
	Without IR glass	With IR glass	
100%	23.4 ± 0.3	20.4 ± 0.1	87
25%	11 ± 0.2	9.5 ± 0.2	86
1%	4.0 ± 0.4	3.5 ± 0.2	88

5.2.2.1 Experimental design

IR treatments at product temperatures of 90, 95, and 100°C generated by variable near-IR heat fluxes were tested in paprika powder with a_w values of 0.84 or 0.88. The temperature profiles, product a_w, reductions in *B. cereus* spore concentration, and colour were assessed during or after IR treatment. In addition, pH levels of 4.0 and 4.5 were compared for their effects on spore concentration at 90 and 100°C. The total IR process time was monitored and consisted of warm-up (from 20°C up to desired product temperature) and holding/inactivation periods (starting when the desired product temperature was reached in the powder bed). Studying the impact of product temperature, a_w, and pH on the reduction of *B. cereus* spore concentrations required a particular experimental design (Table 5.3). The spore concentration was counted after certain holding times at 90, 95, and 100°C; four replicates were made of each combination of holding time and temperature. Experiments were performed using variable heat fluxes, as shown in Table 5.4. The total IR process time consisted of a fast (phase I) and a moderate (phase II) warm-up period followed by a holding period at the desired product temperature (phase III). In phase III, infrared energy was applied in pulses (turning the near-IR radiators on and off) to keep the product temperature constant; for example, at an a_w of 0.84 and 90°C the radiators were turned on for 20 s and then off for 60 s (see Table 5.4).

Table 5.3: Experimental design for testing combined effects of temperature, pH, and a_w value on thermal inactivation of *B. cereus* spores in paprika powder using near-IR radiation

Water activity (a_w)	pH	Product temperature (°C)
0.84	4.0	90
		100
	4.5	90
		95
		100
0.88	4.0	90
		100
	4.5	90
		95
		100

Table 5.4: Holding times with variable near-IR heat fluxes for paprika powder (10 mm thickness) with a_w values of 0.84 and 0.88 during warm-up periods (phases I and II) to reach product temperatures of 90, 95, and 100°C and on/off times (pulsed IR operation) to maintain the desired product temperatures (phase III)

Phase	IR heat flux (kW/m^2)	Product temperature (°C)	a_w 0.84	a_w 0.88
			Time (s)	Time (s)
I	20	90, 95, 100	90	90
II	9.5	90	120–140	140–160
		95	150–170	170–190
		100	180–200	200–220
III	3.5 (pulsed)	90	20/60	30/60
		95	50/30	60/20
		100	90/20	90/20

5.2.3 Temperature profile

The measured temperature profiles at a_w values of 0.84 and 0.88 at a product temperature of 100°C obtained after heating with variable near-IR are shown in Fig. 5.9. The profiles at 90 and 95°C display the same behaviour and are therefore not shown. Measured surface temperatures at product temperatures of 90, 95, and 100°C in phases I–III are presented in Table 5.5.

Use of a high heat flux (20 kW/m^2) in phase I caused a large temperature difference within the powder bed of 90 and 70°C at a_w values of 0.84 and 0.88, respectively, due to high heat absorption on the surface and limited heat conduction within the powder bed

(Ginzburg, 1969). When the surface temperature reached 115–125°C (after 1.5 min) phase II began, during which deeper parts of the powder (3 and 8 mm) reached the desired product temperature by means of heat conduction. When the desired product temperature was reached, temperature differences between the surface and a depth of 8 mm were 40 and 20°C at a_w values of 0.84 and 0.88, respectively. During phase III, the temperature gradient within the powder decreased to 10–15°C at both a_w values.

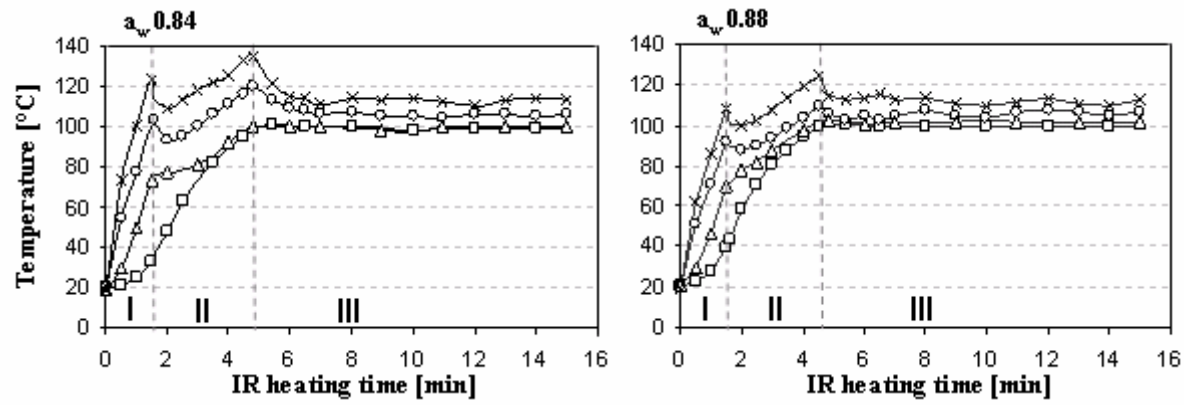

Figure 5.9: Temperature of paprika powder measured on the surface (+) and at depths of 1 (○), 3 (Δ), and 8 mm (□) at a_w values of 0.84 and 0.88 during heating until reaching 100°C at a depth of 8 mm with variable near-IR heat fluxes of 20 (I), 9.5 (II), and 3.5 kW/m^2 (III, pulsed operation).

Table 5.5: Surface temperatures of paprika powder with a_w values of 0.84 and 0.88 exposed to near-IR heat fluxes of 20 (phase I), 9.5 (phase II), and 3.5 kW/m^2 (phase III, pulsed operation) at product temperatures of 90, 95, and 100°C

Product temperature (°C)	a_w 0.84			a_w 0.88		
	Surface temperature (°C)			Surface temperature (°C)		
	Phase I	Phase II	Phase III	Phase I	Phase II	Phase III
90	125 ± 2	125 ± 3	101 ± 2	115 ± 2	105 ± 2	100 ± 2
95	125 ± 2	135 ± 2	110 ± 2	115 ± 2	115 ± 2	110 ± 2
100	125 ± 2	140 ± 3	115 ± 4	115 ± 2	120 ± 2	115 ± 3

5.2.4 Effect of heating on a_w value and total water loss

In Fig. 5.10, changes in the surface and overall a_w values of paprika powder are presented for IR treatment at 100°C. Due to the use of a closed sample holder, the overall a_w value decreased only slightly from the initial values of 0.84 and 0.88 to 0.80 and 0.84, respectively. However, the surface a_w value, which had already decreased significantly ($P < 0.05$) during phase I, levelled out at approximately 0.4–0.5 in phase II, due to surface temperatures over 100°C and unavoidable small gaps between the glass panel and the

insulation of the sample holder (indicated in Fig. 5.11). Further heating in phase III increased the surface a_w value to 0.6–0.7, possibly due to the dispersal of steam within the powder bed. The total water loss during heating was of around 4% mostly due to losses from the surface.

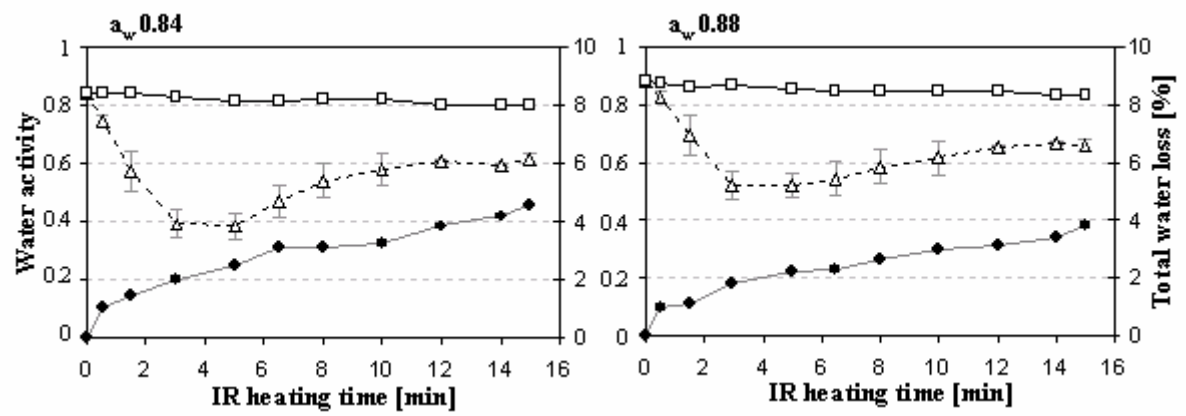

Figure 5.10: Surface (Δ) and overall (□) a_w value, and total water loss (●) measurements of paprika powder at 100°C with initial a_w value of 0.84 and 0.88 during IR heating at heat fluxes of 20 (I), 9.5 (II), and 3.5 kW/m^2 (III).

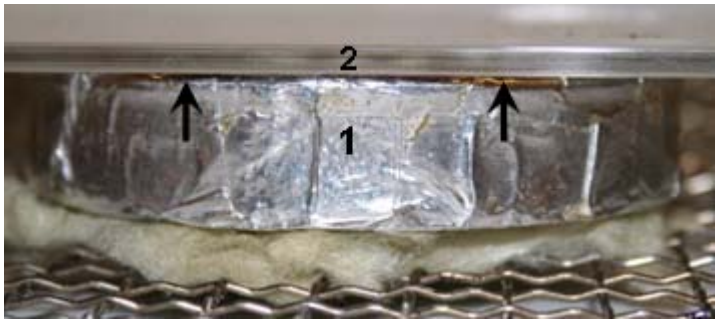

Figure 5.11: Side-view of closed heating unit 2 with insulated sample holder (1) and IR-transparent glass panel as a top lid (2); the arrows indicate the small gaps between the sample holder and the top lid.

5.2.5 Reduction of *B. cereus* spore concentrations

5.2.5.1 Effect of a_w and temperature

Fig. 5.12 shows the remaining population of the total plate count of *B. cereus* in paprika powder at a_w values of 0.84 and 0.88 after IR heat treatment at holding temperatures of 90, 95, and 100°C. The product temperature distribution during heating is presented in Fig. 5.9. A remaining spore concentration of approximately 2.5 $\log_{10}$ CFU/g was observed at 95 and 100°C at an a_w value of 0.88 after 6 min of holding time, which is below the acceptable level for *B. cereus* in spices of 4 $\log_{10}$ CFU/g (Völker et al., 1998). This quality parameter

was not achieved at an a_w value of 0.88 at 90°C or at an a_w value of 0.84 at 90–100°C after 6 min, though the reduction of spore numbers was of 1–2.5 $\log_{10}$ CFU/g. Inactivation times longer than 6 min resulted in tailing (except at 90 and 95°C at an a_w value of 0.84) with viable spore counts of approximately 2 $\log_{10}$ CFU/g at 95 and 100°C at an a_w value of 0.88. Spore inactivation depends on temperature, pH and a_w of the heating medium (Leguerinel et al., 2004). As heating by IR showed a temperature- and a_w-gradient within the powder bed, different inactivation kinetics for spores of *B. cereus* occurred. The remaining part of the spore population is mainly due to the increased heat resistance of spores on the surface due lower a_w values (Jeng & Woodworth, 1990; Ababouch et al., 1995). Notably, the warm-up period was observed to have an additional effect on microbial inactivation at 100°C at an a_w value of 0.88.

D-values were calculated for up to 6 min of holding time. Product temperatures of 90, 95, and 100°C displayed *D*-values of 9.4, 4.1, and 2.8 min at an a_w value of 0.84, and of 4.1, 1.3, and 1.6 min at an a_w value of 0.88. As expected, the higher the temperature and a_w value, the higher the achieved reduction rate, i.e. the lower the *D*-value. Increasing the temperature from 90 to 95°C had a larger impact on *D*-value than did increasing it from 95 to 100°C.

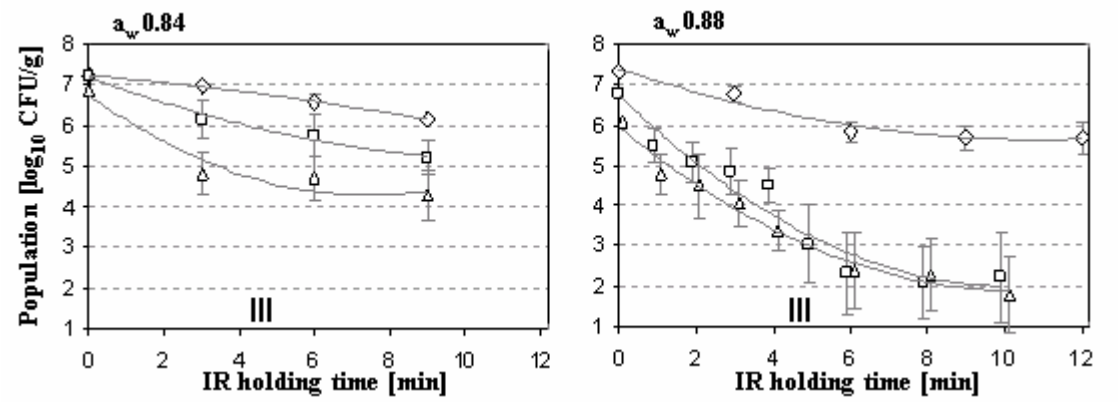

Figure 5.12: Total plate counts of *B. cereus*, SIK 340, in paprika powder with a_w values of 0.84 and 0.88 after near-IR holding times (phase III) at product temperatures of 90 (◊), 95 (□), and 100°C (Δ). The concentration before heating was 7.23 $\log_{10}$ *B. cereus* spores/g and the detection limit was 1 $\log_{10}$ CFU/g.

5.2.5.2 Effects of a_w and pH values

The effects of pH 4.5 and 4.0 on spore concentration were studied at an a_w value of 0.88 at product temperatures of 90 and 100°C (Fig. 5.13). No substantial effect was attained by reducing the pH from 4.5 to 4.0, though there was a tendency for decreased heating medium pH to reduce the apparent heat resistance of *B. cereus* spores at lower product

temperatures (Gaillard et al., 1998; Penna & Moraes, 2002). The microbial reduction at 90°C was approximately 1 $\log_{10}$ CFU/g greater at pH 4.0 than at pH 4.5, whereas at 100°C no difference was observed. However, a pH of 4.5 is considered the boundary for growth of *B. cereus*, and at a pH under 4.35 growth can be inhibited (Raevuori & Genigeorgis, 1975).

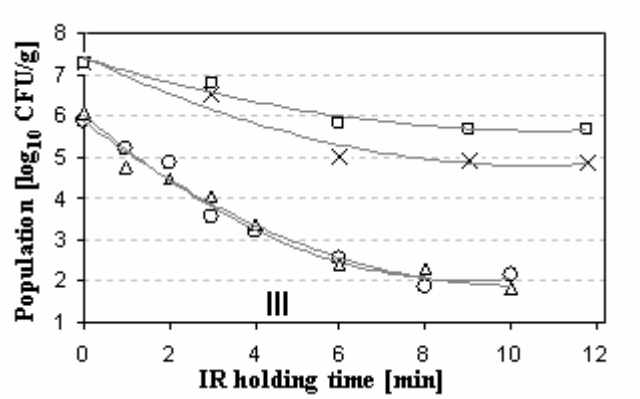

Figure 5.13: Total plate counts of *B. cereus*, SIK 340, in paprika powder with an a_w value of 0.88 after near-IR holding times during phase III at product temperatures of 90°C at pH 4.0 (×) and 4.5 (□), and 100°C at pH 4.0 (Δ) and 4.5 (○). The population before heating was 7.23 $\log_{10}$ *B. cereus* spores/g.

5.2.5.3 Effect on colour

Colour was expressed as the product of the *L* (lightness) and *a* (redness) colour parameters (i.e. $a \times L$), values >500, 500–300, and <300 being rated as red, medium red, and dark, respectively (Ramakrishnan & Francis, 1973). Dry paprika powder (a_w 0.50) displayed an initial colour value of 950. When wetted to a_w values of 0.84 and 0.88, the initial value decreased to 780 and 660, respectively.

The overall (Fig. 5.14) and surface colours (Fig. 5.15) of wetted paprika powder during IR heating were studied at 90, 95, and 100°C. The product temperature distribution during heating is presented in Fig. 5.9. Overall colour remained at its initial value at both a_w values during phase I, but decreased significantly ($P < 0.05$) during phase II. The total pigment content (i.e. mainly carotenoids) is affected by product temperature when it exceeds 60°C, and the effect has been reported to be more obvious at 80°C and higher (Malchev et al., 1982). Heating during phase III did not reduce overall colour values any further, as neither the product temperatures further increased nor overall a_w values changed (Ramakrishnan & Francis, 1973; Ladrón de Guevara et al., 2005).

At an a_w value of 0.84, the surface colour decreased constantly during phases I and II, while at a_w 0.88, colour degradation appeared only in phase I. In phase II, surface temperatures of over 100°C caused surface drying, resulting in a lighter surface colour than overall colour, for example, at a_w values of 0.84, 700, and 650, respectively. Further heating

during phase III induced a constant decrease in the surface colour values to the medium red interval ($a \times L \sim 400$) at an a_w value of 0.84, due to the combination of a lower a_w value and higher surface temperatures of 105–115°C for a longer heating period. At an a_w value of 0.88, the colour value decreased to the red colour interval ($a \times L \sim 500$) at 90 and 95°C after 7 min of IR heating and remained there, whereas at 100°C the colour value continued to decrease to the same level as was seen at an a_w value of 0.84.

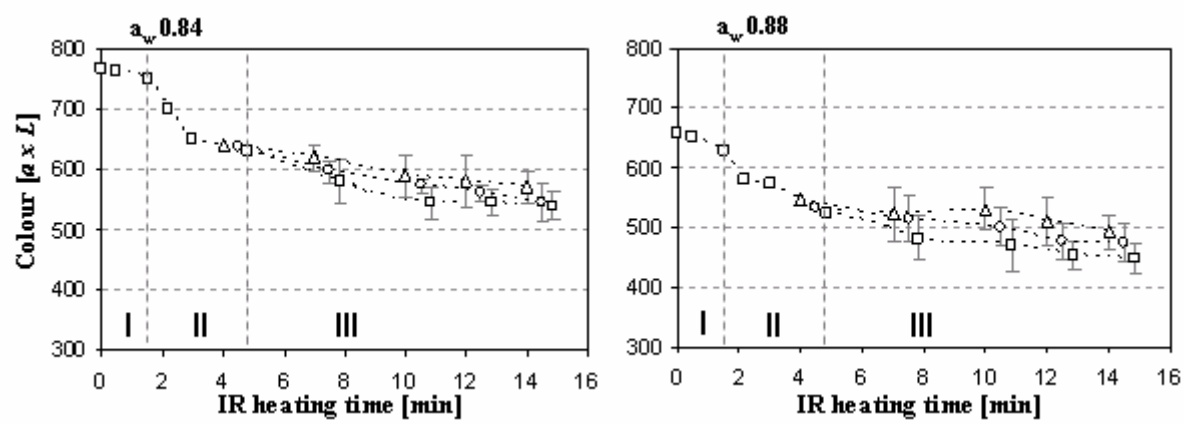

Figure 5.14: Overall colour measurements ($a \times L$) of paprika powder with a_w values of 0.84 and 0.88 at product temperatures of 90 (Δ), 95 (○), and 100°C (□) during heating at near-IR heat fluxes of 20 (I), 9.5 (II), and 3.5 kW/m^2 (III).

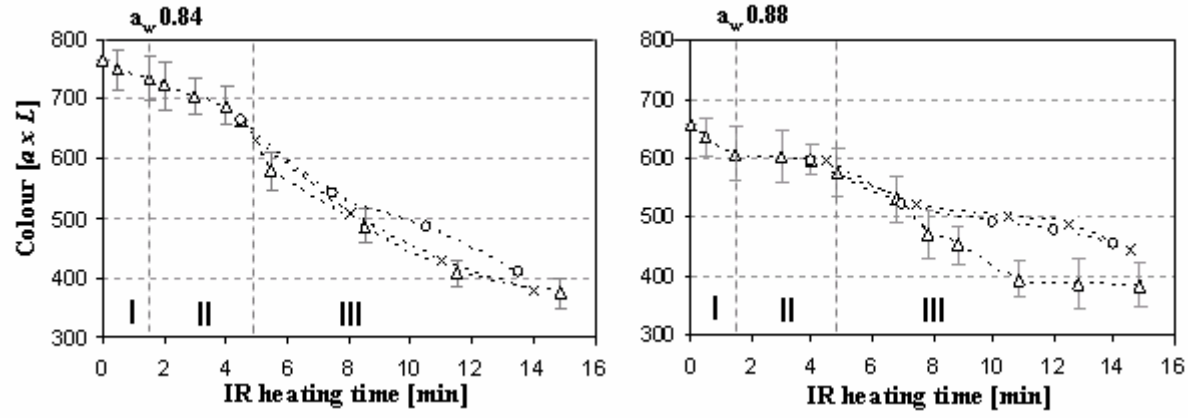

Figure 5.15: Surface colour measurements ($a \times L$) of paprika powder with a_w values of 0.84 and 0.88 at product temperatures of 90 (○), 95 (×), and 100°C (Δ) during heating at near-IR heat fluxes of 20 (I), 9.5 (II), and 3.5 kW/m^2 (III).

5.2.6 Drying of decontaminated powder

The wetted and decontaminated paprika powder was dried to facilitate long-term storage and prevent microbial growth. For stable storage, it is recommended that paprika powder have an a_w value of 0.3–0.5 (Kim et al., 1984; Lee et al., 1991). The final drying operation after IR decontamination was done in an open drying unit (Fig. 5.16), which was an adapted version of open heating unit 1 tested in section 5.1.

In phase I, the wetted decontaminated powder was placed into the open drying unit and mixed. Phase II was the first drying step, when the powder was subject to a constant near-IR heat flux of 9 kW/m^2 for 2 min; then, in phase III, the powder was mixed again (using a sterile spatula) without IR heating. Phase IV, the second drying step, used the same IR heating parameters as in phase II.

Screening tests indicated that a powder bed thickness of 10 mm was too thick for efficient drying, requiring overly long drying times (results not shown). Hence, the powder was transferred to a larger bottom-insulated sample holder (Fig. 5.16) and spread to a bed thickness of 5 mm using a sterile spatula. Mixing the powder loosened it; this decreased the bulk density, which facilitated evaporation of water from the powder.

Before drying, powder with an a_w value of 0.88 was decontaminated in closed heating unit 2 (Fig. 5.8), using the recommended IR heating parameters from Table 5.4 for a product temperature of 100°C and 6 min of holding time. The top lid (i.e. an IR-transparent glass panel) of closed heating unit 2 could be quickly and easily removed, converting the unit to an open drying unit.

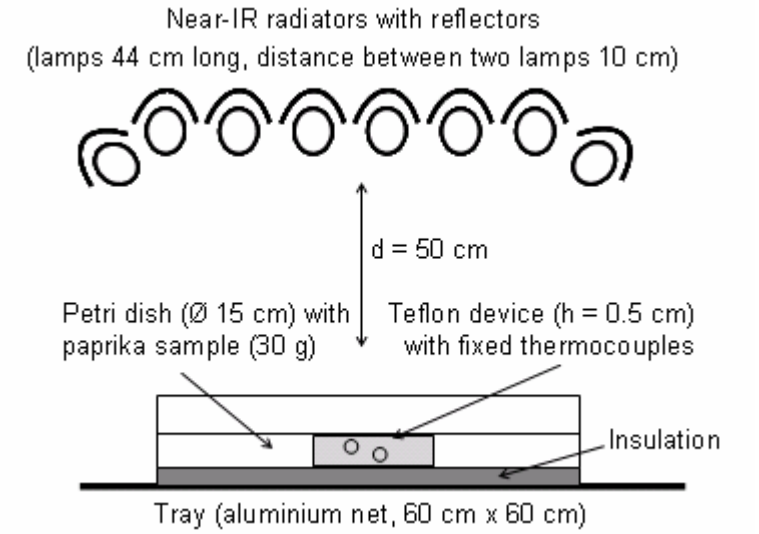

Figure 5.16: Schematic cross-section of the adapted open heating unit 1 for drying paprika powder on a Teflon device fixed with thermocouples at depths of 1 and 3 mm.

5.2.6.1 Reduction of a_w value

The surface and overall a_w values and overall water content of paprika powder during the course of drying are presented in Fig. 5.17. The overall a_w value decreased from approximately 0.84 to 0.50 during drying. Applying IR heating, i.e. during decontamination prior to drying (t_0) and during phases II and IV, caused a faster decrease of the surface than the overall a_w, e.g. to 0.60 and 0.77, respectively, after phase II. Subsequent powder mixing (phases I and III) eliminated this a_w gradient. In each mixing period, the water content declined by a maximum of 10%, while the IR heating periods (phases II and IV) achieved 30 and 40% reductions, respectively. Thus drying is mainly achieved by IR heating, but mixing is required so as not to over-dry the surface.

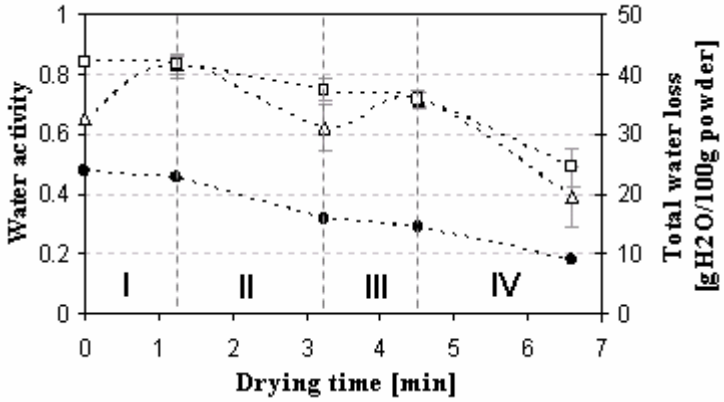

Figure 5.17: Surface (Δ) and overall (□) a_w values and overall water content (●) of paprika powder during drying, with cooling and powder mixing after IR heating at a product temperature of 100°C (phase I), the first drying step with a near-IR heat flux of 9 kW/m^2 (II), cooling and powder mixing (III), and the second drying step with a near-IR heat flux of 9 kW/m^2 (IV).

5.2.6.2 Temperature profile

Temperature was measured at depths of 1 and 3 mm in a 5-mm-deep powder bed (Fig. 5.18). After the decontamination step (t_0), the product temperature was approximately 100°C. During phase I, the temperature decreased to approximately 50°C. The first drying step (phase II), caused a constant increase in product temperature to 75–80°C. During subsequent powder mixing without IR heating (phase III), the temperature declined to 55–60°C. During the second drying step, the temperature increased again to 80–85°C (phase IV). The small observed temperature gradient within the powder bed is due to the moderate applied IR heat flux (causing a slow temperature increase) and low bulk density of the powder.

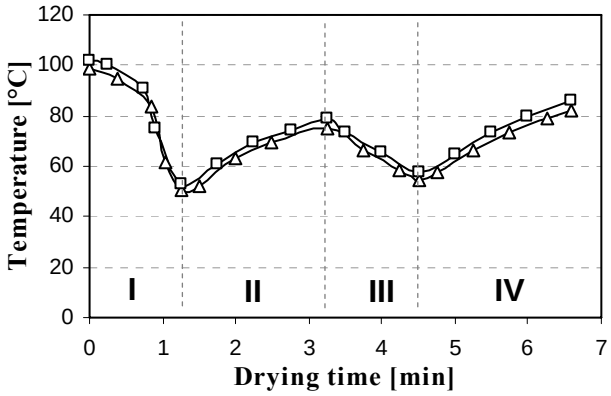

Figure 5.18: Temperature of paprika powder (initial a_w 0.88) measured at depths of 1 (□) and 3 mm (Δ) in a 5-mm-deep powder bed during drying to a_w 0.50; cooling and powder mixing after IR heating at a product temperature of 100°C (phase I), the first drying step at a near-IR heat flux of 9 kW/m^2 (II), cooling and powder mixing (III), and the second drying step at a near-IR heat flux of 9 kW/m^2 (IV).

5.2.6.3 Effect on colour

Overall colour values for paprika powder (a_w 0.88) during drying are shown in Fig. 5.19; the corresponding temperature profile measured during drying is depicted in Fig. 5.18.

Initially wetted paprika powder with an a_w value of 0.88 had a colour value of 680. When decontaminating the paprika using near-IR, the overall colour value decreased after 6 min of holding time at 100°C to 470 (Fig. 5.14). Subsequently drying the powder produced a marginal increase of the overall colour to a final value of 508 (at the final a_w of 0.50), similarto the increase in the colour of paprika at the lower a_w described earlier (section 4.1.3). Comparing this final dried colour value with the initial value for dry paprika (i.e. $a \times L \sim 950$) indicates that the near-IR decontamination treatment irreparably damaged the overall colour value, mostly due to the heat damage of carotenoids at temperatures over 60°C. However, the final dried colour value is still considered in the red range.

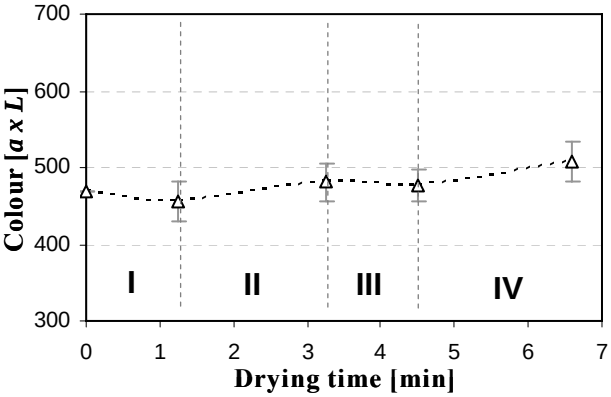

Figure 5.19: Overall colour measurements during drying of near-IR-heated paprika powder (initial a_w 0.88), with cooling and powder mixing after IR heating (phase I), the first drying step with a near-IR heat flux of 9 kW/m^2 (II), followed by cooling and powder mixing (III), and the second drying step with a near-IR heat flux of 9 kW/m^2 (IV).

5.2.6.4 Effect on microbial numbers

The microbial concentration of *B. cereus* spores inoculated in paprika powder before decontamination was measured before drying and after the second, i.e. final, drying step (Table 5.6). The drying operation had no additional effect in terms of reducing the final microbial concentration, due to moderate product temperatures and decreasing a_w values during drying.

Table 5.6: Effect of drying decontaminated paprika powder (initial a_w 0.88) to a final a_w of 0.50 on total plate counts of *B. cereus*, SIK 340, applying two drying steps, each with 2 min at a near-IR heat flux of 9 kW/m^2

Measurement	Log_{10} CFU/g
Before drying	2.4 ± 0.9
After final drying step	2.1 ± 0.5

5.2.7 Summarized process observations for closed heating unit 2

Variable near-IR heat flux applied during heating enabled fast and effective temperature regulation in the powder. The fast warm-up of the powder (within 4 min) was facilitated by applying a higher IR heat flux, while a lower heat flux allowed the desired product temperature to be maintain for microbial inactivation.

The overall a_w value remained at initial levels throughout IR heating, i.e. water was retained in the closed sample holder. However, due to small unavoidable gaps between the IR-transparent glass panel and the insulation, small amounts of water could escape, as indicated by the decrease in surface a_w. The presence of water in the paprika powder helped preserve its colour during heating. *Overall* colour changes were a result of the initial wetting level and product temperatures over 60°C. Changes in *surface* colour were more pronounced, depending more on surface temperature and a_w value, but could be minimized by controlling the surface temperature.

Higher temperatures and a_w values significantly reduced the numbers of *B. cereus* spores. A temperature increase from 90 to 95°C had a larger impact on inactivation than did an increase from 95 to 100°C. Paprika powder with an a_w value of 0.88 and heated to product temperatures of 95–100°C displayed a reduction in *B. cereus* spore concentration of 4.5 $\log_{10}$ CFU/g within 6 min. The final spore concentration remained at levels of approximately 2 $\log_{10}$ CFU/g due to tailing. However, even at an a_w value of 0.84, a reduction of 2–3 $\log_{10}$ CFU/g was achieved at 95–100°C after 9 min of heating. Lowering the pH to 4.0 did not result in a significantly greater reduction in the concentration of *B. cereus* spores than occurred at pH 4.5.

The decontaminated powder was finally dried to a_w 0.50 by placing it in an adapted open heating unit and mixing it. Use of a thinner powder bed thickness facilitated fast drying within 4 min of heating (i.e. 2 × 2 min). Heating resulted in a gradient between the surface and overall a_w values, which could be eliminated by subsequent powder mixing. Final drying subsequent to IR decontamination did not affect the colour value as well as the level of *B. cereus*.

5.3 Closed IR heating unit 3 using a plastic pouch

5.3.1 IR heating system

Using closed heating unit 2 for the IR decontamination of paprika powder wetted to a_w 0.88 produced a significant reduction in the *B. cereus* spore load by 4.5 $\log_{10}$ CFU/g after 6 min at 100°C. However, due to small gaps between top lid and the insulation, water was able to evaporate during IR heating. If this leakage could be eliminated, the product a_w could probably be reduced to a degree where a similar microbial reduction rate of 4.5 $\log_{10}$ CFU/g could be achieved. Regarding the handling of paprika powder, microbial recontamination could occur in subsequent decontamination steps, such as during packaging and storage. Closed heating unit 3 was thus developed to facilitate both the decontamination and the subsequent handling of paprika powder without recontamination, by using a sealed near-IR-transparent plastic pouch (Fig. 5.20).

In this study, paprika powder was placed in a 8 × 11-cm pouch and sealed using an impulse sealing apparatus (model QS T 300 C, Maskin-Lindell AB, Kista, Sweden). The pouch was of PET plastic material (Maskin-Lindell AB, Kista, Sweden) stable up to temperatures of 121°C; the material was 90 µm thick, and its flexibility facilitated easy powder handling.

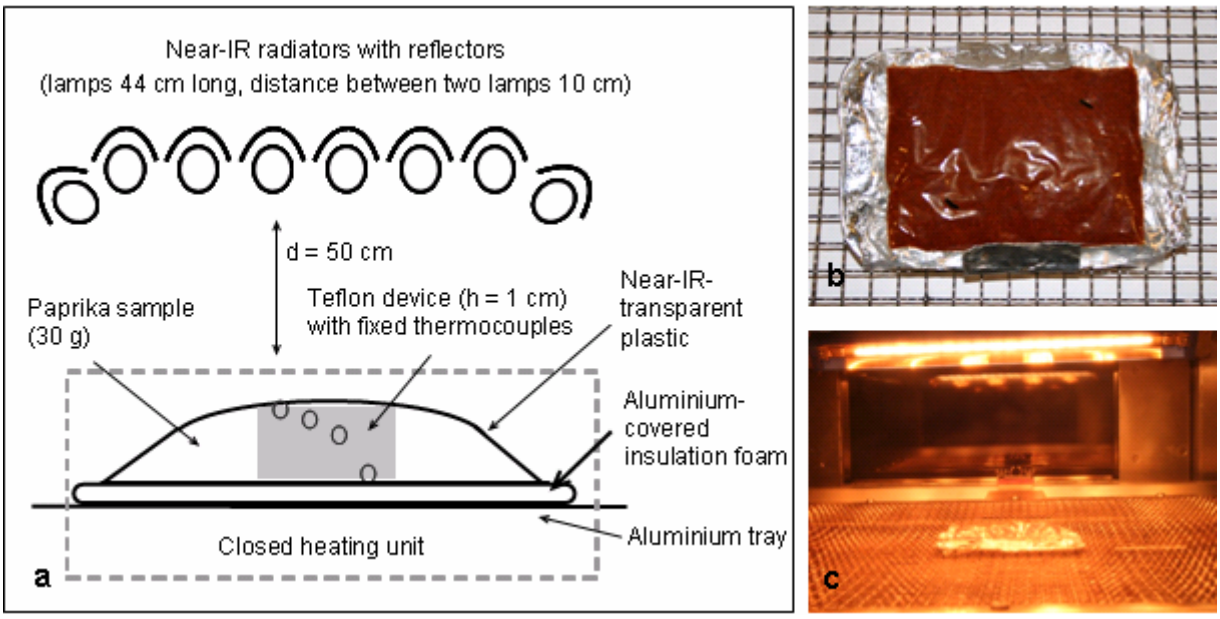

Figure 5.20: Schematic cross-section of the IR heating chamber and closed heating unit 3 (a). Top view of closed heating unit 3 with IR-transparent plastic material, the size of the pouch was 8 × 11 cm (b); and side view of closed heating unit 3 during near-IR heating (c).

The thickness of the powder in the pouch was approximately 10 mm at its thickest, but declined towards the edge of the pouch (Fig. 5.20 a). During the warm-up period, two small cuts (indicated in black on the plastic pouch in Fig 5.21 a) had to be made on the upper surface of the plastic pouch using a sterile scalpel. That was necessary to allow water steam escape from the system during the warm-up period, as steam requires a larger volume when heated (i.e. the "pressure cooker" effect). Otherwise, the plastic material would have started to stretch, possibly bursting the seal. After the warm-up period, the two cuts were sealed using sterile plastic tape (see Fig. 5.21 b), and powder was kept at desired product temperature of 100°C for a certain time.

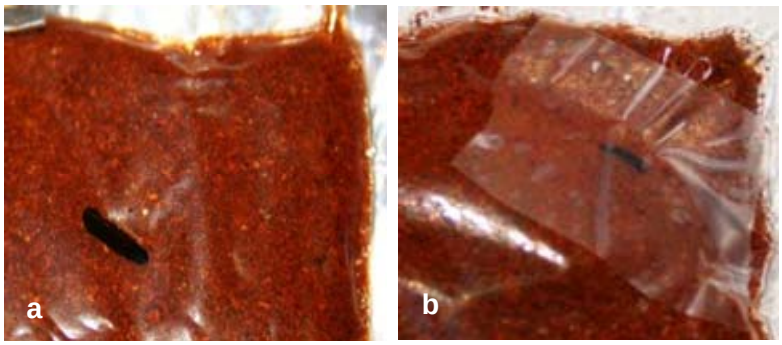

Figure 5.21: Indicated position of a single cut in the plastic pouch before IR heating (a), and the single cut sealed with sterile tape after the IR warm-up period (b).

5.3.1.1 Heat fluxes through IR-transparent plastic

The near-IR output powers and respective calculated heat fluxes for the plastic material are shown in Table 5.7. The calculated transparency factors of the material to selected near-IR heat fluxes were 92–99%. Since no product specification was given for the IR transparency of the plastic material, the transparency factor was compared with the given transparency of the near-IR-transparent glass, used in heating unit 2, which was 90%. The tested plastic material was also considered highly transparent, so the pouches are called "near-IR-transparent plastic pouches".

Table 5.7: Selected IR output powers and their corresponding near-IR heat fluxes obtained with and without use of a near-IR-transparent plastic material between the IR source and the black body; with the IR transparency factor for plastic

Output power	Heat flux (kW/m^2)		IR transparency factor (%)
	Without IR plastic	With IR plastic	
100%	23.4 ± 0.26	21.9 ± 0.07	94
25%	11 ± 0.22	10.6 ± 0.39	96
15%	8.8 ± 0.34	8.1 ± 0.26	92
1%	4.0 ±0.42	4.0 ± 0.24	99

5.3.1.2 Experimental design

Paprika powder with initial a_w values of 0.72, 0.76, 0.80, 0.84, and 0.88, placed in sealed near-IR-transparent plastic pouches, was heated to a product temperature of 100°C using variable near-IR heat fluxes. The concentration of spore of *B. cereus* was determined before, during, and after IR heating; four replicates were made of each combination. Temperature profiles, product a_w value, and colour were assessed for all initial a_w values, except a_w 0.88. The total IR process time was monitored and consisted of warm-up (from 20°C up to 100°C product temperature) and holding/inactivation periods (starting when the 95–100°C product temperature was measured at a depth of 8 mm in the powder bed).

Experiments were performed using variable heat fluxes, as shown in Table 5.8. The total IR process time consisted of a fast (phase I) and two moderate (phases II and III) warm-up periods totalling 4 min in duration, followed by a holding period at a product temperature of 100°C (phase IV). In phase IV, IR was applied in pulses (turning the near-IR radiators off and on) to keep the product temperature constant.

Table 5.8: Holding times under variable near-IR heat fluxes needed to heat paprika powder (a_w 0.84, placed in sealed plastic pouches) during warm-up periods to reach a product temperature of 100°C (phases I, II, and III) and the on/off times (pulsed IR operation) needed to maintain the desired product temperature (phase IV)

Phase	Near-IR heat flux (kW/m^2)	Time (s)
I	22	30
II	10.5	60
III	8	150
IV	4	120/60

5.3.2 Temperature profile

The measured temperature profile at an a_w value of 0.84 for a product temperature of 100°C, obtained after heating with variable near IR, is shown in Fig. 5.22. The profiles at a_w values of 0.72, 0.76, 0.80, and 0.88 displayed the same behaviour and are therefore not shown.

The high heat flux of phase I and moderate heat flux of phase II caused large temperature differences within the powder bed of 40 and 50°C, respectively, due to high heat absorption

on the surface and limited heat conduction within the powder bed (Ginzburg, 1969). During phase II, however, the rate of surface temperature increase was already slowing; the surface temperature continued increasing, but more slowly, in phase III, heating the entire powder bed, mainly by conduction. Approaching phase IV, the surface–interior temperature difference decreased to 15°C and remained at 10°C for the duration of phase IV.

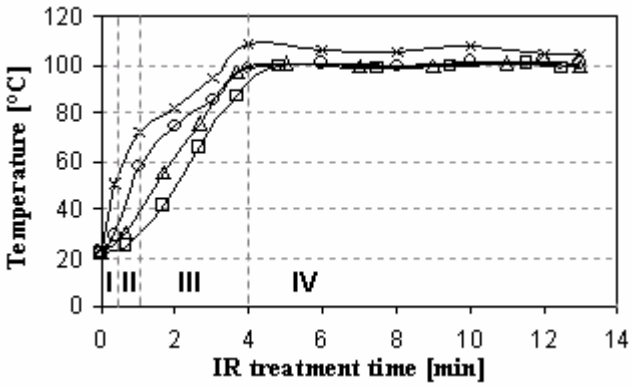

Figure 5.22: Temperature of paprika powder measured on the surface (+) and at depths of 1 (○), 3 (Δ), and 8 mm (□) at an a_w value of 0.84 during heating to 100°C at a depth of 8 mm with variable near-IR heat fluxes of 22 (phase I), 10.5 (II), and 8 kW/m^2 (III), and at a holding temperature of 100°C at a near-IR heat flux of 4.0 kW/m^2 (IV, pulsed operation).

5.3.3 Effect of heating on a_w and total water loss

Surface and overall a_w and total water loss were studied for paprika powder with initial a_w values of 0.72, 0.76, 0.80, and 0.84 during heating using variable near IR (Fig. 5.23). Overall a_w values remained constant throughout the heating. Surface a_w decreased by 10–15% during the warm-up period (phase I), but remained similar to the overall a_w during the holding period (phase II). The maximum total water loss was 2%; most water loss was observed during the warm-up period, when water was able to escape from the heating system through two small cuts in the plastic pouch. Due to closed environment during the holding period the water loss was negligible, but might be existing due to tiny holes in the seal or to softening of the plastic material caused by IR exposure.

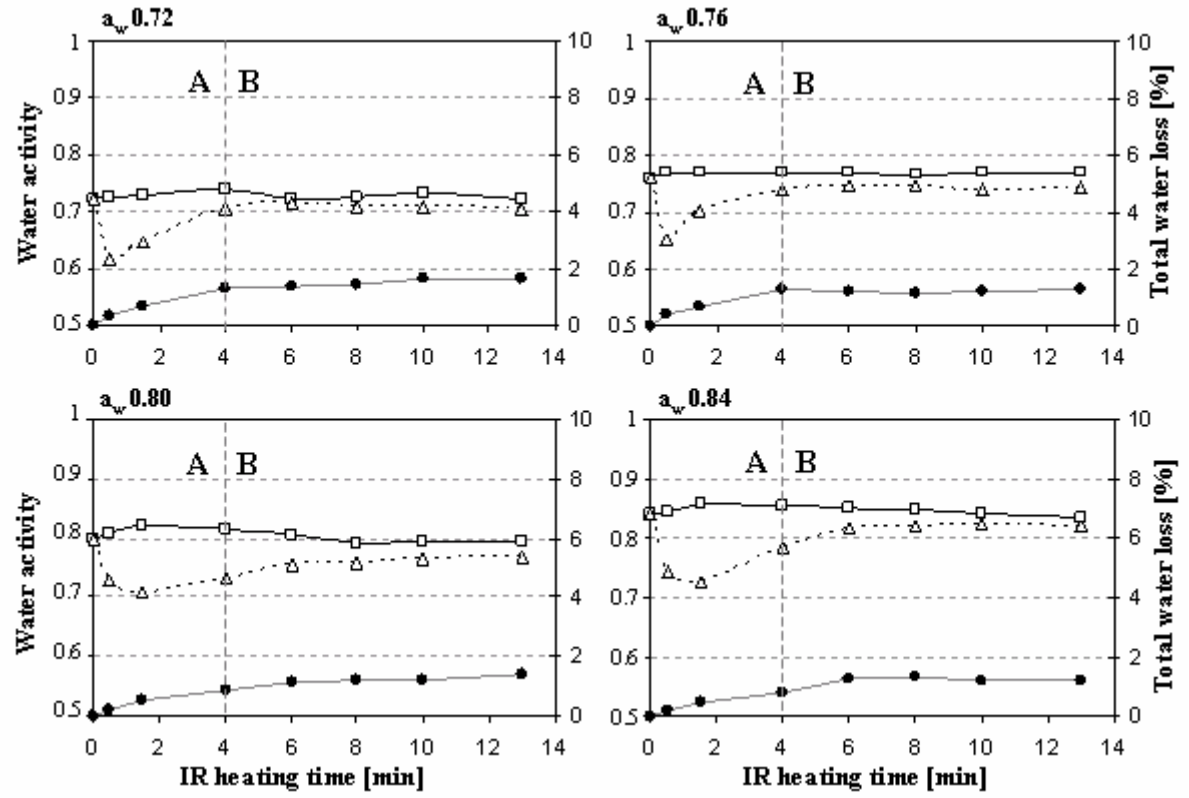

Figure 5.23: Surface (Δ) and overall (□) a_w and total water loss (●) in paprika powder with selected initial a_w values, placed in sealed pouches during near-IR heating with a warm-up (A) and holding period (B).

5.3.4 Reduction of *B. cereus* spore concentration

The aim of this part of the research was to optimize the reduction of spores of *B. cereus*, SIK 340, found in paprika powder placed in a sealed pouch, by finding the ideal a_w value for a given heating unit. The first microbial reduction tests used paprika powder wetted to a_w values of 0.72, 0.80, and 0.88 during near-IR heating at a product temperature of 100°C (Fig. 5.24). At all tested a_w values, there was a significant reduction in the number of *B. cereus* spores from 7 to approximately 3 $\log_{10}$ CFU/g within 6, 3, and 2 min for a_w values of 0.72, 0.80, and 0.88, respectively. The *D*-values for the linear part of the reduction were 2, 1, and 0.75 min, respectively. The effect of the warm-up period (A in Fig. 5.24) on microbial reduction was negligible at a_w 0.72, but considerable at a_w values of 0.80 and 0.88, being 1 and 2 $\log_{10}$ CFU/g, respectively.

An a_w value of 0.80 is said to limit the germination of *B. cereus* (Hagen et al., 1967). As shown above, microbial reduction was possible even at an initial a_w of 0.72, because heating conditions prevented the evaporation of water from the heating system. However, the

achieved *D*-value was 2 min, which is notably higher, compared with 1 min at a_w 0.80. Thus further optimization was done using initial a_w values of 0.76 and 0.84, i.e. below and above the limits for *B. cereus* spore germination (i.e. a_w 0.80).

The reduction in the *B. cereus* plate counts at a_w values of 0.76 and 0.84 is shown in Fig. 5.25. As expected, at a_w 0.84 a higher reduction rate could be achieved than at a_w 0.76. A linear reduction from 7 to 3 $\log_{10}$ CFU/g was observed within holding times of 2 and 4 min for a_w 0.84 and 0.76, respectively. Further heating caused remaining spore concentrations of around 2-3 $\log_{10}$ CFU/g at both tested a_w values. That was due to lowered a_w on the surface causing increased heat resistance of the spores (Jeng & Woodworth, 1990; Ababouch et al., 1995, Leguerinel et al., 2004). That could not been compensated by higher ambient surface temperatures. It should be noted that after 6 min of holding time (period B in Fig 5.25), the same remaining spore concentration, namely, 2 $\log_{10}$ CFU/g, was observed for both a_w values.

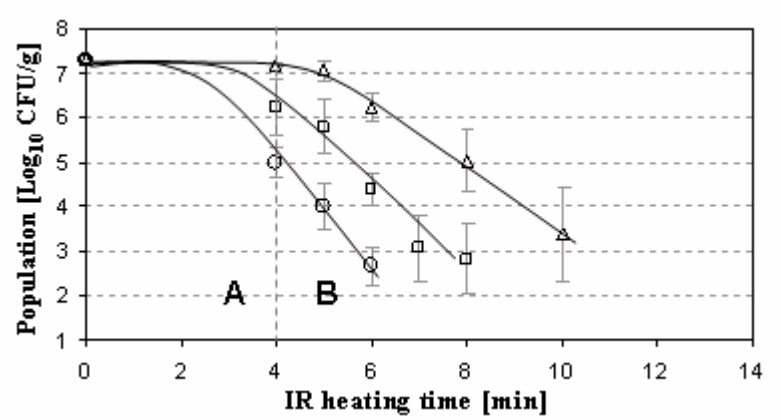

Figure 5.24: Total plate counts of *B. cereus*, SIK 340, in paprika powder with a_w values of 0.72 (Δ), 0.80 (□), and 0.88 (○) after heating with near-IR, with a warm-up period (A) and holding times at a product temperature of 100°C (B). The concentration before heating was 7.23 $\log_{10}$ *B. cereus* spores/g and the detection limit was 1 $\log_{10}$ CFU/g.

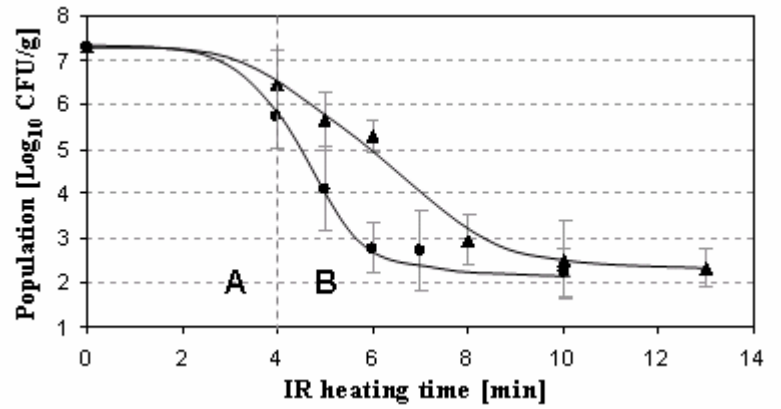

Figure 5.25: Total plate counts of *B. cereus*, SIK 340, in paprika powder with a_w values of 0.76 (▲) and 0.84 (●) after heating with near-IR, with a warm-up period (phase A) and holding times at a product temperature of 100°C (B). The concentration before heating was 7.23 $\log_{10}$ *B. cereus* spores/g and the detection limit was 1 $\log_{10}$ CFU/g.

5.3.5 Effect of heating on colour

Surface and overall colour values were measured for paprika powder with initial a_w values of 0.72, 0.76, 0.80, and 0.84 (Fig. 5.26). Colour was expressed as the product of the *L* (lightness) and *a* (redness) colour parameters (i.e. $a \times L$), values >500, 500–300, and <300 being rated as red, medium red, and dark, respectively (Ramakrishnan & Francis, 1973). Initial colour values were 870, 840, 790, and 750 at the respective a_w values. As expected, colour values decreased significantly during the warm-up periods and remained fairly constant during the holding period; the surface colour values were 500–600 at all tested a_w values except 0.84, at which the colour values were 450–500. Though the overall colour value was less affected by heating than the surface value was, it still decreased significantly during warm-up, due to temperatures over 60°C (Malchev et al., 1982); the overall colour value remained constant at approximately 600–700 at all a_w values except 0.84, at which it was 550–600, which is rated in the red range.

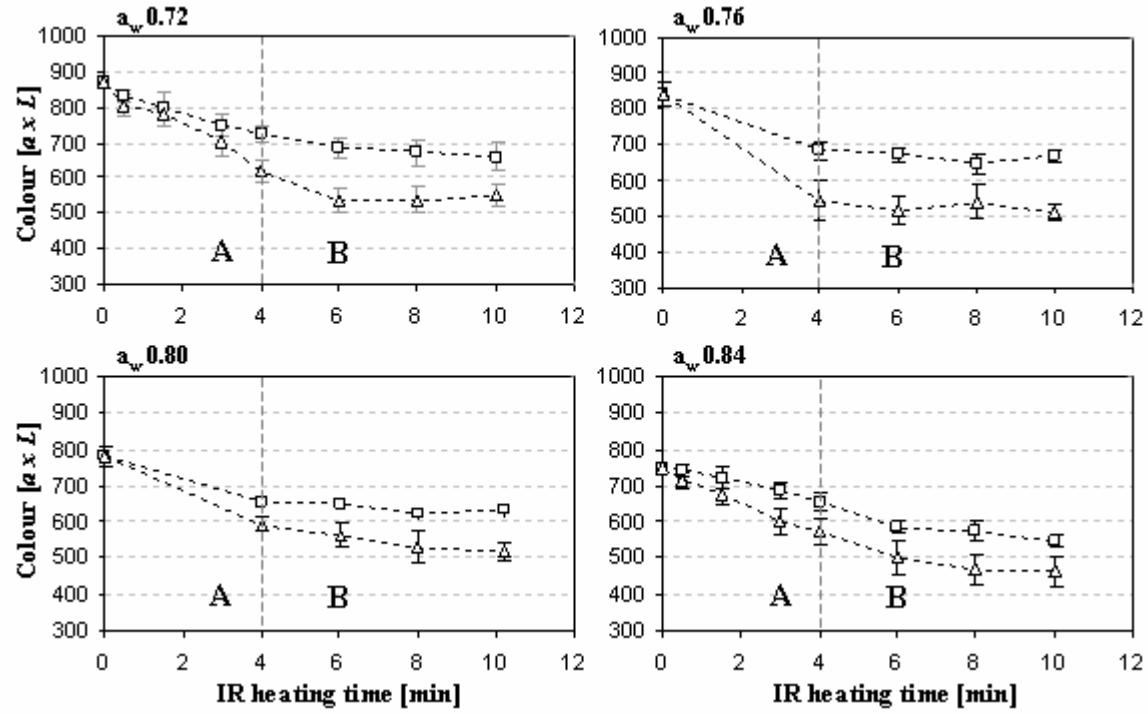

Figure 5.26: Surface (Δ) and overall colour (□) of paprika powder with selected a_w values exposed to variable near-IR heat fluxes of 22, 10.5, and 8 kW/m^2 while warming up to a 100°C product temperature (A), and exposed to a near-IR heat flux of 4.0 kW/m^2 during the holding period at 100°C (B).

5.3.6 Summarized process observations for closed heating unit 3

Closed heating unit 3 was a sealed near-IR-transparent plastic pouch with a transparency of more than 90%. A plastic pouch facilitates easy handling of paprika powder even after the decontamination step, due to the flexibility of the plastic from which it is made. Using the sealed pouch, paprika powder was successful decontaminated even at a_w values below that required for the germination of *B. cereus* spores (i.e., a_w 0.80), as water evaporation was prevented during IR heating.

Overall colour decreased significantly during the warm-up, declining from initial values of 870–750 to 700–600 at all a_w values except 0.84, at which it declined to 600–550, but remained constant throughout the holding period. Overall a_w remained constant, and the decrease of surface a_w, particularly during the warm-up, could be reduced. Total water loss was marginal, reaching a maximum of 2%, most of the loss occurring during the warm-up.

A significant reduction of *B. cereus* numbers, i.e. of 4 $\log_{10}$ CFU/g, was achieved within 2–6 min for a_w values of 0.84–0.76, respectively. A_w values higher than 0.84 resulted in no further reduction in *D*-value, and a_w values lower than 0.76 required longer heating times to obtain a final spore concentration of 3 $\log_{10}$ CFU/g. Reduction treatments to below spore concentrations of 3 $\log_{10}$ CFU/g caused tailing of approximately 2 $\log_{10}$ CFU/g and are thus inefficient. The effect of heating on microbial reduction was already notably for a_w 0.80 and higher during the warm-up period.

5.4 Summary of results obtained from the IR heating units

Heating unit		
1	**2**	**3**
IR heating assembly		

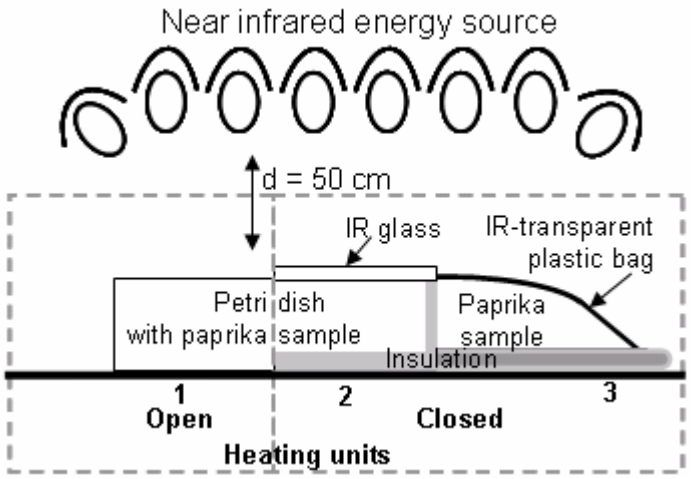

Figure 5.27: Schematic summary of IR heating units used for decontaminating paprika powder.

No prevention of water evaporation	Prevention of water evaporation; use of insulation IR transparency factor of glass and plastic material over 90%

IR Process

Constant IR heat flux | Variable IR heat flux

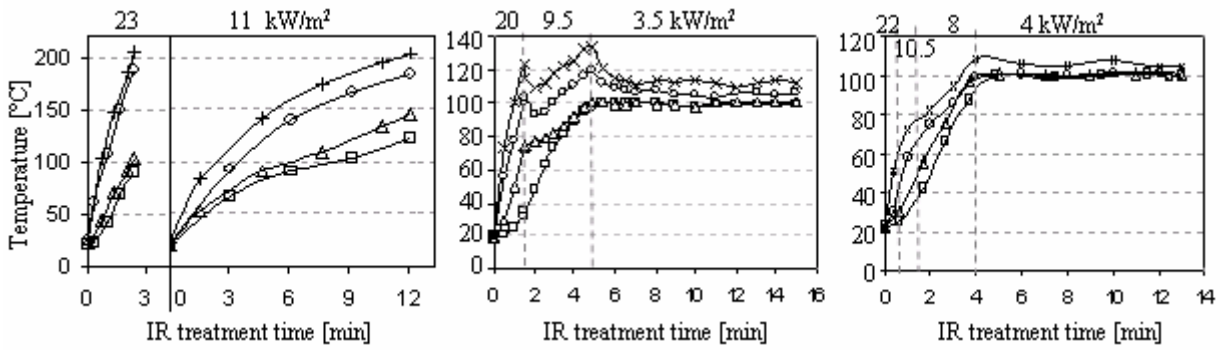

Figure 5.28: Temperature profiles for paprika powder with an a_w value of 0.84 measured on the surface (×), and at depths of 1 (○), 3 (Δ), and 8 mm (□) during heating with near-IR heat fluxes (kW/m^2).

Temperature

▪ Constant temperature increase, resulting in undesired high surface temperature ▪ The higher the heat flux, the faster the heating	▪ Controlled temperature increase to maximum surface temperatures of 120–130°C achieved by applying different heat fluxes ▪ Combination of high/moderate heat flux to reach product temperature of 100°C, and then low heat flux to hold the desired product temperature

Temperature gradient during heating

Increasing	Decreasing

Microbiology

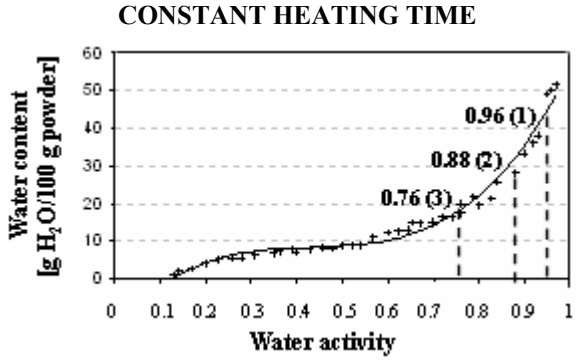

Figure 5.29: A_w value and corresponding water content of paprika powder required to reduce spores of *B. cereus* from 7 to approximately 3 $\log_{10}$ CFU/g during near-IR heating for 6 min at a product temperature of 100°C using heating units (1), (2) and (3).

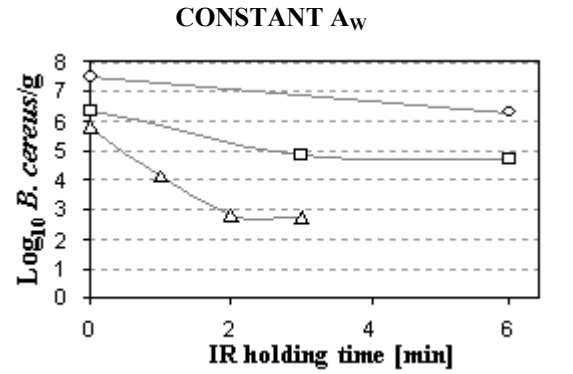

Figure 5.30: Obtained reduction of spores of *B. cereus* in paprika with a_w values of 0.80–0.84 during near-IR heating at product temperatures of 100°C using heating units 1 (○), 2 (□) and 3 (Δ). Initial population was 7.23-7.48 CFU/g; lower populations at t_0 were due to the warm-up period needed to raise the temperature from 20 to 100°C.

Water loss

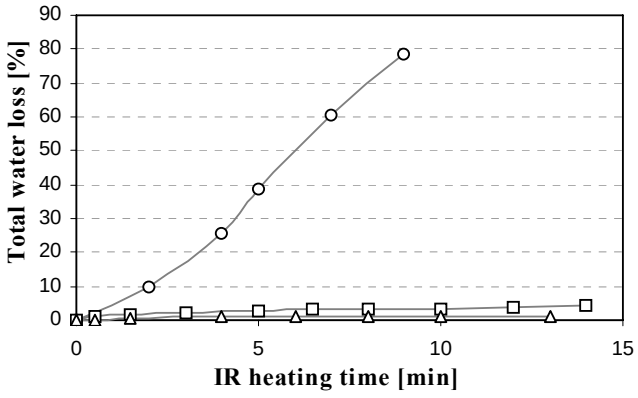

Figure 5.31: Total water loss from paprika powder with an a_w value of 0.80–0.84 during near-IR heating using heating units 1 (○), 2 (□) and 3 (Δ).

- Water was effectively removed from the powder using the open heating unit (1)
- Negligible evaporation of water of 4 and 2% achieved using heating units (2) and (3), respectively

Water activity

SURFACE

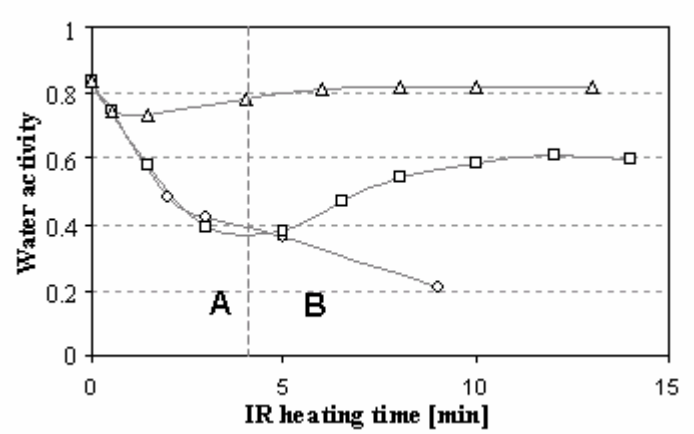

Figure 5.32: Surface a_w in paprika powder with an initial overall a_w value of 0.80–0.84 during the warm-up period (A) and while holding the temperature of 95-100°C (B) by near-IR heating using heating units 1 (○), 2 (□) and 3 (Δ).

- Decrease of surface a_w during warm-up was significant with heating units (1) and (2)
- A constant surface a_w decrease was noted with (1)
- Surface a_w increased with (2) and (3) due to constant product temperature and low applied heat fluxes
- A lowered surface a_w was noted during the holding time with (2) due to small gaps in the heating unit allowing evaporation

OVERALL

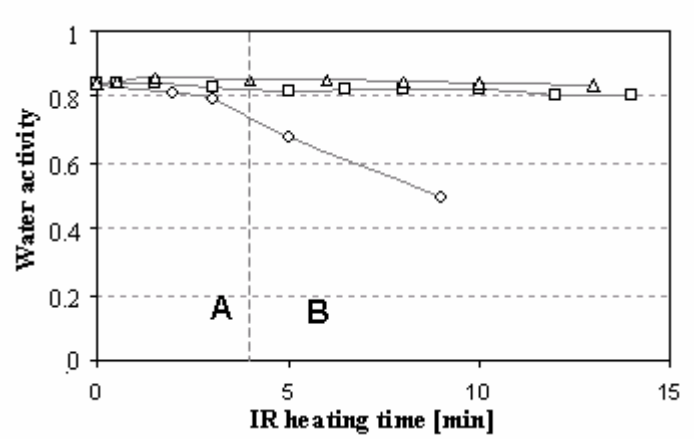

Figure 5.33: Overall a_w value in paprika powder with an initial a_w value of 0.80–0.84 during the warm-up period (A) and while holding the temperature of 95-100°C (B) by near-IR heating using heating units 1 (○), 2 (□) and 3 (Δ).

- Decrease of overall a_w occurred with heating unit (1)
- Constant overall a_w was achieved with heating units (2) and (3) due to prevention of water evaporation

Colour

SURFACE

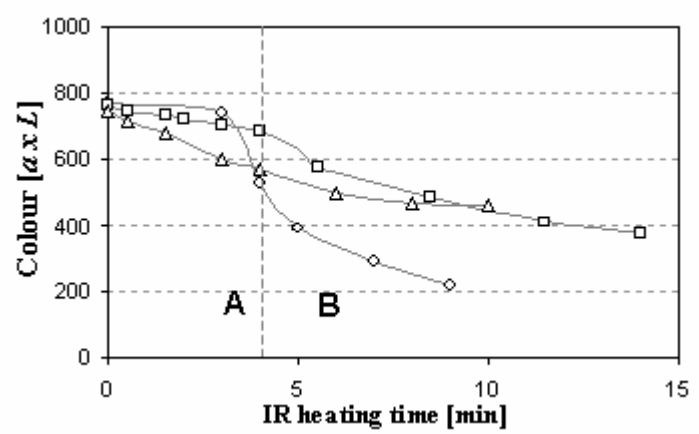

Figure 5.34: Surface colour of paprika powder with an a_w value of 0.80–0.84 during the warm-up period (A) and while holding the temperature of 95-100°C (B) by near-IR heating using heating units 1 (○), 2 (□) and 3 (Δ).

- Constant decrease of surface colour, i.e. carotenoid degradation at higher temperatures
- Higher surface colour with use of heating units (1) and (2), due to higher colour values at lower a_w values (drying effect)
- With use of (2) and (3) colour remained in the medium-red range, while use of (1) caused a decrease to the dark colour range (<300)

OVERALL

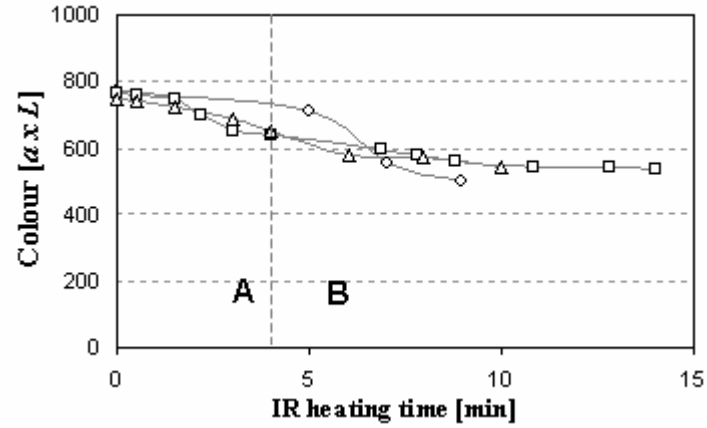

Figure 5.35: Overall colour of paprika powder at an a_w value of 0.80–0.84 during the warm-up period (A) and while holding the temperature of 95-100°C (B) by near-IR heating using heating units 1 (○), 2 (□) and 3 (Δ).

- Constant decrease of overall colour, i.e. carotenoid degradation, occurred at temperatures over 60°C
- The remaining overall colour for all heating units was approximately 550–600, i.e. red colour range

6 Food applications of IR-decontaminated paprika powder

This chapter examines possible applications of interest to the food industry in terms of subsequent use of the IR decontaminated paprika powder:

(1) storage of moderate wetted decontaminated powder (a_w 0.76 and 0.84) in a sealed plastic pouch and
(2) use of decontaminated powder (a_w 0.84) by mixing into crème fraîche and spiking onto the surface of pork tenderloin.

Paprika powder was decontaminated using near-IR heating and heating unit 3, i.e. within sealed plastic pouches. The processing parameters for IR decontamination were taken from Table 5.8.

6.1 Storage of IR-decontaminated powder in sealed plastic pouches

It is recommended that paprika powder be stored at a_w of 0.3–0.5; long-term storage at a_w values over 0.70 and relative humidity levels over 82% should be avoided, due to the possibility of microbial growth, especially of moulds (Kim et al., 1984; Lee et al., 1991). A product temperature of 100°C applied during IR decontamination eliminates moulds and other vegetative cells (Gerhardt, 1974). However, spores of *B. cereus* can germinate at a_w 0.80 and grow at a_w 0.90 (Hagen et al., 1967). Thus spores may be a critical microbial quality factor affecting the storage of wetted decontaminated powder. Studying the reduction of *B. cereus* spores with IR (using heating unit 3) indicated that at a_w values lower than 0.80, significant spore reduction was obtained (see section 5.3). Another critical factor affecting storage is that paprika powder loses part of its pigmentation during storage; the degree of loss is mainly affected by duration and temperature of storage, oxygen level, light, moisture content, and kind of packaging (Malchev et al., 1982). The amount of water added to the powder before

decontamination is thus critical for the attainment of fast microbial reduction and for colour stability during storage.

In the present work, IR heat treatment and the subsequent storage of the wetted decontaminated paprika powder was done by placing the powder in a sealed IR-transparent plastic pouch, i.e. in a closed environment (for more detailed description, see section 5.3). In this way, the risk of recontamination after treatment was eliminated. The flexible properties of the plastic material facilitate the transportation, storage, and handling of the powder packages. *B. cereus*-inoculated paprika powder was used in studying the effects of storing IR-decontaminated paprika powder, in terms of changes in overall colour and a_w and in volatile content.

Paprika powder was wetted to a_w values of 0.76 and 0.84, which are below and above the germination point, respectively. Heating with IR was done using heating unit 3 for holding times of 6 and 9 min for a_w 0.76, and 2 and 6 min for a_w 0.84. After heating and cool down, the pouch was shaken by hand to achieve a homogeneous powder mixture, i.e. to eliminate a_w and colour differences within the powder sample, and stored at 20°C without light exposure.

6.1.1 Effect on microbial numbers

The level of *B. cereus* during the storage of decontaminated paprika powder in sealed plastic pouches at 20°C was determined for paprika with a_w values of 0.76 (Fig. 6.1) and 0.84 (Fig. 6.2).

During IR decontamination of paprika powder having an a_w value of 0.76, counts of *B. cereus* decreased from 7.23 to 2.6 and 1.9 $\log_{10}$ CFU/g after 6 and 9 min, respectively. Subsequent storage for up to 16 weeks produced no further change in the level of *B cereus*, i.e. no growth of spores below the germination point of *B. cereus*.

Paprika powder having an a_w value of 0.84 and decontaminated for IR holding times of 2 and 6 min displayed a reduction of *B. cereus* concentration from 7.23 to approximately 2.0 $\log_{10}$ CFU/g. Storage for up to 4 weeks produced an increase in spore concentration of 1 $\log_{10}$ CFU/g. This slight increase in *B. cereus* spore concentration was probably caused by the recovery of heat-damaged spores or outgrowth of some of the spores. However, no further growth was noted up to 16 weeks of storage, rather a decrease of 1 $\log_{10}$ CFU/g to the initial spore concentration of 3 $\log_{10}$ CFU/g.

The final microbial number obtained in both powders during storage of 4 months, i.e. 2–3.5 $\log_{10}$ CFU/g, was below the acceptable level for spices, i.e. 4 $\log_{10}$ CFU/g (Shelef, 1983). Comparing the holding times used in the decontamination treatments indicated that increasing the holding time from 2 to 6 min (a_w 0.84) and from 6 to 9 min (a_w 0.76) did not produce a significant additional improvement in the microbial quality, so the shorter IR heating times should be preferred.

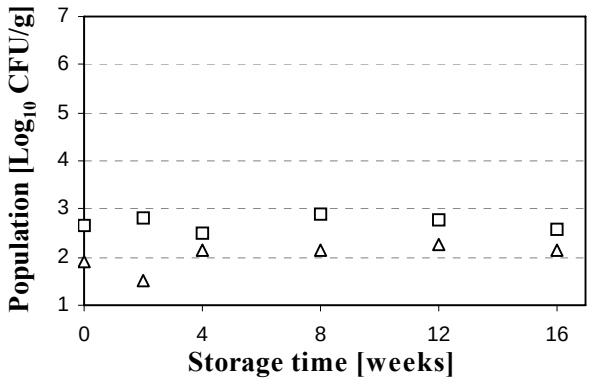

Figure 6.1: Storage of paprika powder (a_w 0.76) in sealed plastic pouches at 20°C and the effect on total plate counts of *B. cereus*, SIK 340, after heat treatment with near-IR at a product temperature of 100°C for holding times of 6 min (□) and 9 min (Δ).

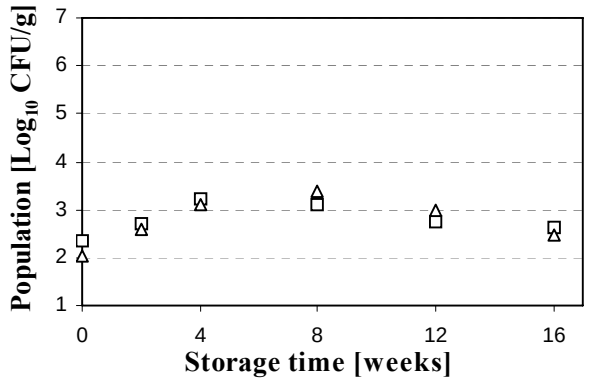

Figure 6.2: Storage of paprika powder (a_w 0.84) in sealed plastic pouches at 20°C and the effect on total plate counts of *B. cereus*, SIK 340, after heat treatment with near-IR at a product temperature of 100°C for holding times of 2 min (□) and 6 min (Δ).

6.1.2 Effect on overall colour and a_w

Initially wetted powder had colour values of 817 and 720 at a_w values of 0.76 and 0.84, respectively. After IR heat treatment, overall colour values were, for a_w 0.76 powder, 650 and 610 after 6 and 9 min of holding time, respectively, and, for a_w 0.84 powder, 620 and 540 after 2 and 6 min (Fig. 6.3). The overall colour differences between the two a_w values

disappeared during storage. Thus the overall colour of a_w 0.76 powder remained in the 600–650 range, while the colour of a_w 0.84 powder decreased significantly to 530 after one week of storage for both tested holding times. However, even after further storage, the colour value remained between 550 and 500, which is on an acceptable for paprika powder. An visual expression of the stored powder after 16 weeks is given in Fig. 6.4.

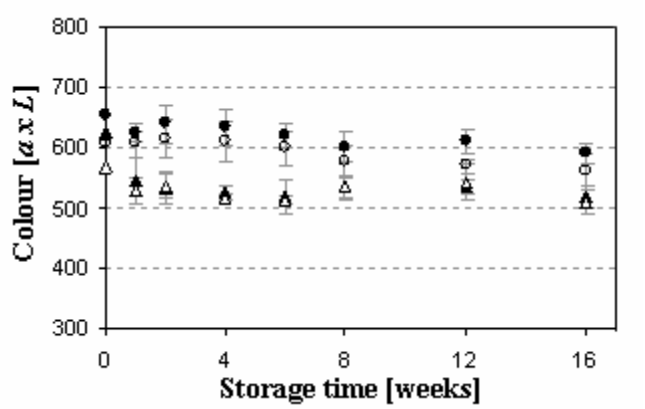

Figure 6.3: Overall colour of decontaminated paprika powder with a_w values of 0.76 and 0.84 during storage in sealed plastic pouches at 20°C. Near-IR heat treatment was done at product temperature of 100°C and for holding times of 6 (●) and 9 min (○) for a_w 0.76 and 2 (▲) and 6 min (Δ) for a_w 0.84.

Figure 6.4: Image of overall colour of paprika powder after 16 weeks of storage at 20°C at a_w 0.76 (1), and 0.84 (2); and paprika powder at a_w 0.50 (3).

The overall a_w value was evaluated for paprika powder with initial a_w values of 0.76 and 0.84 after storage for up to 16 weeks (Fig. 6.5). Under all testing conditions, the a_w value remaining constant for the entire storage period, due to the sealed plastic barrier.

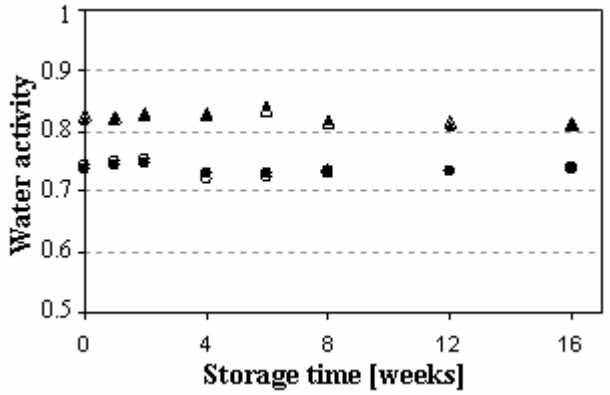

Figure 6.5: Overall a_w of decontaminated paprika powder with a_w values of 0.76 and 0.84 during storage in sealed plastic pouches at 20°C. Near-IR heat treatment was done at product temperature of 100°C and for holding times of 6 (●) and 9 min (○) for a_w 0.76 and 2 (▲) and 6 min (Δ) for a_w 0.84.

6.1.3 Effect on volatiles

Volatile components of treated and untreated paprika powder were analysed before and after heating and after storage at 20°C, using dynamic headspace GC (Fig. 6.6–6.9).

Untreated paprika powder contains no volatile constituents in significant amounts (Fig. 6.6), so untreated, wetted paprika can be considered rather weak in volatiles (Wilkins, 1994). Likely, the hexanal (an off-flavour component formed as an oxidation product of unsaturated fatty acids) found in unheated paprika powder comes from earlier drying of the powder. Hexanal can be used as a marker for identifying the heating of paprika (Cremer & Eichner, 2000a).

Heating paprika (Fig. 6.7) increases the amounts of acetaldehyde, 2-methylpropanal, 2-methylbutanal, and 3-methylbutanal – regularly found in the low-boiling fraction of volatile compounds of processed plant foods – give an odour impression of cacao, spicy and sweaty. These components are typical Strecker aldehydes, indicating that the Maillard reaction has occurred during processing operations. The formation of Strecker aldehydes is not influenced by pH, but by the concentration of the corresponding amino acids, such as alanine, valine, isoleucine, and leucine. Furans and furfural contribute importantly to desirable aromas, because of their very low threshold values and their pleasant almond scent. Moreover, dimethyl sulphide (DMS) and 6-methyl-5hepten-2-one were identified. DMS is produced via the hydrolysis of *S*-methylmethionine (SMM), an amino acid typically found in many plants. The substance 6-methyl-5-hepten-2-one, well known as a degradation product of lycopene and carotenoids, easily contributes to off-flavours by giving a grassy, lettuce-like and fruity odour impression (Luning et al., 1995; Cremer & Eichner, 2000a–c; Ramirez & Cava, 2007).

In untreated stored paprika (Fig. 6.8), besides the Strecker-derived components, high amounts of ethanol (produced by microbial growth during storage) and acetone (produced by sugar and carotenoid degradation) were detected. In addition, 2-ketone was found; it is associated with the aroma of surface-riped cheese, which arises from fatty acids by means of the chemical or enzymatic oxidation of free-fatty acids by moulds (Ramirez & Cava, 2007).

Treated stored powder (Fig. 6.9) contained the same volatiles as were found in the sample analysed directly after IR heating. Ethanol was not detected in any significant amount, so microbial growth had been avoided during the five weeks of storage. However, as the powder was stored in air-tight glass flasks, no general statements can be made regarding the release of volatiles through the plastic material.

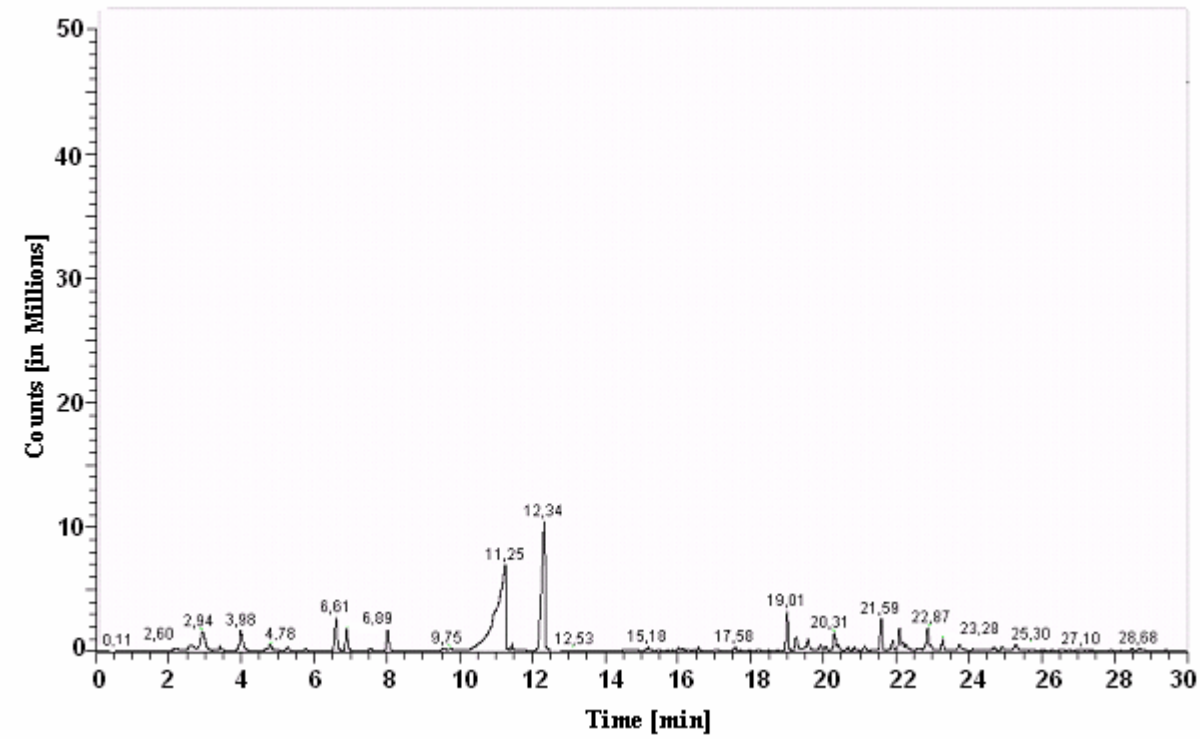

Figure 6.6: Dynamic headspace GC analysis of IR-untreated, wetted paprika powder (a_w 0.84): 2.60 ethanol, 2.94 acetone, 3.98 methyl-acetone, 4.78methyl-propanol, 6.61 3-methyl-butanol, and 12.34 hexanal.

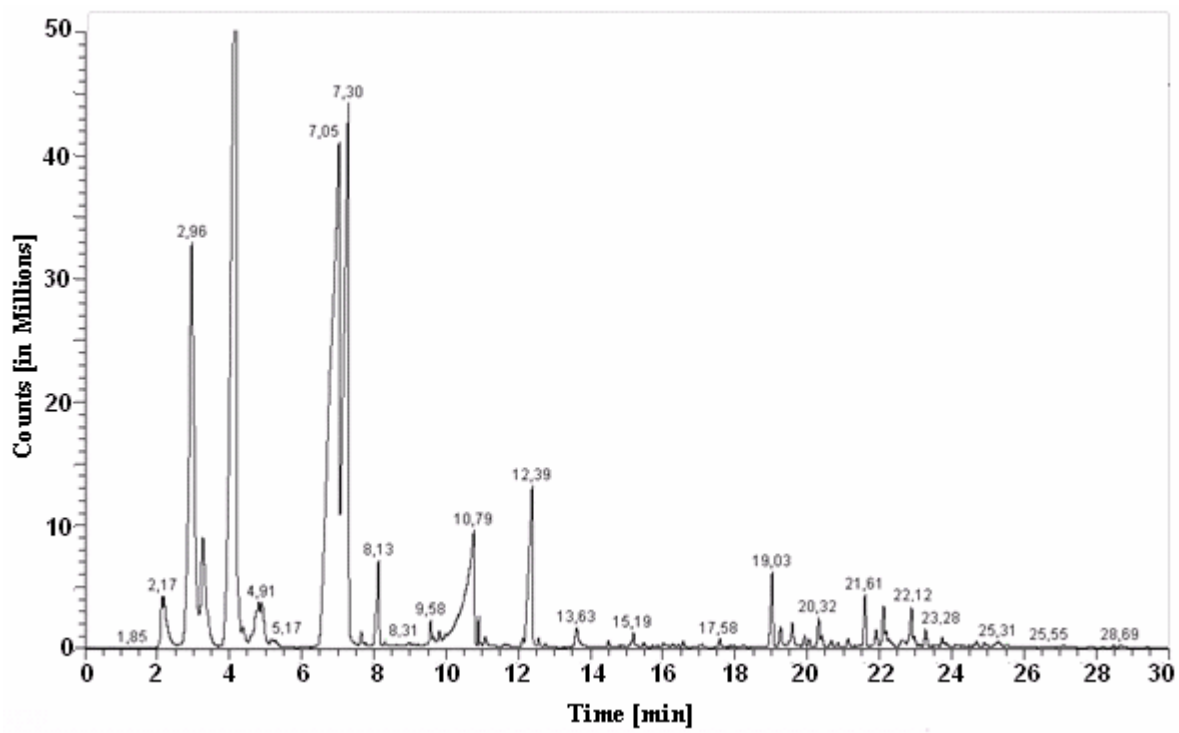

Figure 6.7: Dynamic headspace GC analysis of IR-treated, wetted paprika powder (a_w 0.84): 2.17 acetaldehyde, 2.96 acetone, 3 dimethyl sulphide, 4 methylpropanal, 4.91 Diacetyl and 2-methyl-furan, 7.05 3-methyl-butanal, 7.30 2-methyl-butanal, 8.13, pentanal, 12.39 hexanal, and 13.63 furfural.

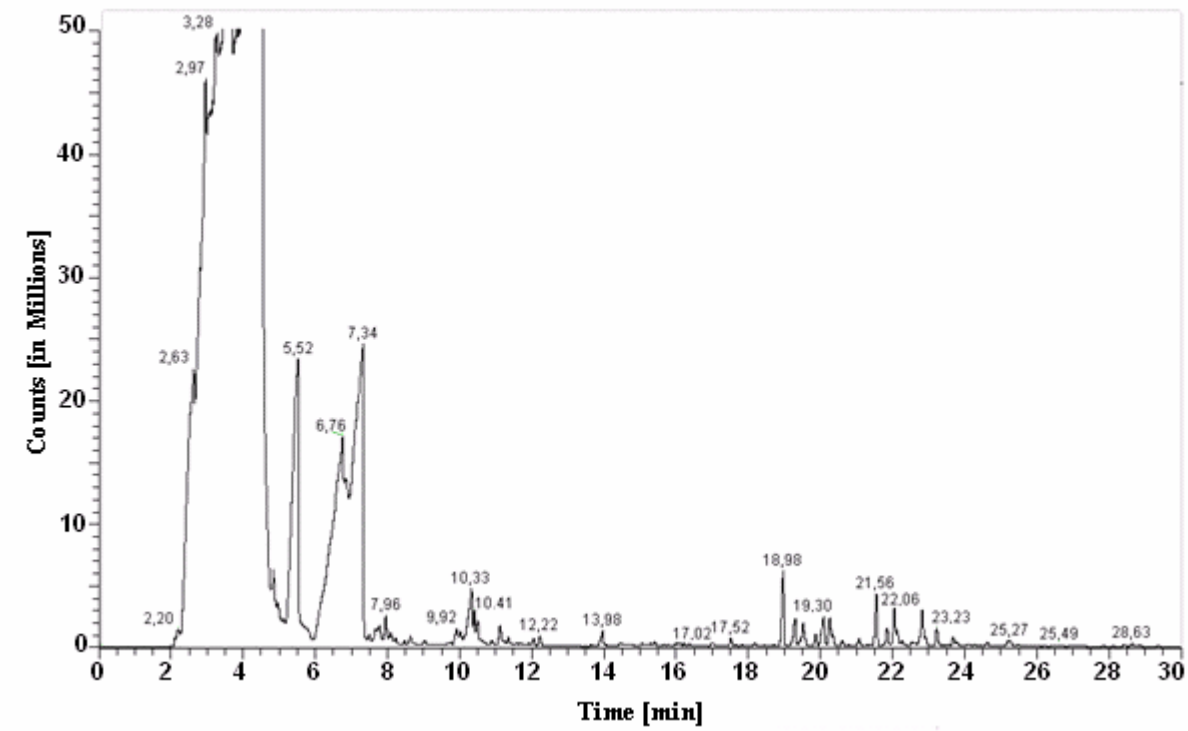

Figure 6.8: Dynamic headspace GC analysis of IR-untreated, wetted paprika powder (a_w 0.84) after storage for five weeks in closed glass flasks at 20°C: 2-5 ethanol, acetone, methyl-acetate, methyl-propanole, 7.34 2-methyl-propanole, 7.96 2-ketone, 10.33 3-methyl-butanole, 11.13 2-methyle-proyl-acetone, and 19.30 6-methyl-5 hepten-2-on.

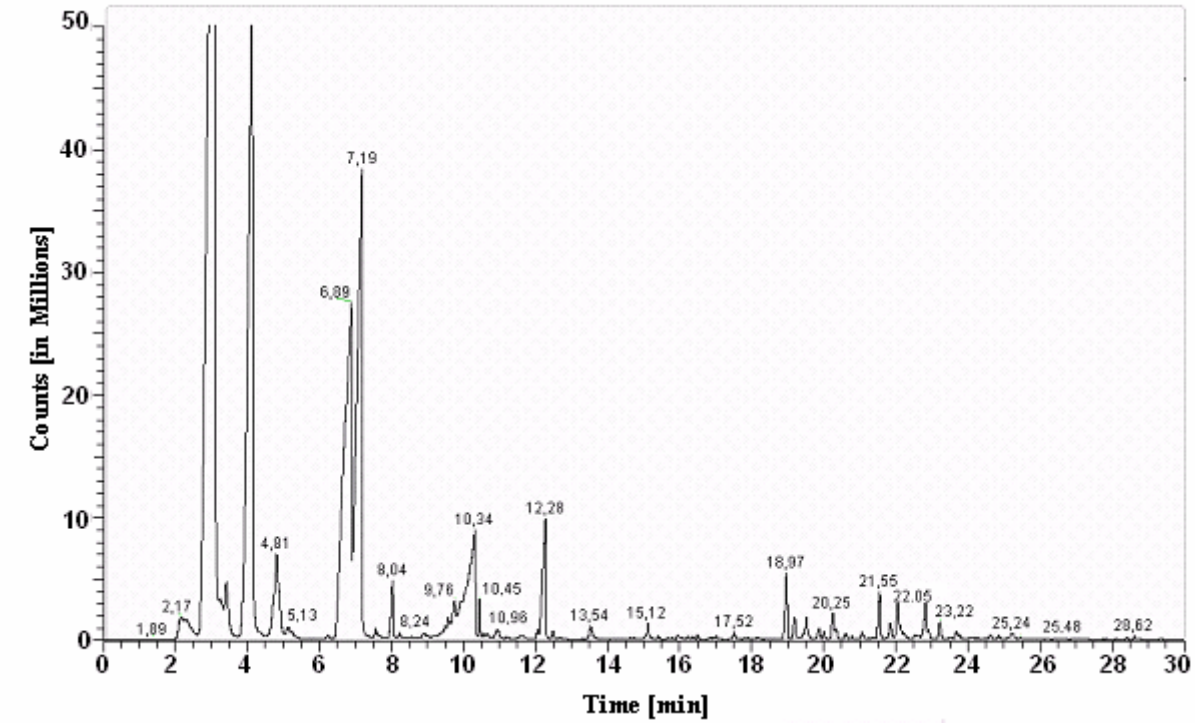

Figure 6.9: Dynamic headspace GC analysis of IR-treated, wetted paprika powder (a_w 0.84) after storage for five weeks in closed glass flasks at 20°C: 2.17 acetaldehyde, 2.96 acetone, 3.98 methyl-propanole, 4.81 2-butanon, 5.13 methyl-furan, 6.89 3-methyl-butanol, 7.19 methyl-butanol, 8.04 pentanal, 9.76 Dimethyl-disulphide, 10.45 hydroxy-propanole. 12.28 hexanal, and 13.54 furfural.

6.2 Decontaminated paprika powder added to high-water food

The micro-organisms present in dried spices cannot grow and multiply because of the limited amount of available water. Nevertheless, these micro-organisms may still be viable and retain the potential to multiply when added to high-water foods (Laroche & Gervais, 2003b; Fine & Gervais, 2005), such as meat and milk products. Refrigeration at 5–7°C is the major factor in stabilizing or extending shelf life of meat and milk (Sadovski et al., 1980).

Fresh meat, with a moderate pH of over 5.5 and high nutrient availability, is highly susceptible to spoilage micro-organisms (Kong et al., 2007). Meat products are highly surface contaminated with micro-organisms, even before adding spices; the contaminating micro-organisms include lactobacillus, enterobacteria, and pseudomonades originating in livestock and subsequently cross-contaminating the meat in the processing environment (e.g. in slaughterhouses) (Grohs & Kunz, 2000; Grohs et al., 2000). A contamination of 5 $\log_{10}$ CFU/cm^2 is commonly found on meat surfaces, while 7 $\log_{10}$ CFU/cm^2 is the upper acceptable limit during storage (Martinez et al., 2007).

Fermented milk products contain high concentrations of lactic acid bacteria (LAB) of 6–9 $\log_{10}$ CFU/g resulting in a pH lower than 4. Due to pH decrease and the possible production of bacteriocins, such as nisin, LAB are capable of weak to moderate inhibition of several food spoilage and pathogenic bacteria, such as *B. cereus* (Varadaraj et al., 1993).

Paprika is a good source of carotenoids and vitamins A and C, which are important dietary antioxidants (Howard et al., 1994; Lee et al., 1995). Ascorbic acid (vitamin C) is water soluble and decreases the pH of paprika powder. Antimicrobial agents, such as organic acids, have been used to inhibit food-borne bacteria and extend shelf life of processed food. Many naturally occurring compounds found in herbs and spices have been shown to possess antimicrobial properties and could serve a source of antimicrobial agents to be used against food pathogens (Kim et al., 1995). Paprika powder displays only a weak inhibitory effect (Zaika, 1988), though it has some potential due to its low pH. Other spices, such as garlic, cinnamon, or rosemary, have a higher antimicrobial activity. For example, a mixture of rosemary and vitamin C significantly reduced microbial growth and extended the shelf life of beef from approximately 10 d to almost 20 d; high antimicrobial activity against *B. cereus* has also reported for garlic, sage, rosemary, and cloves (Djenane et al., 2002).

This study evaluated the growth of *B. cereus* and of the natural background microbial flora during storage at 7°C in decontaminated paprika powder when (1) spiked on pork and (2) mixed with crème fraîche.

The untreated paprika powder (a_w 0.84) had an initial *B. cereus* concentration of 7.30 $\log_{10}$ CFU/g due to deliberate inoculation, in addition to 4.90 $\log_{10}$ CFU/g of natural background flora. Paprika powder was decontaminated using IR heating unit 3 at product temperatures of 100°C for 2 and 6 min, achieving concentrations of *B. cereus* of 2.89 and 2.35 $\log_{10}$ CFU/g, respectively, and of natural background flora of 1.99 and 1.45 $\log_{10}$ CFU/g. The concentration of lactic acid bacteria (LAB) on meat was also analysed during storage, as they were the dominant microflora due to the vacuum packaging of the meat prior to use.

6.2.1 Paprika-spiked pork

Fig. 6.10 shows the visual appearance of the meat at day 0 and after storage for 20 d. Paprika powder spiked on meat had a darker appearance at day 0 due to absorption of water by the powder. During storage, the paprika colour became lighter due to moisture loss. At day 15 and beyond, moulds were observed on some of the untreated samples (data not shown, but visually evident in Fig. 6.10).

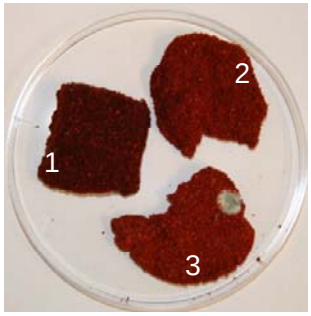

Figure 6.10: Image of piece of raw pork tenderloin immediately after dipping in paprika powder (1); pieces of pork after storage for 20 d at 7°C dipped in IR-treated (2) and IR-untreated (3) paprika powder.

The effects of microbial evolution during the storage of raw pork meat spiked with paprika powder were analysed in terms of counts of LAB, *B. cereus*, and natural background flora.

6.2.1.1 Effect on lactic acid bacteria

Since the pork was vacuum packed before use, the dominant bacteria on the meat surface were LAB. After surface decontamination of the raw pork tenderloin using near IR, a microbial contamination on the meat surfaces of 2.2 $\log_{10}$ CFU/g was achieved; this was considered the initial concentration of the control sample, i.e. fresh meat without paprika.

The concentration of LAB on the non-spiked sample during storage was compared with that of fresh meat spiked with paprika (Fig. 6.11). Within 9 d, the control sample displayed a fast increase in the surface growth of LAB of 4 $\log_{10}$ CFU/ cm^2, whereas further storage up to 30 d produced an additional moderate increase of 1 $\log_{10}$ CFU/ cm^2. The LAB concentration on the paprika-spiked meat samples increased during storage by 3 $\log_{10}$ CFU/ cm^2 after 20 d. The maximum attained concentration for the spiked meat was approximately 1.5-2 $\log_{10}$ CFU/g lower than for the non-spiked meat during the entire storage period. Thus, the paprika had an inhibitory effect on the growth of LAB present on the meat surface. However, when paprika was tested for its influence on the growth of *Lactobacillus curvatus* at 20°C, it was even found to produce a slight increase in growth rate, possibly due to the presence of sugar and other microelements (Shelef, 1983; Verluyten et al., 2004). LAB are usually quite resistant to the possible antimicrobial activity of spices; from a hygienic viewpoint, their increase during storage is not critical, as they can support meat maturation (Grohs et al., 2000).

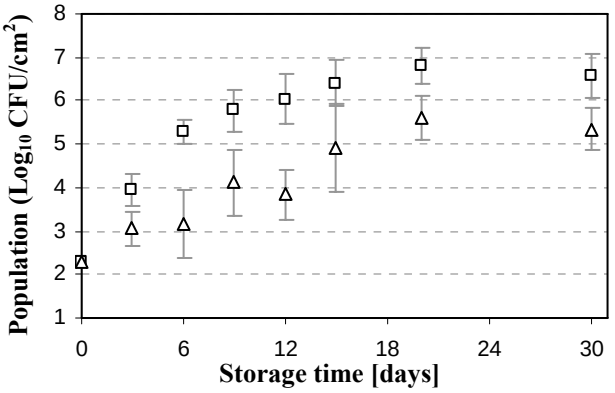

Figure 6.11: Evolution of lactic acid bacteria on fresh meat (□) and on meat spiked with paprika (Δ) after storage at 7°C. The detection limit was 1 $\log_{10}$ CFU/cm^2.

6.2.1.2 Effect on *B. cereus*

Fig. 6.12 shows the growth of *B. cereus* on pork surfaces, spiked with treated and untreated paprika powder, during storage. Pork surfaces spiked with paprika powder inoculated with *B. cereus* spores, but untreated with IR, had a concentration of 6.20 $\log_{10}$ *B. cereus*/cm^2. This microbial population remained constant at approximately 6 $\log_{10}$ CFU/cm^2 during storage.

Adding decontaminated powder to the meat surface resulted in low concentrations of *B. cereus* of 1.56 and 1.13 $\log_{10}$ CFU/cm^2 for powder treated with IR for 2 and 6 min, respectively. Differences between powder treated for 2 and 6 min were not significant during storage. Results from the six replicates for each IR treatment time were highly variable. Note that some of the samples displayed no detectable organisms from days 0 to 12. The microbial population remained at low levels below 3 $\log_{10}$ CFU/cm^2 up to day 12, and increased up to 4–4.5 $\log_{10}$ CFU/cm^2 by day 30. The high microbial concentration of the untreated powder was not attained for the IR-treated samples during storage. The expected shelf life of naturally contaminated pork stored in air at 7°C is unlikely to exceed one week. During this time span, the increase of *B. cereus* spores used for inoculation was approximately 2 log units. It can be concluded that a fraction of the *B. cereus* spores survived IR decontamination of the paprika powder, and that these spores were able to germinate and slowly grow on the surface of the pork. Since there was great variation between the samples, the probability of growth varied.

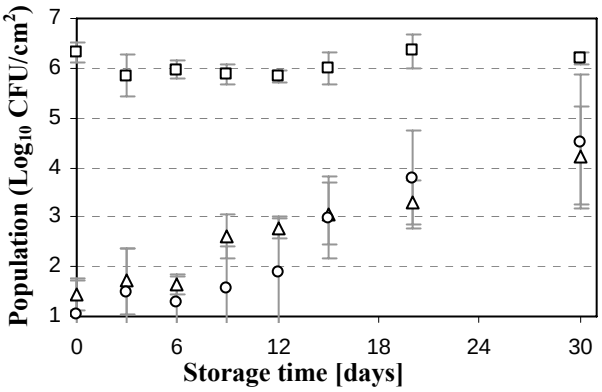

Figure 6.12: Evolution of psychotropic *B. cereus*, SIK 340, on a meat surface spiked with paprika powder (a_w 0.84) and stored at 7°C. The paprika powder used was untreated (□) or treated with near-IR at a product temperature of 100°C for holding times of 2 min (Δ) and 6 min (○). The detection limit was 1 $\log_{10}$ CFU/cm^2.

6.2.1.3 Effect on natural background flora

The natural background flora in treated and untreated paprika powder on meat surfaces was studied during storage (Fig. 6.13). Meat spiked with untreated paprika powder displayed an initial background flora concentration of 3.50 $\log_{10}$ CFU/cm^2. During storage, this concentration increased slightly by 1 $\log_{10}$ CFU/cm^2 until day 15 and remained at approximately this level until day 30.

Treated paprika powder initially displayed very low bacterial concentrations near the detection limit of 1 $\log_{10}$ CFU/cm^2, and remained at levels not exceeding 2 $\log_{10}$ CFU/cm^2 up to days 12 and 30 for 2 and 6 min of IR treatment, respectively. Further storage caused a slight increase to 2.5–3 $\log_{10}$ CFU/cm^2. Such levels are low for microbial contaminations on meat.

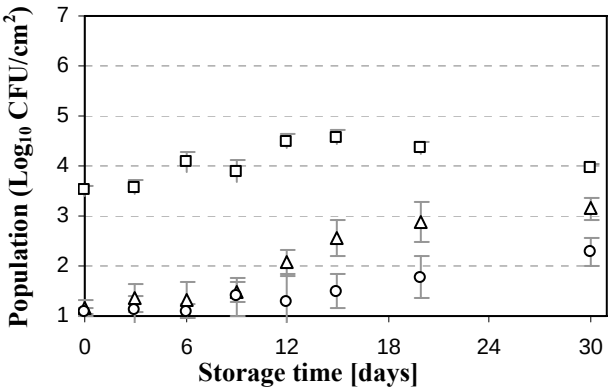

Figure 6.13: Total plate counts of the natural microbial background flora in paprika powder (a_w 0.84) adhering to a raw meat surface after storage at 7°C. Concentration was measured for paprika powder untreated (□) and treated with near-IR at a product temperature of 100°C for holding times of 2 min (Δ) and 6 min (○). The detection limit was 1 $\log_{10}$ CFU/cm^2.

6.2.2 Paprika-spiced crème fraîche

B. cereus concentration was evaluated for paprika powder mixed with crème fraîche after up to 60 d of storage (Fig. 6.14). The initial spore concentration in untreated, but inoculated paprika powder mixed with crème fraîche was approximately 7 $\log_{10}$ CFU/g. IR-decontaminated powder mixed with crème fraîche displayed an initial concentration of 3 and 2.2 $\log_{10}$ CFU/cm^2 for treatment times of 2 and 6 min, respectively. During storage, the microbial population remained constant under all tested conditions. However, an insignificant increase to 3 $\log_{10}$ CFU/g was observed for powder treated for 6 min, probably due to recovery of heat damaged spores, and a slight decrease to 6.5 $\log_{10}$ CFU/g for untreated powder.

The constant microbial values were caused by the pH of crème fraîche, which is under 4; this is below the pH needed for the germination and growth of *B. cereus*, but is unable to kill the bacteria. Other studies observed a slow decrease in bacterial concentration over time and were depending on the initial inoculum value. In studies of mayonnaise, low inoculum levels introduced into the product were unable to survive (Jagannath et al., 2001).

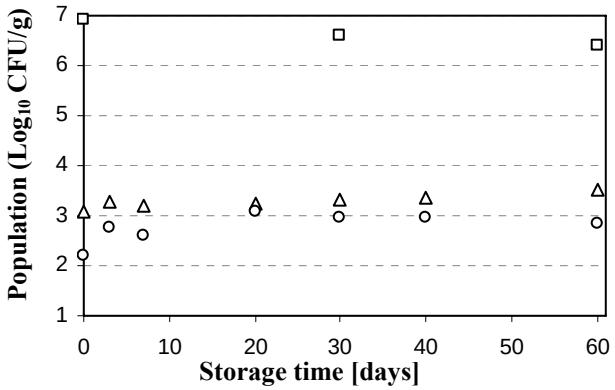

Figure 6.14: Total plate counts of *B. cereus*, SIK 340, in paprika powder (a_w 0.84) mixed with crème fraîche after storage at 7°C. Concentration was measured for paprika powder untreated (□) and treated with near-IR at a product temperature of 100°C for holding times of 2 min (Δ) and 6 min (○). The detection limit was 1 $\log_{10}$ CFU/g.

6.3 Summarized observation of food applications

Storage of powder in sealed plastic pouches was tested for paprika powder with a_w values of 0.76 and 0.84. For paprika powder with an a_w value of 0.76, the *B. cereus* population remained constant at 2–3 $\log_{10}$ CFU/g, while paprika powder with an a_w value of 0.84 displayed an increase in the number of *B. cereus* of 1 $\log_{10}$ CFU/g over the first four weeks of storage, but remained at approximately 2.5–3 $\log_{10}$ CFU/g. The obtained final microbial count in both powders of 2–3.5 $\log_{10}$ CFU/g was below the acceptable level of 4 $\log_{10}$ CFU/g in spices. The shorter IR treatment times, i.e. 6 min for a_w 0.76 and 2 min for a_w 0.84, are recommended, as longer times did not produce any additional reduction of microbial numbers. Overall colour and a_w remained constant during storage for all tested IR heating times, though for powder with an a_w value of 0.84, a slight colour decrease was observed in weeks 1–2. Dynamic headspace GC analyses of the volatile component content revealed an increase of commonly found Strecker-derived aldehydes caused by the heat treatment.

Adding decontaminated powder to the meat surface resulted in a low concentration of *B. cereus* of approximately 1.3 $\log_{10}$ CFU/cm^2. Differences between powder treated for either 2 or 6 min were not significant during storage. Results from the six replicates for each treatment time were highly variable. Note that some of the samples displayed no detectable organisms from days 0 to 12. The microbial population remained low, below 3 $\log_{10}$ CFU/cm^2, up to day 12, and increased to 4–4.5 $\log_{10}$ CFU/cm^2 by day 30. Thus, there was a fraction of *B. cereus* spores that survived the IR decontamination of paprika powder and that were able to germinate and slowly grow on the pork surface. Since there was great variation between the samples, the probability of growth varied.

Regarding the natural background flora, treated paprika powder initially displayed very low values near the detection limit of 1 $\log_{10}$ CFU/cm^2, which remained at levels no higher than 2.5–3 $\log_{10}$ CFU/cm^2 up to day 30 of the test. These levels are low for microbial contaminations of meat.

Paprika powder had an inhibitory effect on the lactic acid bacteria present on the meat surface of approximately 1.5–2 $\log_{10}$ CFU/g during the entire storage period of 30 days.

The microbial population of *B. cereus* in crème fraîche remained constant under all tested conditions at 2.2–3 $\log_{10}$ CFU/cm^2 up to day 60 of the test. The constant microbial values are caused by the low pH of crème fraîche, which at 4 is below the level needed for the germination and growth of *B. cereus*, but is unable to kill the bacteria.

7 Concluding remarks and perspectives

In the literature, little research describes the use of IR heating for the decontamination of food powders. The present research used paprika powder spiked with spores of *B. cereus* as a model system in testing the suitability of IR treatment as a technology for decontaminating powders. The heating equipment used was a pilot-sized near- and medium-IR tunnel oven constructed by IRCON Drying Systems. Water had to be added to the powder to overcome the heat resistance of the spores and to improve heat transfer inside the powder.

- *Effect of temperature development and distribution during heating at different IR wavelengths and heat fluxes*

Selected heat fluxes in the near- and medium-IR range were used to heat the paprika powder. Higher heat fluxes and lower a_w values produced higher surface temperatures, but also larger temperature differences between the surface and interior of the powder bed. At the same heat flux and at $a_w < 0.8$, higher surface temperatures were observed with medium-IR, while near-IR produced a greater penetration depth. Differences in surface temperature and penetration depth disappeared at $a_w \geq 0.8$. However, the penetration depths for both wavelengths were rather low, i.e. 1–2 mm, because the rough surface of the powder bed caused surface reflection and had a high scattering effect. Thus the heat transfer inside the powder bed was mainly due to heat conduction.

As constant heat fluxes produced undesirable surface overheating and a constantly increasing temperature gradient within the powder bed, the use of variable near-IR heat fluxes – i.e. a combination of higher, moderate, and lower heat fluxes – was advantageous. In this way, the surface and internal product temperature could be controlled and the temperature gradient within the powder bed reduced during heating. Limiting the rapid heating of the powder mass was the poor conductive heat transport inside the powder and the need to avoid surface temperatures over 120–130°C, to prevent product quality degradation.

The selected process for the IR heating of paprika powder consisted of a warm-up period following by a holding period at the desired product temperature. The IR heating operation was based on a HTST treatment, i.e. rapid heating to a high temperature, which preserved product quality due to shorter holding times. Using high and moderate near-IR heat fluxes facilitated rapid heating to the desired product temperature of 95–100°C within 4 min for a 10-mm-deep paprika powder bed; maintaining the desired holding temperature was best accomplished using a low IR heat flux in pulsed operation.

- *Effect of IR heating on paprika powder with different a_w and pH levels and its effect on microbial reduction*

During IR heating, the powder surface (from the top up to 2 mm of a 10-mm-deep powder bed) was the most critical part of the powder mass, displaying a fast temperature increase due to radiative heat transfer. This caused undesired browning (i.e. degradation of carotenoids) and evaporation of water (i.e. decrease of a_w), making it more difficult to inactivate spores. However, the changes on the surface did not significantly affect the colour or a_w of the *overall* sample (i.e. mixture of the entire sample) but did affect the overall microbial reduction. The existing a_w gradient (i.e. dry zone formation) between the surface and the bottom of the powder bed resulted in larger *D*-values for spores on the surface, i.e. longer reduction times. The overall colour was the most important factor in terms of visual impression, and colour changes were a result of powder wetting prior to decontamination and product temperatures over 60°C during IR heating.

The degree of microbial reduction was primarily dependent on the amount of water remaining in the paprika powder during heating, product temperature, and IR holding time. Higher microbial inactivation was achieved with higher initial and final product a_w values, higher product temperatures, and longer IR holding times. Powder heated to a product temperature of 95–100°C displayed a higher microbial inactivation than did powder heated to 90°C, while the impact of lowering pH from 4.5 to 4.0 was negligible. IR heating of paprika powder produced a linear spore reduction from approximately 7 to 3–2 $\log_{10}$ *B. cereus* CFU/g within 2–6 min, depending on the heating unit used. A non-linear reduction curve was achieved for microbial loads of *B. cereus* below 3–2 $\log_{10}$ CFU/g, which required longer heating times; this was because the higher heat resistance of the microbial spores at the surface of the powder mass significantly affected the overall microbial population, and because of the tailing phenomenon commonly found in spore-containing media.

Besides spores of *B. cereus*, the mixed natural background flora were also investigated and seemed to be more sensitive to IR heat treatment. A great reduction in natural background flora, from 5 $\log_{10}$ CFU/g to near the detection limit of 1 $\log_{10}$ CFU/g, was observed using the same IR heating parameters as were used for *B. cereus*.

- *Designing an IR process*

Much of this PhD project concerned the design and improvement of IR heating methods and of the decontamination sample holder for paprika powder. A total of three IR decontamination sample holders were developed at a laboratory scale, which are referred to as IR heating units.

(1) Open heating unit

Powder was placed in an open heating unit that offered no prevention against water evaporation. Heating was performed using constant heat fluxes, which produced a constant temperature increase in the paprika powder. This caused undesired colour degradation, especially on the surface, and decrease of product a_w, especially at a_w 0.5 and 0.8, resulting in unsatisfactory spore reduction. However, powder bed areas with a high remaining a_w value of 0.96 displayed a reduction of >6 $\log_{10}$ CFU/g for *B. cereus*. IR decontamination thus offers considerable potential when water is available and remains in the powder. Due to the effective removal of water from paprika powder by IR heating, this heating unit can be used to dry the wetted and decontaminated powder to a storage stable level of a_w 0.50.

As the fast decrease of a_w on the powder mass surface caused inefficient spore reduction, it was necessary to find a way to prevent water evaporation from the surface. To this end, two closed heating units were developed. To avoid product overheating and consequent quality degradation, the IR heating processes in these units used variable near IR, which allowed product temperature control.

(2) Glass panel heating unit

A closed sample holder was developed, using a near-IR-transparent glass panel. At an a_w value of 0.88, the load of *B. cereus* spores was reduced by 4.5 $\log_{10}$ CFU/g within 6 min. However, this heating unit had the disadvantage of allowing small gaps between the sample holder and the glass panel, permitting some evaporation of water even during the holding period. Nevertheless, this kind of heating unit using a near-IR-transparent glass top lid is

recommended when the powder is to be dried after decontamination, as the top lid can be easily removed, resulting in an open heating unit.

(3) *Plastic pouch heating unit*

Powder was IR treated in a closed near-IR-transparent plastic pouch of PET material. This heating process is recommended for IR decontamination treatment, as it facilitated the best prevention of evaporation during heating and thus produced the largest microbial reduction, i.e. at an a_w value of 0.80–0.84 a significant reduction of 4 $\log_{10}$ CFU/g was achieved within 2–3 min. The flexible consistency of the plastic material allowed convenient handling of the powder prior to/during decontamination, and prevented recontamination during subsequent storage and/or transport. For industrial applications, it is important to choose a proper plastic material suited to IR heat treatments; this could be a challenge, as the material must be inert to the powder product and transparent to the used IR wavelengths.

The heat treatment of sealed portioned food is similar to the process used in high-class cuisine, in which food is sealed and heat-treated under vacuum, a process called *sous vide* (French for "under vacuum"). Due to the vacuum in the plastic pouch, the process temperature can be reduced and product quality remains higher. However, it is important to achieve and hold a certain product temperature to inactive micro-organisms.

In preliminary screening tests of the IR heating of sealed powder portions, as air was evacuated, the paprika powder and the plastic pouch tightened (Fig. 7.1). Subsequent heating caused the pouch to expand, as temperatures over 100°C caused steam production and water evaporation; this finally led to a tear in the sealed plastic seam. Furthermore, vacuum packing the powder in the pouches caused it to agglomerate, due to the presence of water and the lack of air; the agglomerated powder could regain the character of powder by subsequent crumbling/milling (Fig. 7.2). In any further enhancement of the IR decontamination of powdered foods, the use of powder sealed in a vacuum package should be considered.

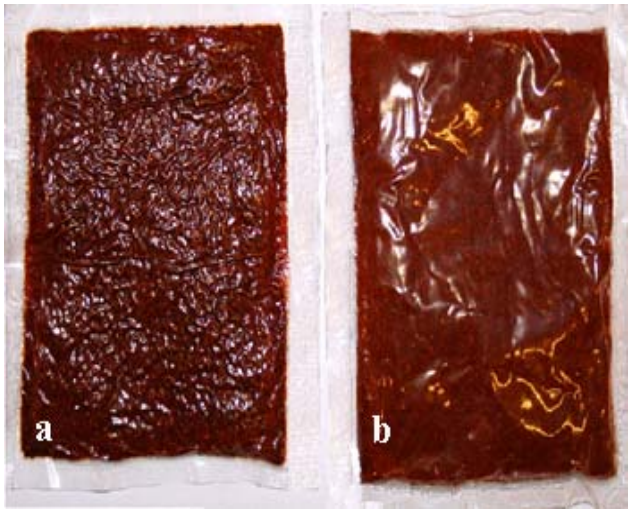

Figure 7.1: Paprika powder (a) placed on vacuum sealed plastic pouch and (b) sealed under atmospheric pressure.

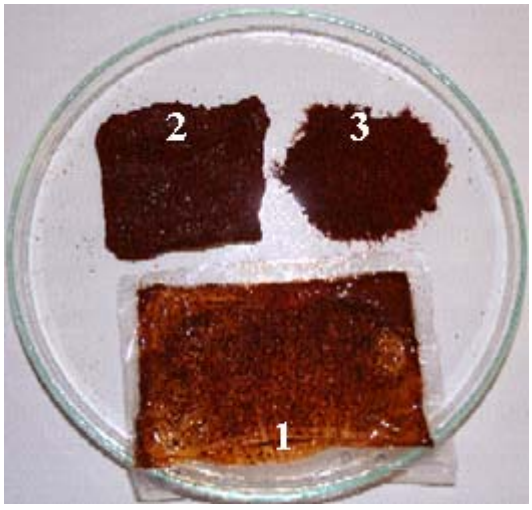

Figure 7.2: (1) Empty vacuum sealed plastic pouch with remaining paprika powder, (2) powder agglomerated due to vacuum sealing and IR heating, and (3) after restored powder consistence.

- *Product quality of the decontaminated powder during storage and after addition to meat and crème fraîche*

Decontaminated powder was tested in different applications of interest for the food industry, such as the final drying of decontaminated powder to a_w 0.50, the storage of wetted, decontaminated powder, and the addition of the powder to high-water foods.

The final drying of decontaminated powder to a storage-stable a_w of 0.50 was done within 4 min by heating with near-IR for two times 2 min, interrupted by a mixing step to eliminate the emerging a_w gradient. The drying operation had no additional effect on the microbial population of *B. cereus* and the final overall colour remained at post-decontamination levels. The disadvantage of this open drying operation is that subsequent handling and storage could recontaminate the dried powder.

To test for any recontamination subsequent to IR heating, the decontaminated powder (having a_w values of 0.76 and 0.84) placed in sealed plastic pouches was stored for 4 months. Storage at 20°C resulted in a constant a_w, an acceptable red colour, and constant to slow decreasing microbial counts of *B. cereus* of 2–3 $\log_{10}$ CFU/g over the entire testing period. These observed microbial levels are low for spices. The development of volatiles during the IR heating of powder was measured using dynamic headspace GC. Heating caused an increase in volatile emissions from the powder, mainly due to the products of the Maillard reactions; however, after storage of decontaminated powder for 5 weeks, no significant loss in volatiles was observed. Further information is required concerning the impact of the plastic

pouch material in terms of release of volatiles from it into the product during heating and storage, as well as the impact of heating on the inactivation of enzymes, such as lipase.

Adding paprika powder to crème fraîche and storing the mixture at 7°C produced constant microbial levels of approximately 3 $\log_{10}$ *B. cereus*/g over the tested storage period of 2 months. The growth inhibition was achieved due to the low pH of crème fraîche, which was under 4.

The decontaminated paprika powder added to raw tenderloin contained 1–2 $\log_{10}$ *B. cereus* spores/cm^2. During storage at 7°C, the spores were able to germinate and grow, and the microbial numbers increased to an acceptable threshold value of 5 $\log_{10}$ CFU/ cm^2 within 12–20 d. This level was still below the *B. cereus* concentration in the untreated paprika powder of 6 $\log_{10}$ *B. cereus*/cm^2. Notably, due to the low initial concentration of *B. cereus* on the meat surface, some samples had undetectable spore counts for up to 9–12 d.

For the food industry, it would be desirable to be able to store stable sealed seasoning powder portions of high microbial quality, which then could be added to high-water foods. The high-water food application of decontaminated powder should be extended to other products, such as highly spiced ready-to-eat meals, cheese, sausages, and chicken and egg products. Granular foods, such as nuts and cereals, can also contain high amounts of spoilage organisms, such as moulds and aflatoxin producers, especially on their surfaces, presenting another interesting area for microbial inactivation using IR.

Any industrial IR heating process used on herbs and spices should implement simultaneous decontamination and drying treatments directly after harvest in (sub)tropical countries. However, for spice importing countries, the wetting step after drying should still be considered.

The microbial load of herbs and spices is fairly high and is related to the environmental conditions before and during processing. Some efforts should be made to decrease the microbial contamination by improving conditions during growing, harvesting, and subsequent processing. For example, using cleaned water and higher hygienic standards in dryers or during grinding and packaging could lead to a reduction of the microbial load by 2 $\log_{10}$ CFU/g (Modlich & Weber, 1993).

Regarding the IR decontamination of food powders, further research is needed focusing on the development of IR equipment. The tested heating units were of laboratory scale and

provided information about various phenomena and difficulties arising during the IR heating of powders. For scale-up or industrial implementation the IR equipment must be enhanced, so some of the following recommendations should be considered:

- A practical IR decontamination process should facilitate continuous IR heating. Powder should be placed on a belt that transports it through different IR sections with selected IR heat fluxes.

- To prevent water evaporation during IR heating, preventative measures may have to be integrated into the equipment. Successfully tested materials for such measures were near-IR-transparent glass and plastic. A range of glasses and plastic materials are available from industrial suppliers, but study is required to determine which of the available materials are suited for a selected IR wavelength.

- The powder should be heated from the upper and lower sides of the powder bed. This would decrease the warm-up times, as heating is required only to the middle of the powder bed.

- It was observed that the surface is the most critical part of the powder bed, due to its direct exposure to IR. The use of stirrers, shakers, or similar devices is recommended, to prevent surface browning and drying and the increased heat resistance of spores, as well as to achieve a homogeneous temperature profile in the powder bed.

- From an engineering point of view, combinations of IR with other techniques are worth studying for their decontamination potential, such as IR and injected steam, IR and microwaves, IR and conductive drum/belt heating, and IR heating of powder in a fluidized bed.

Finally, two process suggestions are made regarding possible industrial implementation:

- Powder should be wetted in thin layers and placed on a pre-heated belt, to reduce warm-up times for the lower parts of the powder bed. The IR oven should be divided into sections with different heat fluxes, starting with a high to moderate heat flux to quickly achieve the desired product temperature. To hold the temperature at the desired level, low heat fluxes would then applied. The belt should be covered by insulation material and an IR-transparent top lid, i.e. making it a closed heating unit. On entering the IR oven, powder should be stirred to avoid surface overheating. Simultaneous water steam should be added to the powder occasionally. After IR decontamination, the powder should be packed or finally dried and stored. However, great care should be taken during packaging, as recontamination is possible.

- To avoid microbial recontamination, the IR decontamination process can be applied to portioned food powder, i.e. packed in vacuum-sealed IR-transparent plastic pouches. The moderately wetted and portioned powder would be IR heated from both the upper and lower sides. The powder would then be cooled down and stored in the dark until transported to a food processor to be added to high-water foods.

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