8 Microwave-assisted drying

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8.1 Introduction

Drying is among the oldest processing techniques for food, and its original purpose is to conserve perishable food in an edible state for a longer time. The deterioration of foods is based on chemical and biological processes that are mainly driven by free or unbound water inside the product and/or the presence of oxygen. (Barbosa-Cánovas and Vega-Mercado, 1996)

However, prolongation of shelf-life is not the only reason for food drying. (Sokhansanj S. and Jayas D. S., 2015) list several other aspects, such as the development of certain product quality parameters, e.g., colour and taste. Certain changes in those quality parameters can be beneficial for the overall customer acceptance of the product. These changes can be summarised as "Quality Enhancement". The "Ease of Handling" is another factor, which is beneficially influenced by drying processes. Packaging, storing, handling and transportation of dried goods are often easier and/or less expensive compared to fresh products. Reasons for this are reduced weight and volume and, especially for powders, better flow characteristics. "Further Processing" procedures, such as milling, mixing and segregation can also be improved by drying processes. Dried products are less sticky during mixing processes and the milling of dried goods consumes less energy. Furthermore, some microorganisms, especially meso- and cryophilic organisms, as well as insects can be partly inactivated depending on the process conditions.

Due to the fact that drying has been conducted for thousands of years all over the world, many different drying processes have been developed. The most important ones are hot-air drying (AD), freeze drying (FD), vacuum drying (VD), solar drying (SD) and osmotic drying (OD). Most of those processes can be assisted by microwaves, and especially microwave-vacuum drying (MWVD) has become an important industrial drying process. To understand the particular benefits of microwave drying processes this chapter will first discuss the principles of drying processes using the example of AD.

8.2 Principles of drying processes

AD is a common and well established industrial drying process. As idealised in Fig. 8.1, a typical drying curve of a foodstuff can be subdivided into three periods. After an initial phase (IP), a steady state is reached in which the drying rate (evaporated water over time) is constant. Therefore, this period is called "constant rate period" (CRP). In this period the product surface is still wet and the passing airflow transfers the heat for water evaporation to the surface. Simultaneously, the air is collecting the water vapour and is transporting it away from the product surface out of the dryer. This is a simultaneous heat and mass transfer. To keep the surface wet, enough water has to flow from the inside of the food to its surface. In the constant rate period this water flow is mainly capillary-driven and the surface temperature of the product is theoretically constant. As the water evaporation consumes energy, the surface temperature is typically significantly lower than the air temperature. The temperature profile in Fig. 8.1 A is adopted from a hot air drying process of carrot slices and it shows that this constant temperature period is not necessarily exactly reflected in real data. The factors that determine and limit the drying rate in the CRP are: the state of the air (temperature and relative humidity) as well as air velocity. By changing any of these parameters the drying process may be accelerated (or delayed) considerably.

This changes in the next drying period, the so called "first falling rate period" (1FRP). This period starts when the surface becomes dry, as water from the inside of the product cannot be transported fast enough to the surface. Now heat must be transported inside the product to enable water evaporation and the water vapour must be transported through the dry product area to its surface. This means that the resistance for heat and mass transfer is increasing, and consequently the drying rate decreases and the surface temperature rises slowly. As the area where evaporation takes place is retracting more and more to the centre of the product, the drying rate is slowed down continuously. It is not easy to increase the drying rate in this period. To enhance heat transfer, higher temperatures or higher air velocities have to be applied both leading to a higher product surface temperature. The product may then suffer from overheating as there is no cooling effect from water evaporation as seen in the constant rate period. The product may also dry out too fast in the outer layers, causing a phenomenon called "case hardening". This leads to the formation of a hard skin around the product that may hinder the further drying process or lead to product cracks.

The "second falling rate period" (2FRP) starts when all free water is evaporated and only bound water is left. For the drying of bound water, an additional desorption

energy is necessary, which is a further resistance for the drying process. Whether or not this period is reached in industrial drying processes depends on the hygroscopicity of the product and the target moisture of the process. A 2FRP is only visible when the drying rate is analysed (see Fig. 8.1 B and C) and not from Fig. 8.1 A. The slope of the curve in Fig. 8.1 C can be different depending on the dried product (Mujumdar, 2015). If the process is continuing, the temperature rises up to the temperature of the hot air.
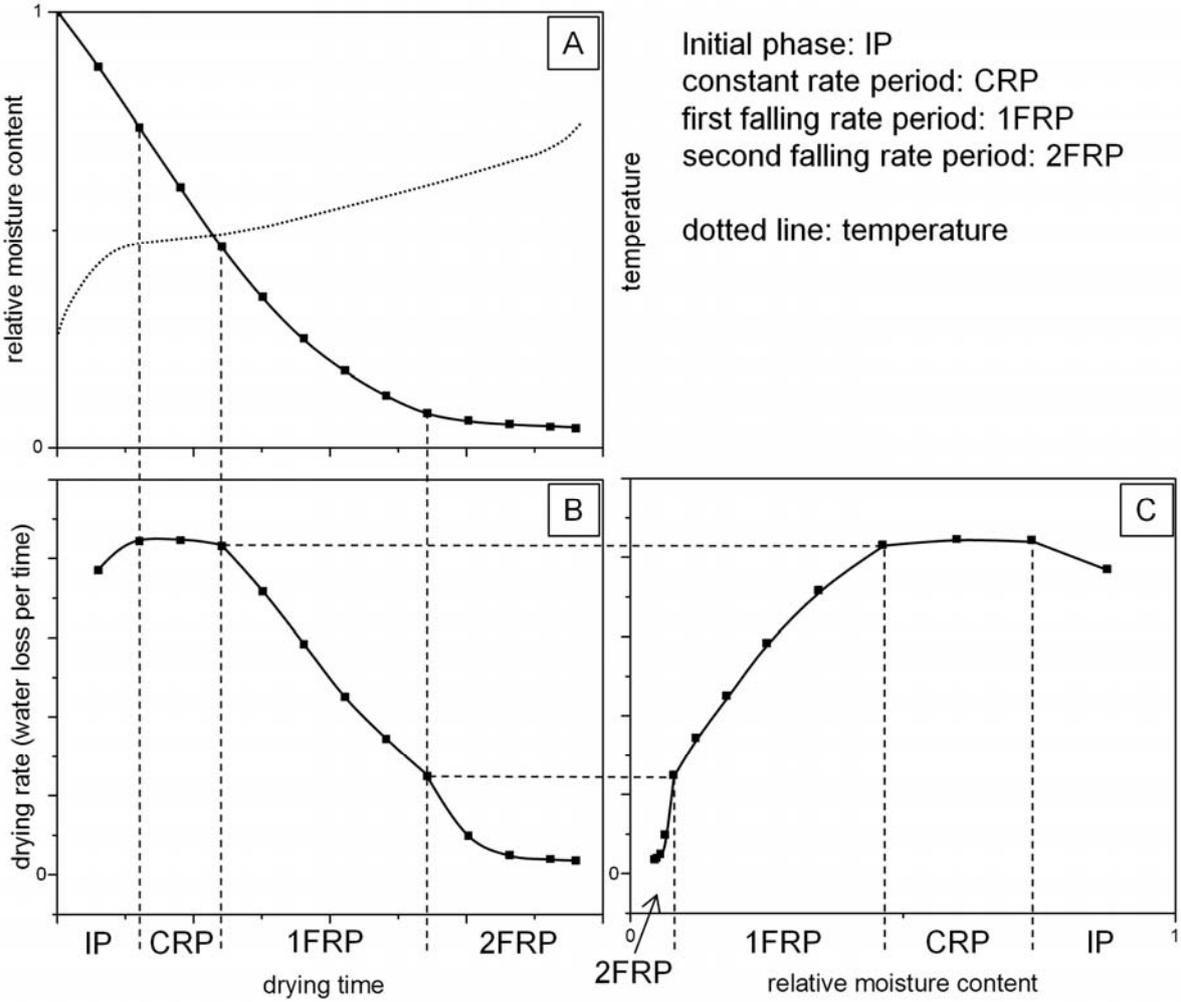
Fig: 8.1: Drying curves adopted from experimental hot air drying data of carrots to illustrate typical drying curves with initial-heating-, constant-, first-falling- and second-falling-rate-period in three typical diagrams. A: (relative) moisture content over drying time together with typical temperature curve. B: drying rate over drying time and C: drying rate over (relative) moisture content.

For industrial food drying processes it is difficult to determine the absolute length of the individual drying periods, because there are no sharp limits between them. As every piece of food is individual and the process conditions are not the same at all positions in the drying chamber, not all food particles are in the same drying period at

the same time. Therefore, it is a rather fluent transition between the drying periods, and the characteristic value to compare drying processes is commonly the time for the whole process until the product has reached its final moisture content. The drying time varies with process conditions and product. At which water content an acceptable dry state is reached depends on the intended purpose of the product (Jangam et al., 2010).

8.3 Specific aspects of MW-assisted drying

As described in chapter 1, microwaves can transmit energy in different ways. Their unique feature is that they provide heating inside the product. The extent of MW absorption depends on the dielectric properties, the size, the shape and the existence of other materials which absorb the microwaves in competition and therefore influence the local field strength.

8.3.1 Heating mechanism and selective drying

As described in chapter 2, the extent to which microwaves are absorbed by a material depends on its dielectric properties. It is the imaginary part of the dielectric constant, ϵ'', that determines the heat generated in the dielectric by an electromagnetic field of a certain strength and frequency. As water molecules- together with dissolved ions - have the main influence on ϵ'', microwave absorption usually decreases with decreasing water content of the material. The real part of the dielectric constant, ϵ', is also governed by water. Figure 8.2 shows this trend for apple pieces, but the curves for foodstuffs all show more or less the same behaviour. Reading from right (high moisture) to left (low moisture), we can see a slight increase of ϵ''. It seems strange that the samples lose a lot of water without a loss in ϵ'' but this effect is levelled out due to the rising concentration of dissolved ions and the shrinkage. At lower moisture levels (below 0.6), the absence of water causes ϵ'' to drop considerably. The material is now absorbing the microwave energy to a significantly lesser extent. When dry and wet food parts are coexisting, e.g., in the falling rate periods, it leads to a selective heating of water rich parts and is referred to as "levelling effect" (Zhang et al., 2006). Besides other reasons, such as focussing effects, this may lead to hot and cold spots or even a reverse temperature gradient in the product (centre warmer than the surroundings). The heating with microwaves overcomes the heat transfer limitations of other heat sources and accelerates the evaporation of water and consequently the mass transfer rate. In microwave drying, the drying rate is proportional to the absorbed microwave power in all phases of the process. This is what makes microwave drying interesting: the larger the products,

the more important become the differences in heat and mass transfer and the more obvious are the key advantages of microwaves.

As the amount of available water decreases over the course of the drying process, at a certain point, the efficiency of the microwave-drying process will decrease. If the microwave power is not adjusted at this point of the drying process, the electromagnetic field strength increases, because the less microwaves are absorbed in the drier, the higher the field strength becomes. This can create other problems, such as thermal runaway, arcing or plasma formation.
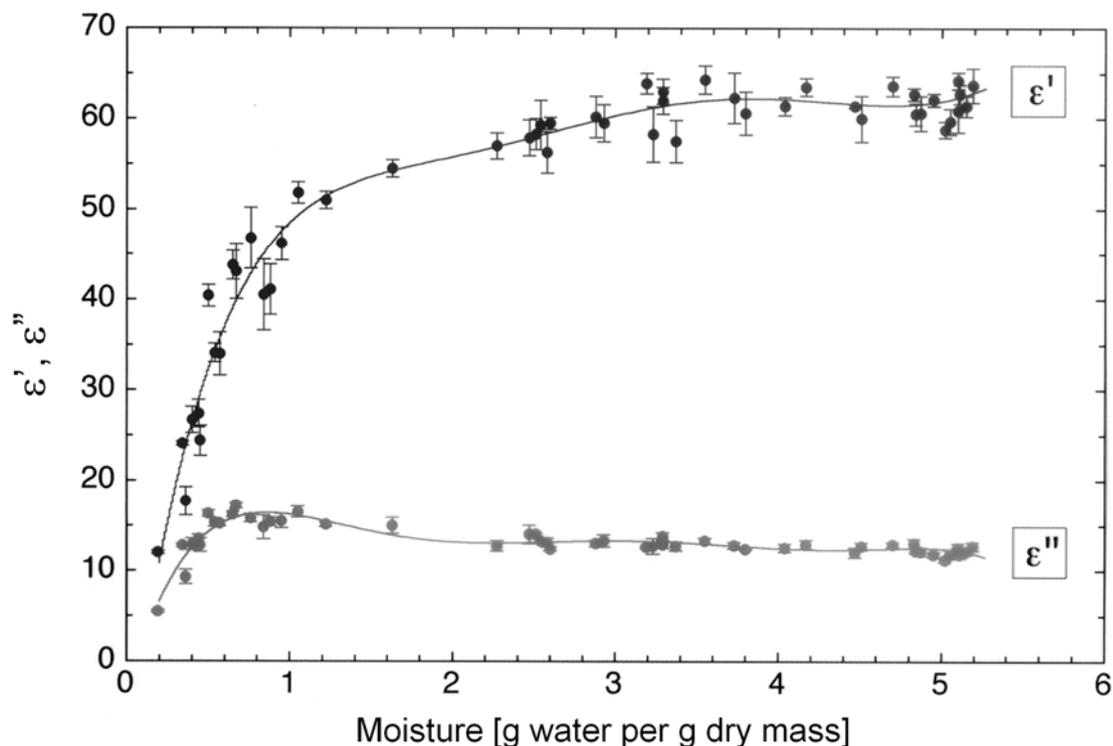
Fig. 8.2: Dielectric properties of apples as a function of moisture (Erle, 2001)

8.3.2 Thermal runaway

Like other polymers, many biopolymers in food show a steep increase in ϵ'' when exceeding a critical temperature. As the substance gets soft with increasing temperature, the molecules can move more freely and thereby absorb more microwaves, which leads to a self-accelerating process. This can also be seen for melting ice during heating of frozen products (see chapter 12). This phenomenon is known as 'thermal runaway'. An unstable system, which bears the risk of sudden overheating and burning of the product, is created. Without vacuum, it may even cause a fire. As a consequence, one is limited with the microwave intensity towards the end of the drying process, because the evaporation of water may not cool the product enough to prevent the thermal runaway. An alternative way to deal with the

low dielectric properties at the end of the drying process is to increase the amount of product exposed to the microwaves, for instance by creating a thicker product layer on a belt.

8.3.3 Plasma formation and arcing

Another reason to limit microwave power during drying has to be considered, especially if a vacuum is applied: a breakdown or discharge of the electrical field. This is basically a flow of electrons or air ions, which may even result in visible phenomena such as plasma or arcing. The field strength necessary for plasma formation at 2.45 GHz is reaching its minimum at a pressure of approx. 2.5 mbar (see chapter 17). While effects like plasma are desired in some technological fields, they are usually unwanted in microwave drying. Plasma formation consumes energy and may damage the equipment as well as the product. For instance, Teflon® parts can turn black and erode due to plasma formation. Foods can get thin black crusts, making them unsuitable for consumption. (Metaxas and Meredith, 1983) have provided a quite detailed description of breakdown effects along with their causes. Important factors are electrical field strength, pressure, and the type of gas in the atmosphere. Local field strength is of course a function of microwave power setting, drier design, mass and dielectric properties of the product, etc. As poorly absorbing products lead to high field intensities, it is often necessary to reduce microwave power towards the end of the process. Once a plasma has been created, it requires much less electrical field strength to be maintained. That is why it is so important to prevent peaks in the microwave intensity. The use of 'low ripple' magnetrons with a steady power supply or a steady state microwave source may reduce the problem.

8.3.4 Microwave field distribution

Another reason not to exaggerate microwave power during drying is the distribution of the microwave field. Although considerable progress has been achieved in drier design, the microwave field is never completely even. The occurrence of 'hot' and 'cold' spots is described in chapter 18. Some manufacturers use mode stirrers to distribute the waves, but it is always a good idea to move the product, so that all parts of it get approximately the same amount of energy. This is why household microwave ovens usually have a turntable. Using microwaves in a 'modest' way means that there is more time for heat conduction to level out any unbalanced microwave absorption. A simple way to improve the uniformity of the microwave field is to increase the size of the drying apparatus as the number of modes increases with the apparatus size.

8.3.5 Product size

Figure 8.3 illustrates one more potential pitfall in microwave drying of particulate foods. This curve for Mie's absorption coefficient stems from a calculation based on spheres of various diameters, which have the dielectric properties of water in an electromagnetic field with the commonly used frequency of 2.45 GHz; the corresponding wavelength is around 12.2 cm. A value of 1 for this coefficient means that 100 % of the incident electromagnetic energy is being absorbed by the object. The calculation reveals that the absorption of electromagnetic waves is increasing slightly with decreasing radius down to approx. 4 cm. In smaller spheres resonant interaction and focussing effects lead to fluctuating values and even values higher than 1. But for small spheres with a radius less than 0.5 cm, the absorption is very poor. This means that a small wet object may not receive enough microwave power and will not be heated. This effect is of practical relevance if the design and operation of the drier allows for single particles. As long as the particles are in contact with each other and form a bulk, the effect is not significant. Nevertheless, due to the focussing effects, objects within a radius size of 0.5 and 3 cm may experience uneven heating or overheating. These effects influence the microwave adsorption especially during the falling rate period of a drying process when the amount of water decreases and the wet part of the product is shrinking. In this phase, the water-rich parts decrease, and the microwave absorption will undergo all the limitations discussed above.
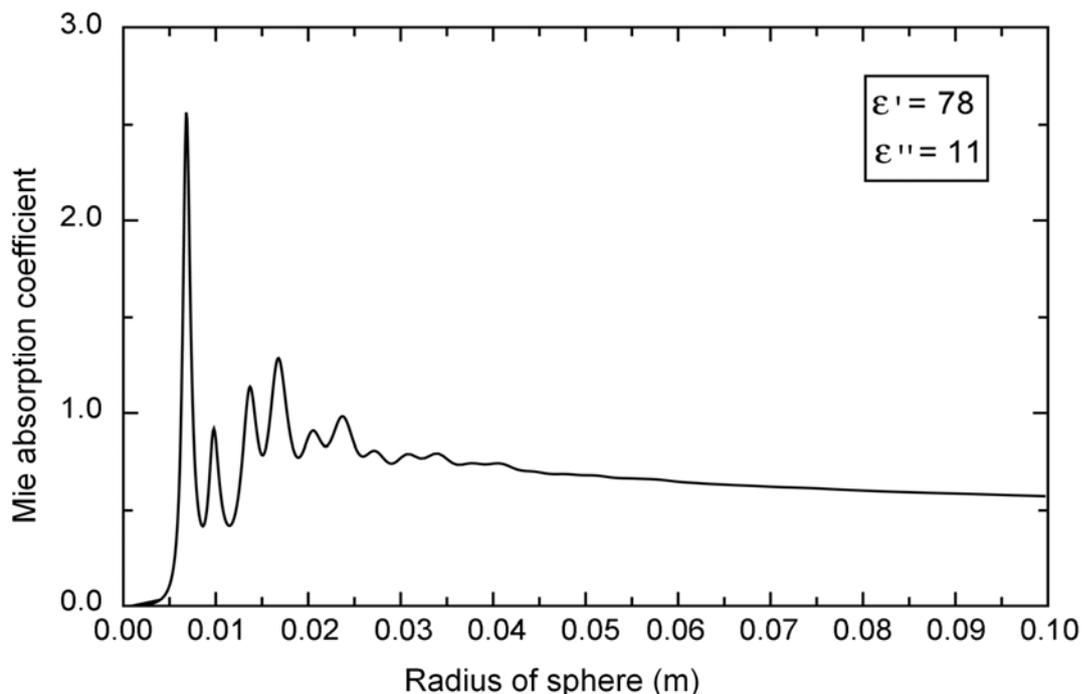
Fig 8.3: Mie´s absorption coefficient - calculated for spheres of water (Erle, 2001)

8.4 MW in drying processes

8.4.1 General aspects of microwave application

Microwaves are used as single or additional heat source in drying processes. The most important use of microwaves as heat source in drying processes are in AD, VD and FD. In the following sub-sections these three processes will be discussed.

Most publications in this field investigate the drying time and report one or two further product parameters like rehydration, shrinkage, ingredient retention, colour retention, hardness, before and after the drying process. The fact, that there is no standard procedure leads to numerous possible ways to investigate quality and process parameters and often makes the direct comparison of results from different authors not possible. In addition, microwaves can be applied in a drying process in various ways, i.e., *continuously*, *intermittent/pulsed* or *temperature controlled*.

Continuous application means that a certain power is used, regardless of any parameters other than the end-conditions of the process. These can be time, weight, or temperature.

Intermittent or pulsed application describes a process which introduces the microwaves in periodic repetitions. The frequency of the pulses can be constant or changing.

Temperature controlled systems regulate the power of the introduced microwaves depending on the product surface temperature. The temperature is hereby commonly measured with infrared temperature sensors. This makes it possible to keep the product temperature more or less constant.

In most of the available literature, the way of microwave application is not described in detail. This is the reason why the influence of the above described ways of microwave application on the drying process will not be discussed further.

8.4.2 Microwave assisted hot air-drying

Conventional hot air-drying (AD) is the most common drying process, because of its low investment and operating cost (Soysal et al., 2009) and ease of operation (Megías-Pérez et al., 2014). It accounts for approximately 85 % of all industrial drying operations (Mujumdar and Beke, 2003). Relatively long drying times (roughly 1-7 hours, depending on temperature, moisture content and product structure) and high air temperatures (up to 180 °C) lead to a degradation of valuable ingredients (Gamboa-Santos et al., 2014; Kowalski et al., 2013; Mclaughlin and Magee, 1998).

Product structure is another characteristic that is drastically changed during hot air-drying. Volume retention of hot air-dried foodstuffs reaches values between 15 and 30 % (Bruijn and Bórquez, 2014; Witrowa-Rajchert and Rzaca, 2009; Aversa et al., 2012). The formation of a hard crust (case hardening) at the outside of the product can also be mentioned as a disadvantage. Taste and colour are changing as well and may be beneficial or detrimental depending on the product and the consumer's taste.

When **microwaves** are used as heat source for an air-drying process the air-flow enables the removal of outside moisture of the product, while the volumetric heating of the microwaves leads to an accelerated moisture-transport from the inside of the product to the outside. This is especially advantageous in the falling rate period where the heat transfer is limited in a classical air drying process. This significantly decreases drying time. Several research groups found a reduction of drying time for many different products, like carrot slices (25-90 %) (Prabhanjan et al., 1995), kiwifruit (40-89 %) (Maskan, 2001), bananas (64.3 %) (Maskan, 2000), cranberries (60-82.5 %)(Beaudry et al., 2003), garlic (up to 80%) (Sharma and Prasad, 2006), Oyster mushrooms (75 %) (Bhattacharya et al., 2015) and collard greens (up to 70 %) (Alibas, 2009). The most investigated quality parameters were rehydration, which could be improved for carrots (Prabhanjan et al., 1995), kiwifruit (Maskan, 2001), bananas (Maskan, 2000) and garlic (Sharma and Prasad, 2006) and colour retention, which was better for banana (Maskan, 2000), garlic (Sharma and Prasad, 2006) and Collard greens (Alibas, 2009). The air used in these processes can be either hot (e.g. 80 °C) to prevent surface cooling or cold (e.g. 20 °C) to prevent overheating of the surface.

8.4.3 Microwave Vacuum-Drying (MVD)

Vacuum-Drying is generally recognised as a method for gentle drying of foodstuffs (Durance and Wang, 2002). Reduced pressures lead to evaporation of water at lower temperatures, which causes only few chemical, sensorial and nutritional changes (Drouzas and Schubert, 1996). Orikasa et al. found that VD of kiwi fruit is superior compared to AD in terms of ascorbic acid and colour retention (Orikasa et al., 2014). The drying times of vacuum-drying (up to 48 hours) are among the longest of all drying processes. The main limitation is the energy transmission. As hot air is not available, contact plates, radiation heat or microwaves are commonly used.

Microwaves are an effective way to transmit energy in this system, because of the volumetric character of the heating process. This helps again in particular in the falling rate drying periods and results in one of the shortest process times of all drying processes.

Figure 8.4 shows experimental curves from a microwave-vacuum drying process of carrots at three different power levels at a pressure of 50 mbar. 250 g of carrot slices with a thickness of 5 mm. Drying experiments were conducted in a modular drying processor with a rotating drum as described in (Rother et al., 2011). After a short heating time to reach the boiling point, the surface temperature stays between 30 and 35 °C until more than half of the water has been removed (the boiling point of pure water would be 32.9 °C at this pressure). When the product surface becomes dry (1FRP) the temperature starts to rise until the process is finished.
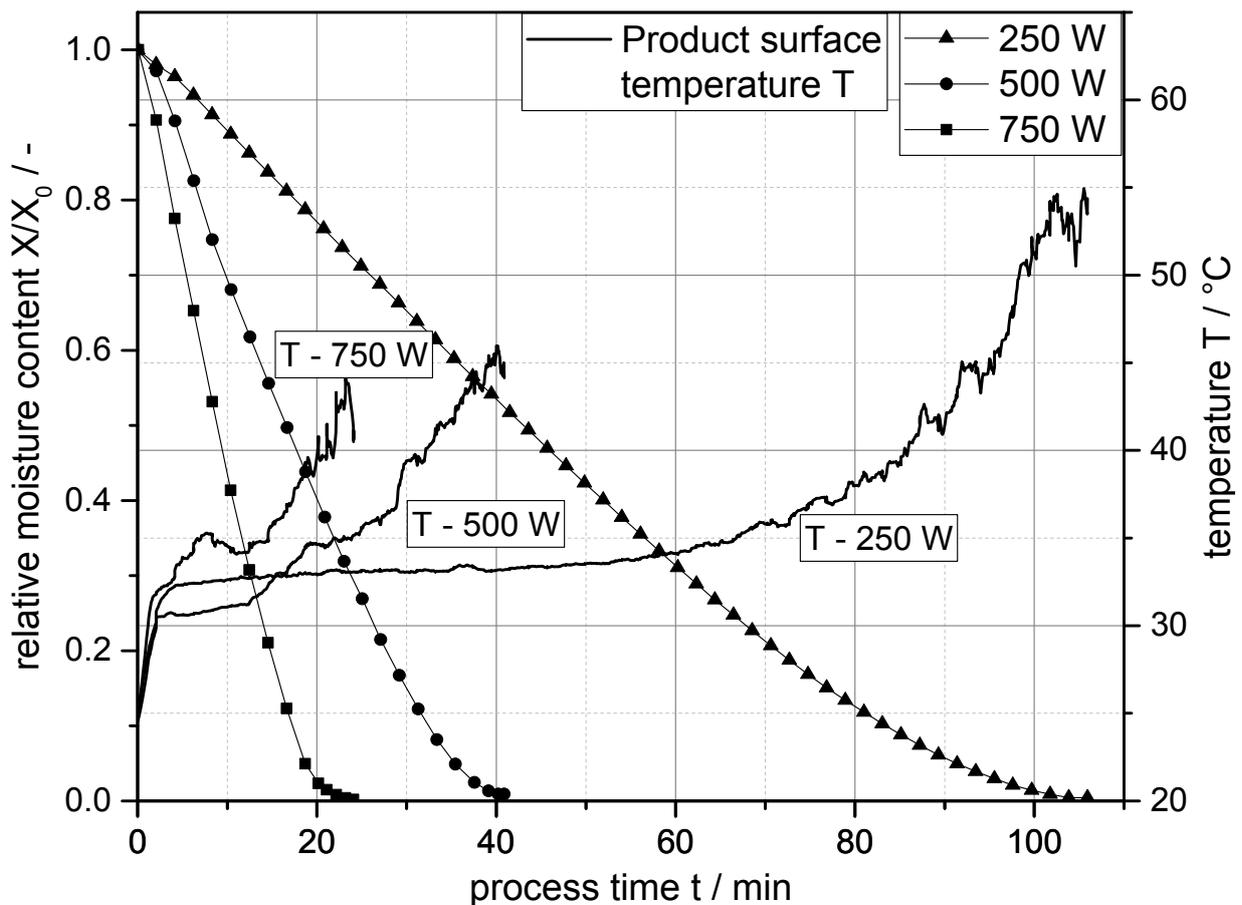
Fig 8.4: Drying curves with surface temperatures in MVD of carrots at a pressure of 50 mbar and three power levels (Siebert et al., 2016).

The key difference from an air drying curve is that the drying rate in MVD is proportional to the absorbed microwave power in all phases of the process. In some literature (Rother and Schuchmann, 2009) it is mentioned that the drying rate is proportional to the applied microwave power but this is only valid if all the applied microwave power is absorbed by the product and the product temperature is comparable, which is not always the case especially during the end of the drying process.

Figure 8.5 shows the drying rate of the carrot-drying experiments shown in Fig. 8.4. The basic shape appears similar to typical hot air drying curves (see Fig. 8.1 C), but the underlying physical principles are not. Reading them from right to left - as the process takes place - the curves have a slight initial increase, because some energy is needed to reach the boiling point. This is followed by a long slight decline of the drying rate. Thus showing that we can speed up the process even in the 'falling rate period' simply by the application of more microwave power. As long as there is enough water left, the product temperature is controlled only by the pressure level and the corresponding boiling temperature. As described in section 8.3.1, at lower moisture levels, the absence of water causes ϵ'' to drop considerably. The material is now much less inclined to absorb the offered energy, and the drying rate decreases. The excess energy may be absorbed by the apparatus, or go back to a safety water load or be absorbed by the dry product parts leading to a fast temperature increase (see Fig 8.4) or cause other side effects described in section 8.3.1. Nevertheless, the drying rate is still higher than in an air drying process as the water-rich parts are still selectively heated.
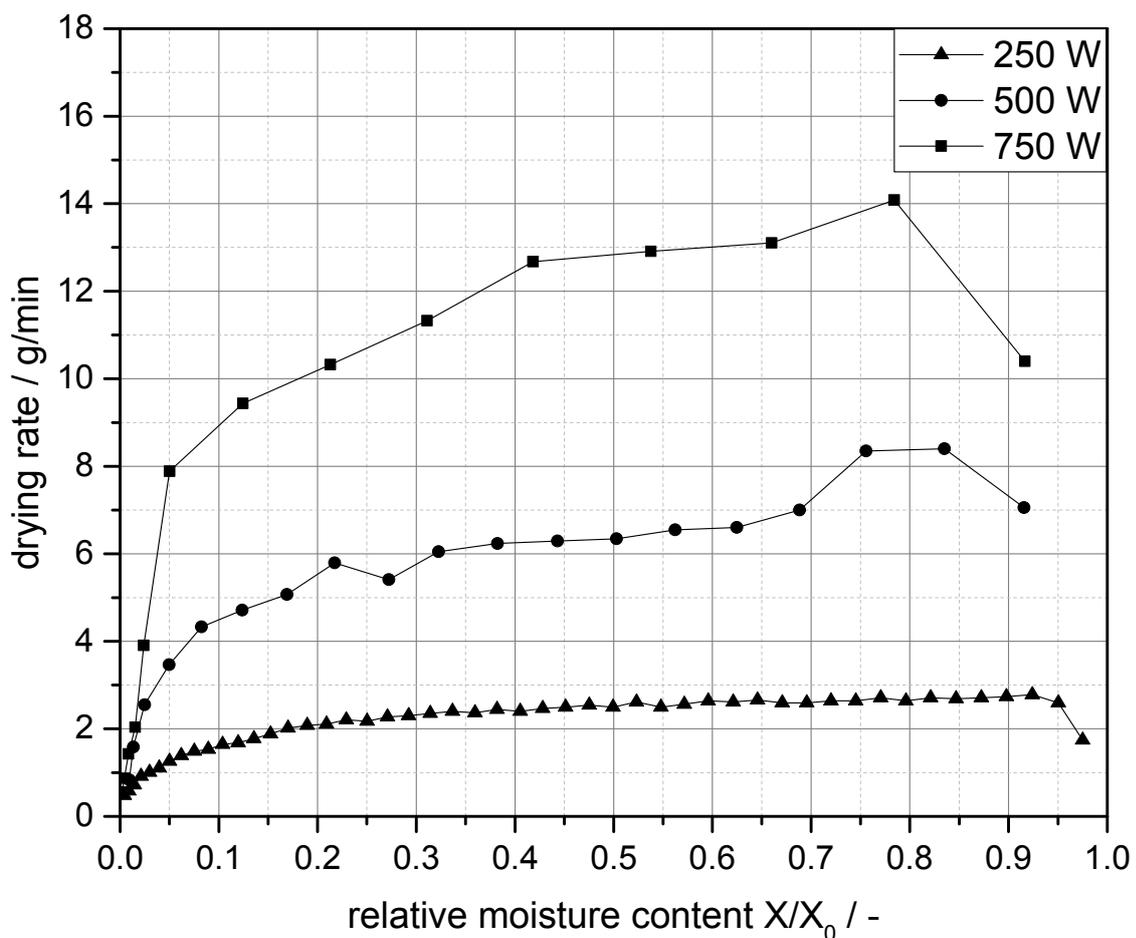
Fig 8.5.: Drying rate over relative moisture content during MVD of carrots at three different power levels.

As described above, not all of the applied microwave energy is necessarily absorbed by the product. Fig. 8.6 shows the absorbed microwave energy versus relative moisture content during the above shown drying process. First, it is interesting that even in the beginning of the drying process only approximately 70 % of the applied microwave power is absorbed by the product. This absorption rate depends strongly on the relation of product (water) volume to the volume of the cavity and can also be adopted to values close to 100 % by tuning the cavity (see chapter 1). But during the drying process the water-load will change and the absorption rate decreases. This is especially pronounced at higher microwave power levels where at the end of the drying process only 30 % of the applied energy is absorbed. For reduction of energy losses it would be advantageous to reduce the microwave power at the end of the process or to adjust the tuning of the cavity automatically.
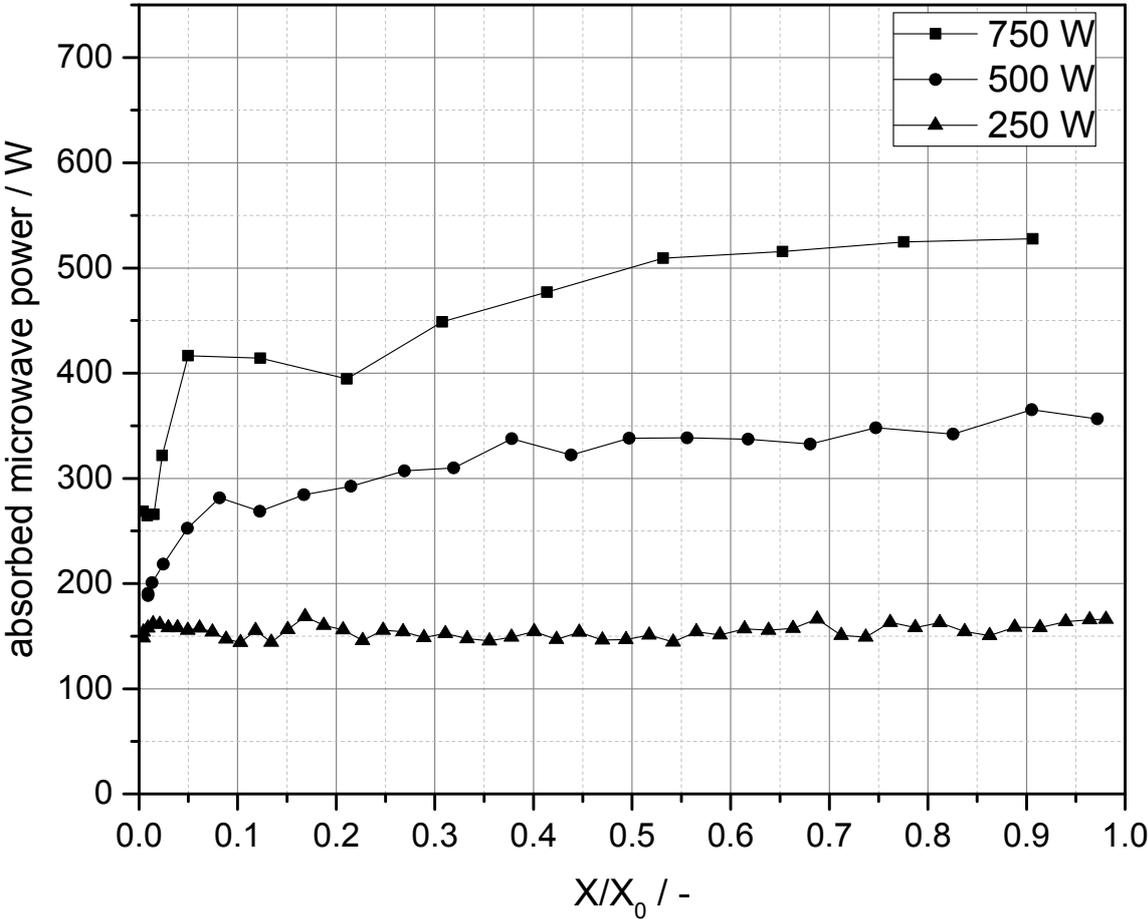
Fig. 8.6 Absorbed microwave energy versus relative moisture content during MVD of carrots at three different power levels (applied microwave energy).

Higher drying rates at comparatively low temperatures contribute to a higher retention of valuable ingredients, which was shown by Cui et al. for the ascorbic acid and carotenoid content of carrots (Cui et al., 2008; Mayer-Miebach et al., 2005). Both

ingredients had a retention ≥ 89 %, which was in both cases over 20 % higher compared to AD.

Volume retention is better compared to hot air-drying which was already shown for strawberries (Bruijn and Bórquez, 2014), carrots (Cui et al., 2008; Rother et al., 2011) and apples (Sham et al., 2001). One reason for that is an effect called puffing, which occurs when the evaporation rate inside the product is higher than the transportation rate of steam to the outside of the product. The resulting overpressure leads to a product expansion which results in a higher volume retention of the final product. This is illustrated in Fig. 8.7 where microwave vacuum dried and puffed carrot cubes are compared with air dried and freeze dried ones. As expected, FD produces very porous, though a little pale products, whereas microwave puffing yields a stronger colour, but the porous pieces look more like pillows, sometimes with only one air bubble inside. The AD control samples exhibit the usual problem of extensive shrinkage. The rate of rehydration is best for freeze dried products. This is because they have an open pore structure and – due to capillary effects – draw up water within seconds. Puffed particles, although quite porous, usually have a closed surface and cannot make use of the capillary effect. Still they are superior to air dried qualities, not necessarily for a higher rehydration rate, but because they get soft quicker in hot water. This is described in detail in (Rother et al., 2011).
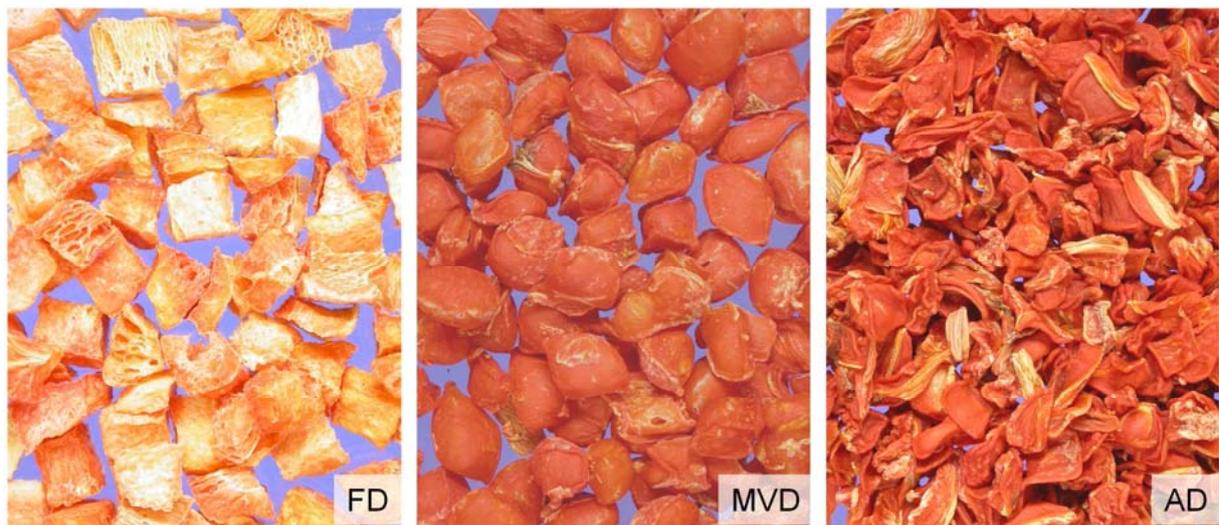
Fig. 8.7: Carrot cubes dried in a freeze-drying (FD), microwave-vacuum-drying (MVD) and an air drying (AD) process.

Furthermore, oxidation reactions are diminished, due to the low pressures and thus the low partial pressure of oxygen. This circumstance is beneficial for the colour, which was proven by (Yongsawatdigul and Gunasekaran, 1996) for cranberries and (Cui et al., 2008) for carrots.

In general, the previously mentioned attributes may lead to a product quality which is sometimes on par with freeze drying, but in most cases somewhere in between freeze- and hot-air drying. Special effects on aroma retention during microwave vacuum drying and microwave processes in general are addressed in section 8.5.

8.4.4 Microwave freeze drying

Freeze drying (FD) can be described as a special form of vacuum drying. The product is frozen in a first process step and subsequently dried in a vacuum chamber using a pressure below the triple point of water. This enables the water to sublimate directly from the solid phase into the gaseous phase. Commonly, heat is transferred like in vacuum drying processes by contact heating plates or radiant heat or both. Usually, plate temperatures between 30 and 40 °C are being used for contact heating or up to 150 °C for radiant heating. This results in low product temperatures throughout the whole process, which resolves in a gentle process. This is why freeze drying in general has the highest retention rates for valuable ingredients of all drying processes. Special quality aspects are discussed in section 8.5. The supportive character of the still frozen parts of the product is responsible for the highest volume retention possible. This goes along with a very porous structure, which lets the products appear a little pale, compared to fresh ones. When it comes to preserving the fresh product in its current state, freeze drying is generally acknowledged as the best drying process. Retention rates for ascorbic acid and carotenoids in carrots were reported to be above 95 % when compared to the fresh value (Cui et al., 2008). The volume retention has been found to be between 70 % (Rother et al., 2011) or even 80 % (Cui et al., 2008), again with carrot as the investigated product. For other products, like quince (Koc et al., 2008) or pumpkin (Nawirska et al., 2009), values of over 90 % are documented. These values are representative for other ingredients, which will most likely have similar dimensions. Because of this fact, the process is mainly used for high-value fruit and vegetable foods (Huang et al., 2009a). The reason for that is the high energy demand due to the need for a continuous vacuum and many phase transitions (freezing, sublimating and condensing / freezing) throughout the whole process and the long drying times of commonly between 12 and 48 h. Freeze drying is also widely used for the drying of pharmaceutical products (Liapis A. I. and Bruttini R., 2015).

The main drawback of freeze drying is the limited heat transfer rate as there is no constant rate drying period. As water is frozen and not liquid, it is not transported to the surface by capillary forces but only by diffusion. In addition, a dry and porous structure is built up in the outer product parts directly after surface drying. This leads to a low and decreasing heat transfer coefficient. It is obvious that microwaves instead or in addition to heating plates could be useful. This method was already

invented in the late 1950s, and it has shown that an acceleration of the process is possible compared to standard freeze drying (Copson, 1958). Regrettably, some major drawbacks have prevented microwave-freeze drying from replacing ordinary freeze drying. Apart from uneven heating properties due to the non-uniform distribution of microwaves, especially plasma formation (see section 8.3.3) is a massive problem at the low pressures, because it can lead to localised thawing of the product (Wray and Ramaswamy, 2015). When unfrozen parts are present besides frozen parts, the unfrozen parts are preferentially heated due to the high differences in the dielectric properties of water and ice. This will lead to a self-enforcing melting and structure collapse of the product.

Nevertheless, at low energy levels microwave assisted FD can be much faster than conventional FD. Experiments of FD of starter cultures (*lactobcillus paracaseii*) showed that a conventional FD of *lactobacillus paracaseii* took 840 min to reach a final water content of 4.5 % in a 100 g batch at 1 mbar and 30 °C contact heating plate temperature compared to 340 min in a microwave assisted FD at the same conditions were the product was initially heated with 200 W (2 W/g) microwave power to a surface temperature of 30 °C. The bacterial survival ratio in the MW assisted process was 86 % of the one in the FD process at less than half of the process time. These results were achieved in a drying processor described in (Rother et al., 2011).

Recently, the problem of uneven heating in freeze drying has been tackled by moving the product in rotating drums. This, however, is not recommended for products that are sensitive to abrasion (see also chapter 17).

8.4.5 Microwave drying in serial combinations

MVD may also be used as one step in a chain of dehydration processes. E. g., when combined with an osmotic pre-treatment, much of the water can already be removed by keeping fruits or vegetables in a concentrated solution for a few hours (Zhang et al., 2006). Meanwhile, some of the solutes, like sugar or calcium ions, may penetrate the tissue and have beneficial effects on the quality of the final product. For example, it has been found that sucrose and calcium chloride from an osmotic treatment reduce shrinkage of apples and strawberries during subsequent microwave-vacuum drying (Erle and Schubert, 2001).

Furthermore, microwave drying processes, especially MVD, is already being used in serial combination with AD and FD processes. The combination of MVD and AD is perhaps the most obvious option: as AD is relatively cheap, the first part of the process is preferably an air drier. During the constant rate period, the drying rate is acceptable and the product remains cool due to the cooling effect of the evaporation.

When the falling rate period starts and AD drying becomes inefficient, microwaves and vacuum can be used to overcome the heat and mass transfer limitations as described above (Cui et al., 2003; Figiel, 2010). Such a process was developed by Battelle Ingenieurtechnik GmbH (Eschborn, Germany) and implemented by Zifru (Zittau, Germany). In this case, the air drying step was designed to create a barrier on the outside of the particles so that the following microwave process produced a puffing effect.

Apart from this combination, especially a combination of MVD with FD is part of more recent research (Huang et al., 2009b; Kumar et al., 2001; Xu et al., 2005; Rother et al., 2011; Pei et al., 2014). FD is, as mentioned above, a rather expensive and time consuming process. By combining it with another drying process, like AD or MVD, it would be possible to reduce the amount of water before FD, and this may lead to a cost and drying time reduction. A crucial factor for this is the development of the product quality during the individual drying processes, because such a serial combination is only relevant if quality parameters, such as volume retention, rehydration properties, colour and ingredient retention are better, or energy consumption is lower compared to single drying processes.

(Rother et al., 2011) found that the order of combination is of great importance. They combined FD of carrots either with AD or with MVD. FD could be the pre-drying or the finish-drying step. A pre-drying with hot-air or microwave-vacuum drying led to a significant shrinkage of the product, which could not be made up for in the finish-drying step. Furthermore, achieved time-savings during the first phase of the process were more than compensated by a longer finish-drying phase. The reason for that is the formation of an outer crust, which acted as a diffusion barrier for the escaping moisture. It was found that it is much more beneficial for the product, in terms of volume retention, rehydration properties and drying time, when freeze drying is used as a pre-drying step. In this combination, the structure of the product was preserved in the pre-drying step, which allowed an even faster finish-drying step due to a very porous, already dried outer layer, which allowed the moisture to escape more quickly. This was investigated at fixed cross over points (relative moisture ratios at which the change between the processes is conducted) between the processes. However, the cross over point is very important in terms of product quality and drying time. Table 8.1 shows the influence of cross over points between FD and AD as well as between FD and MVD on volume retention, rehydration ratio and drying time of carrot cubes. The experimental procedures were the same as described in detail in (Rother et al., 2011). 250 g carrot cubes of 9 mm length were dried in the drying processor at 50 mbar and 515 W. AD was conducted at 95 °C air temperature. FD was performed under plate heating at a temperature of 30 °C and a pressure of 2 mbar. The volume

retention was measured as a bulk volume in a measuring cylinder before and after the drying, the rehydration ratio is the product weight after 10 min in 80 °C water relative to the fresh weight before drying. The finishing point for all experiments was when the drying rate was less than 0.03 g/min. FD was always used as pre-drying step and the % value after FD gives the relative moisture content at which the cross over between the processes was conducted. This means the higher the value the shorter is the pre-drying FD process.

Table 8.1: Influence of cross over point (relative moisture ratios in %) between FD / MVD and FD / AD on selected quality attributes and drying time for drying of carrot cubes.

Process	Drying time t / min	Volume retention V/V_0 / %	Rehydration ratio after 10 min
Pure FD	551	68.4	0.68
Pure AD	266	12.2	0.3
Pure MVD	68	24.4	0.37
FD10%-AD	493	57.2	0.5
FD27%-AD	442	39.2	0.44
FD44%-AD	376	24	0.38
FD61%-AD	350	16.2	0.3
FD10%-MVD	441	67.5	0.46
FD27%-MVD	336	48.7	0.41
FD44%-MVD	269	31.7	0.38
FD61%-MVD	204	22.2	0.41

Considering the drying time, it can be seen that as expected for the pure drying techniques, FD takes significantly longer than AD, and MVD is by far the fastest process. In addition, it can be derived in general that as more FD is involved in a particular process combination, the longer takes the drying process. As pure FD develops the best product structures in terms of volume retention and rehydration ratio also the combination processes with the longest FD part produce the best qualities. For volume retention this was the FD10%-MVD process and the best value for rehydration ratio is observed for FD10%-AD. The results show also that a puffing effect of MVD is not observed when FD is used prior to MVD, due to FD creating a porous microstructure from which moisture can easily migrate from the core to the surface, hence there is no pressure build up possible.

Also, (Kumar et al., 2001) investigated a combination of a FD pre-drying step with an AD process, instead of a MVD finish drying step with various, time dependent crossover points. Their results showed that an increase in pre-FD entailed a higher

volume retention, a higher rehydration ratio and a shorter rehydration time for carrots as well as pumpkins. (Xu et al., 2005) found that the opposite combination (AD, followed by FD) had an even worse overall quality than single AD. Nevertheless, a combination of FD and MVD has proven by many authors to be superior to a combination of FD and AD in terms of drying time (Huang et al., 2009b; Rother et al., 2011; Pei et al., 2014), structure (Rother et al., 2011) and ingredient retention (Pei et al., 2014).

In summary, serial combinations of drying processes are mostly beneficial for product quality parameters when compared to AD or MVD as single processes. In terms of energy consumption, they are more effective than single FD (Huang et al., 2009b; Xu et al., 2006; Xu et al., 2005; Kumar et al., 2001), but the drying time is not necessarily shorter.

8.5 Special Quality attributes of microwave assisted (vacuum) drying

If we look specifically at the retention of aroma, it becomes necessary to distinguish between two basic cases. In most foods, the aroma molecules are present in very small amounts so that they are likely dissolved in the aqueous phase. In this situation, the volatility of the aroma molecule in water is essential. Considering the fact that we perceive aroma – as opposed to taste – with our noses, it is quite clear that aroma molecules are generally volatile. Otherwise they would stay in the food during eating and not contribute to the aroma. In other words: If there is an interface between a water phase (i.e., a food) and a gas phase, the aroma molecules tend to choose the gas phase. In AD, the surface, where the aroma molecules can escape, is mainly the outer surface of the particles. This is also where the water molecules evaporate. So the aroma molecules already on the surface of the food particles will disappear into the gas phase, but more aroma molecules only reach the surface with the capillary water flow from within. As a result, the loss of aroma cannot happen at a higher rate than the loss of water, based on the respective starting contents.

This theory is supported by the measured curve in figure 8.8, in which the aroma loss (hexanol) of apple slices in AD and MVD are presented as a function of the water loss. For AD the measured values are initially on the diagonal, that means the aroma loss is proportional to the water loss. Later, as the product becomes drier, the capillary flow stops and other effects, such as binding with carbohydrates come into play. Here, the aroma retention is even better than proportional to the remaining water.
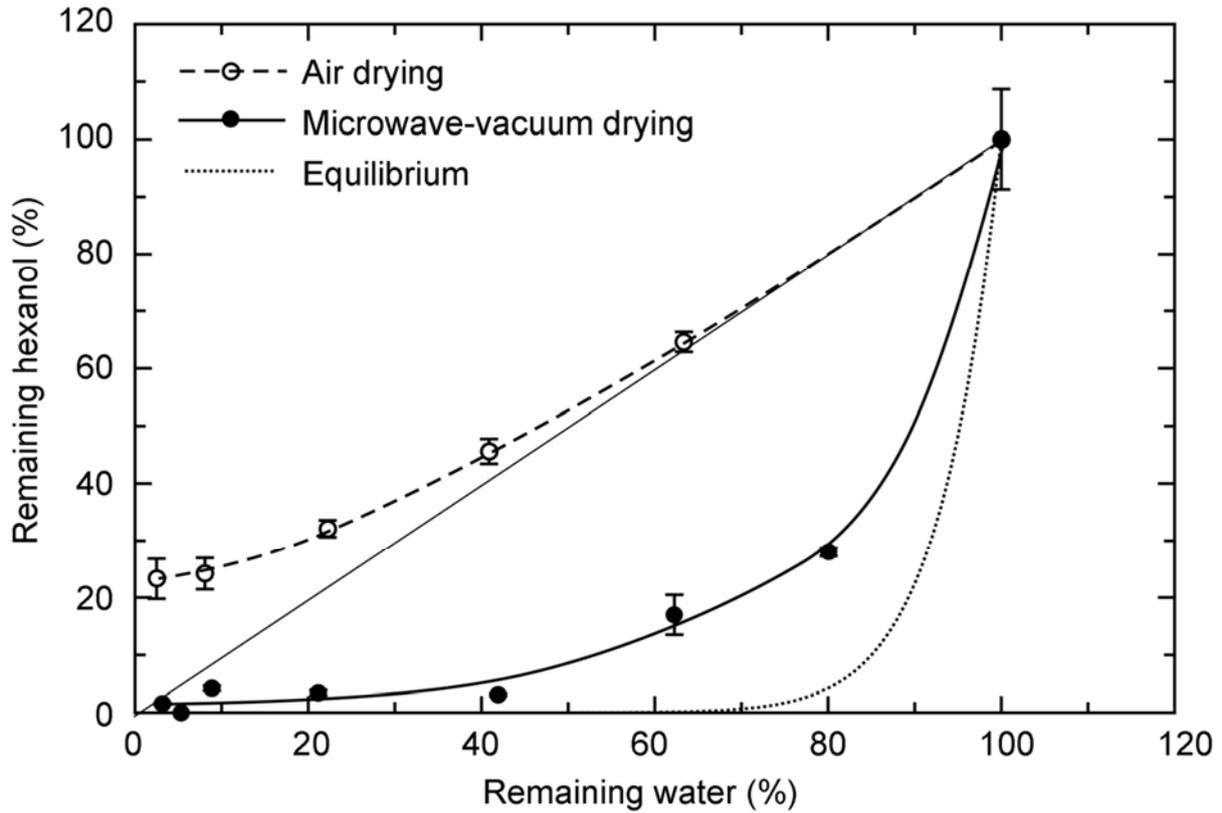
Fig 8.8.: Hexanol losses during MVD and AD of apple slices. Equilibrium according to Eq. 8.1

In MVD, the situation is different. Due to the combination of volumetric heating and lowered pressure, the boiling point is reached throughout the material. Consequently, steam bubbles are created in the apple slices. As mentioned before, the aroma tends to accumulate in the gas phase, and when there is more surface area, the losses are higher. Figure 8.8 shows the corresponding measurements. The losses of hexanol are in fact much more pronounced than for air drying. Only towards the end of the process (on the left hand side of figure 8.8), some of the aroma is bound to the almost dry matrix. The calculated curve, using equation 8.1, is an approximation based on the assumption of an infinite inner surface and thus represents the worst case of thermodynamic equilibrium. It shows how high values for Henry's Constant, H_{aroma}, (high volatility) lead to high aroma losses (Erle, 2001):

$$\frac{X_{aroma}}{X_{aroma,0}} = \left(\frac{X}{X_0}\right)^{\frac{H_{aroma}}{p^*}-1} \qquad 8.1$$

with relative aroma content ($X_{aroma}/X_{aroma,0}$) "remaining aroma" and relative water content (X/X_0) "remaining water" and p^* as vapour pressure of pure water.

For a more precise calculation, the amount of the inner surface and the diffusion coefficients of the aroma molecules would be needed. These are not available, but the simple, equilibrium based curve has the right tendency.

In herbs, the aroma is much more intense, because the portion of aroma compounds, called 'essential oils', is higher. Considering the poor water solubility of these compounds, we can assume that they form a phase of their own. Only very small amounts are dissolved in the aqueous phase. Under these circumstances it is not so much the volatility, i.e., the tendency to escape from the aqueous phase, but more the boiling point that determines the aroma retention. As essential oils mostly have a higher boiling point than water, it is possible to evaporate water without losing much of the aroma. This is supported by the results shown in figure 8.9, which compares microwave drying at various power levels. As demonstrated, it is better to apply high microwave power levels in order to evaporate the water quickly at its boiling point. In this example, there was practically no loss of essential oils at the two highest power levels. At lower power levels we give the oils more time to escape, either directly into the air or via the aqueous phase.
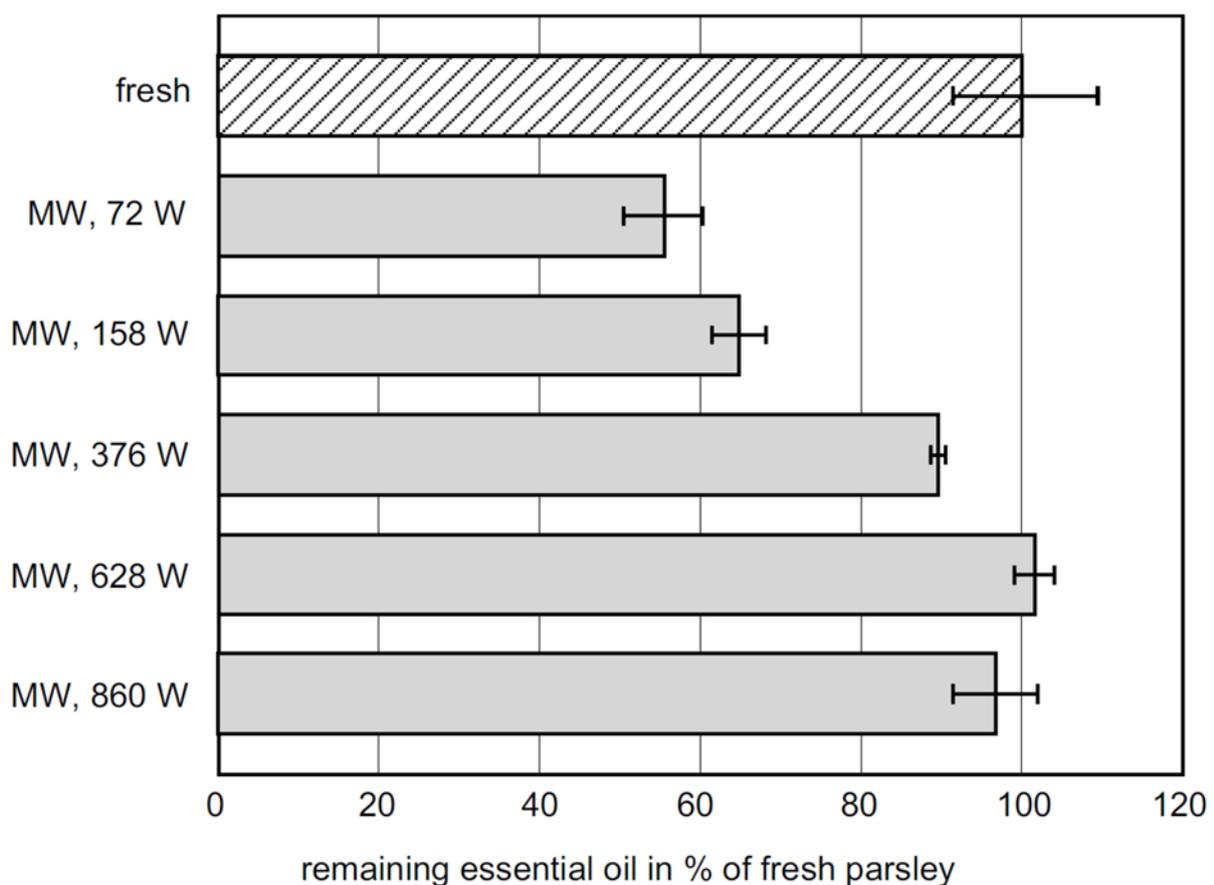
Fig 8.9: Essential oil losses of herbs after MVD at different power levels (Erle, 2001).

8.6 Microwave drying applied in food industry

Microwave drying is still not common in the food industry. There are many reasons for its limited use: the technical problems described above were not well understood in the past. This has led to some failures, which have surely discouraged other potential users. (Schiffmann, 2001) has listed a number of formerly successful applications that have been discontinued. Among these are the finish drying of potato chips, pasta drying, snack drying, and the finish drying of biscuits and crackers. It is apparently not always the microwave process itself but rather changes in the circumstances of production that make competing technologies more successful.

In spite of these difficulties, there are some applications. (Schiffmann, 2001) mentions cereal cooking and drying with a production rate of nearly 1 ton/h. Pasta drying with microwaves is carried out in Italy. MVD is being used for meat extract and, at least for a number of years, for the production of a powder made from orange juice concentrate (Attiyate, 1979). The combination of air drying and microwave-vacuum puffing is being used in Germany and Poland for fruits and vegetables. As the food industry does not disclose all its production processes, we cannot expect this list to be complete but additional actual aspects of industrial microwave applications are given in chapter 17.

(Hauri F.W., 1989) has provided values for the necessary investment and the specific energy requirements of five different drying methods (see Table 8.2). Based on the same throughput, the investment needed for MVD is rather high, while the energy figures are more favourable than for air drying.

Table 8.2: Specific energy demand and investment costs for selected drying processes according to (Hauri F.W., 1989).

Type of drying process	Specific energy demand kWh/kg water	Specific investment costs for equal throughput
Air band drying	1.9	100 %
Spray drying	1.6	120 %
Vacuum contact drying	1.3	150 %
Microwave vacuum drying	1.5	190 %
Freeze drying	2.0	230 %

8.7 Modelling microwave drying

What is modelling of microwave drying? The first thing most scientists would think about is the prediction of drying time, temperature and moisture distribution in

dependency of process parameters like MW-power, air temperature and velocity or pressure in case of vacuum processes. Consequently, this "process modelling" is also the focus in literature. But it has to be pointed out, that especially concerning the drying of foods, there are other important parameters, mostly related to product quality, like rehydration, colour, ingredient retention, volume retention, sensory aspects and more, which are also determined by the process parameters. Consequently, it would be obvious to develop models for the prediction of product quality in dependency of process parameters ("product modelling"). The latter is very rare in literature and most studies are limited to the monitoring of product quality parameters and just show qualitatively how they are influenced by process parameters; like "increasing air temperature leads to decreasing colour values". Quantification of these effects is rare and only possible with empirical equations. This was for instance recently shown with response surface methods for the rehydration ratio of jerusalem artichokes in dependency of microwave power (Karacabey et al., 2015) or for colour change, rehydration ratio and hardness of okra in dependency of microwave power and air temperature during a microwave air drying process (Kumar et al., 2014). Nevertheless, the correlation coefficients range from good to fair. To the best knowledge of the authors, there is no publication showing the attempt to develop a physical based model for the simulation of product quality parameters in dependency of process parameters. After having a closer look on the physical background and the efforts in "process modelling" it will get clear why "product modelling" is so to speak not existing.

Like for every modelling attempt, *„process modelling"* of microwave drying can be achieved using two fundamentally different methods for the model development. There are empirical models that are simply based on correlation equations which describe the observed effects, and models based on physical principles. Sometimes, these methods are also mixed, and physical based relationships are used when known and other unknown relationships are described with empirical equations. A common way to describe an unknown relationship is also to neglect it. This is not necessarily negative, as validation experiments can be performed in a model-supporting suitable way like "thin-layer" drying. In addition, it is the way how models developed over many years of research from very simplified models to more complex ones after the simplified models helped to understand basic drying principles. Not to forget the increase in computational power, which makes it possible to solve complex implicit equation systems in a short period of time.

(Feng et al., 2012) recently reviewed models for microwave drying of foods. They provide an overview about which parts of the drying process are addressed by the

model and they also include the special aspects of microwave heating. The first part is the basic theory of the drying model:

Simple *empirical equations* are often used to correlate the drying rate with moisture content, microwave power density, microwave frequency, temperature, air flow rate and sample geometry (Feng et al., 2012). Frequently used equations are the page model (Page, 1949) along with other exponential equations, e. g., shown in (McMinn, 2006) for drying of lactose, which has also been applied by (Khraisheh et al., 1995), (Tulasidas et al., 1995), (Diaz et al., 2003), (Bhattacharya et al., 2015) and many others. MVD drying has been modelled for apples, kiwi fruits, and pears by a statistical regression method in order to show the influence of microwave power and pressure on a drying constant (Kiranoudis et al., 1997). From a scientific point of view, these empirical models are very limited in use, because the physical principles are not included. But for an industrial process, these are useful and easy to handle tools for the control of known processes.

Another approach is the *"diffusion model"* which limits the drying to a mass transfer problem by calculating an effective diffusion coefficient (D_{eff}) from the measured drying rates. This is possible from Fick's second law for several geometries (Crank, 1976) and the temperature dependency of D_{eff} may be attributed by the law of Arrhenius (Sharma and Prasad, 2004). Several studies have been done underlying these principles (see (Feng et al., 2012)), but especially for the modelling of microwave assisted drying, this method is limited as a part of the idea of microwave heating is to overcome diffusion barriers, e. g., by steam creation inside the product. This changes the physical principles underlying Fick's second law and turns the model to a quasi-empirical one. E.g., (Erle, 2001) showed that models on MVD can be simplified by assuming that the absorbed energy is just used for the evaporation of water, which leaves the object as a convective flow of steam. This makes the calculations of mass transfer obsolete and has yielded a good correlation with measured drying curves of apple cuboids.

The most physically based approach for the modelling of microwave drying processes is the application of *heat and mass transfer* balances. There are different models developed from different assumptions like the "irreversible thermodynamics approach" (Luikov, 1964), the "unsaturated porous media theory" (Philip and Vries, 1957), the "volume average technique" (multiphase model) (Whitaker, 1977), the "two region model" (Peishi and Pei, 1989) and the "continuous-thermo-hydro-mechanical model" (Kowalski et al., 2009). Several simplifications of these models exist for special geometries, like sliced or spherical food (Lu et al., 1998; Lu et al., 1999), one-dimensional models for infinite slab or cylinder (McMinn et al., 2003), or two-dimensional models (Lian et al., 1997). Additional to the principles of the calculation

approach, different conditions can be taken into account. There are the assumptions for free water transport, which may be capillary or diffusion-controlled, the consideration of bound water and the consideration of vapour and/or air pressure differences (e.g., for vacuum drying) (Feng et al., 2012).

A general challenge for microwave assisted processes is the calculation of the energy source in the simulation. It ranges from empirical calculations over values based on the applied MW-power evenly distributed in the product or changing in the product according to Lambert's law to a full electrical field analysis distribution in the cavity and the sample according to the equations of Maxwell (Feng et al., 2012). From the local electric field strength, the local heat dissipation can be calculated with the knowledge of the dielectric properties (see chapter 2). However, as well as the local transfer coefficients for heat and mass transfer and the local heat and mass properties of the product, the dielectric constants are functions of local moisture, temperature and structure of the product. A deep understanding on the dependencies between these parameters and their local distribution would be necessary for an accurate simulation. In addition, all these parameters are not only dependent on each other, but also changing with time. Nevertheless, to improve the existing models, several empirical correlations are available between moisture content or temperature and material properties or transfer coefficients, e.g. (Jia et al., 2003) for carrot and potato samples. The influence of temperature, moisture content and porosity on the dielectric properties are described in literature for several foods, exemplarily shown in (Ozturk et al., 2016; Solyom et al., 2013; Piyasena et al., 2003; Venkatesh and Raghavan, 2004; Sosa-Morales et al., 2010; Jha et al., 2011; Knoerzer et al., 2004), see also chapter 2. This shows the complexity of an accurate physical-principles-based simulation of a microwave assisted drying process even if only a "process modelling" is considered.

Addressing "product modelling" would mean to transfer the simulated data of local product temperature, pressure and moisture into product quality changes. These are mainly structural aspects which determine volume retention, hardness and rehydration rate, and ingredient-dominated aspects leading, e.g., to colour and concentration changes, not to forget the sensorial quality. The latter are often complex chemical reactions which are not fully understood. Even if the physical/chemical principles underlying these product changes would be clear (which they are not), the heterogeneous multiphase nature of many foods, consisting of cellular structure, fibres, suspensions or emulsions, makes "product modelling" to date impossible. As described above, only empirical correlations are reported (Kumar et al., 2014; Karacabey et al., 2015; Bal et al., 2011; Wang et al., 2009; Qi et al., 2014).

A major drawback for all drying models and simulations is their validation. The first challenge is a proper measurement of the part of microwave power which was absorbed by the product and not lost in the cavity or reflected back to the magnetron. Cavity tuning and diodes measuring the returning microwave power are necessary. As the recent models deliver a lot of information on a microscale dimension (e.g., finite-elements / differences methods) the validation of temperature, moisture and pressure data is already demanding for integral values and even more for 3D resolved measurements. Possible process variables to measure are temperature and pressure with, e.g., optical fibres, which are limited due to the fibre size, costs and resolution (Feng et al., 2012) ,see also chapter 15. Magnetic Resonance Imaging (MRI) has provided the opportunity to investigate the distribution of water and temperature inside the product online and non-destructive during the process. This is a valuable tool to verify 3D calculations, comprising heat capacity, thermal conductivity and diffusion coefficients or moisture distribution measurements. With 3-D-MRI data on product composition together with a microwave field simulation, it was possible to simulate and validate the heating of a model food and a chicken wing (Knoerzer et al., 2008).

In a recent study (Zhu et al., 2015; Gulati et al., 2015), a drying model was established using the volume averaging method for heat and mass transfer calculations as well as a full microwave field calculation, capillary driven water flux and consideration of vapour and air pressure. They validated the model for potato spheres of different sizes. Some changes of material properties were introduced based on empirical calculations. Measured were the surface temperature during drying, the spatial temperature distribution in the middle plane offline via infrared camera and one fibre optical temperature probe as well as a fibre optical pressure probe inside the potato and an integral moisture measurement with an online balance. Focusing effects were highest for the 3 cm diameter potato and very low for small spheres of 1.2 cm diameter. The small spheres experienced a very even heating. In summary, good agreements were found between the model predictions and the measurements concerning absorbed power, temperature and pressure.

A different strategy on "process and product modelling" is the use of *artificial neural networks (ANN)*. The basic idea is to have a self-learning system based on empirical correlations between the input and output parameters. A comprehensive review on these techniques used in drying modelling is given by (Aghbashlo et al., 2015). In the concluding remarks, the authors state, that these methods principally have a high potential to model, optimise, monitor, and control drying processes and even outperform other available techniques, but they also advice that ANNs are preferentially used as complement and not as alternative techniques. A major

drawback from a scientific point of view is that even if an ANN can predict a wanted parameter in dependency of several process parameters very well, we cannot learn anything on the physical principles of this correlation as it is completely empirical and not extractable from the ANN in detail. This aspect may jeopardise the use of ANNs in the real-world drying technology (Aghbashlo et al., 2015).

From a practical point of view, none of the physical or ANN based modelling attempts has achieved much significance in the food industry. So far it has always been easier to use trial and error, based on former practical experience and to use simple empirical calculations which correlate well with the observed parameter changes than to collect all necessary information on thermo-physical properties, dielectric properties, field distribution in the drier, etc.

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