

# Microwave Vacuum Drying for advanced Process Technology

## Rapid and gentle Vacuum Drying for thermo sensitive Products with low thermal Conductivity

### Introduction

Heat-drying has become important in almost all areas of industrial processing. Apart from the popular conventional procedures based on conduction, convection, or infrared radiation, heat-drying utilising microwave energy is an attractive solution to many problems in process technology. In microwave drying, heat is generated by directly transforming the electromagnetic energy into kinetic molecular energy, thus the heat is generated deep within the material to be dried. Especially in vacuum drying the fact of volume heating gives an enormous importance in order to dry bulk and viscous products with low thermal conductivity.

### Basics of Microwave Drying

The applied microwave energy will be transformed depending from the depth of vacuum directly into evaporation heat.

If all the microwave energy is absorbed, the power density can be calculated using the following formula

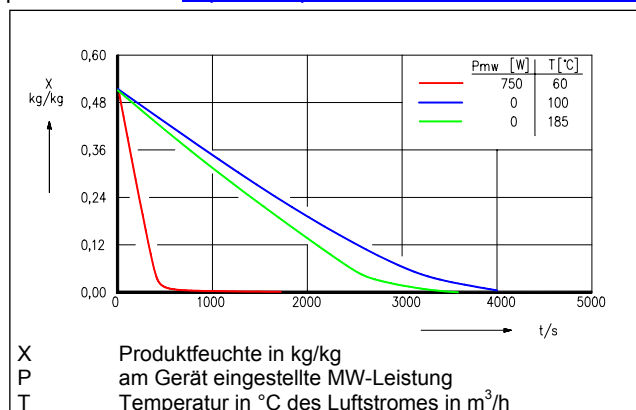
$$P = 2 \cdot \pi \cdot f \cdot E^2 \cdot \epsilon_0 \cdot \epsilon'_r \cdot \tan \delta \quad \text{in } W/m^3 \quad (1)$$

The penetration depth can be calculated with

$$d = \frac{\lambda_0 \sqrt{\epsilon'_r}}{2 \cdot \pi \cdot \epsilon''_r} \quad \text{in cm} \quad (2)$$

- $f$  frequency, measured in Hz
- $\epsilon_0$  absolute dielectric constant (DC) =  $8,85 \times 10^{-12}$  As/Vm
- $E$  electrical field strength, measured in V/m
- $\epsilon = \epsilon_0 \cdot (\epsilon'_r - j \epsilon''_r)$  complex dielectric constant  $\tan \delta = \epsilon''_r / \epsilon'_r$
- $\delta$  dielectric loss angle, measured in degrees
- $\lambda_0$  wave length, measured in,  $\lambda_0 = c / f$

[ Reference see company brochure „Dielectrical heating using Microwaves“ (1) or see also the abstract published under <http://www.pueschner.com/de/basics/index.html> ]



**Figure 1.** Comparison between microwave and conventional heating drying Al<sub>2</sub>O<sub>3</sub>-Granular, 2mm

The most important parameter for microwave heating is the dielectric losses of the product to be dried. For a big range of products the dielectric losses were measured by Hippel [2]. If no dielectric losses are known from literature, we are able to measure these properties in our laboratory.

## Fields of Application for Microwave Vacuum Drying

- **Pharmaceutical Industry**
- **Food Industry**
- **Chemical Industry of Fine Chemicals**
- **Semi-Conductor Manufacturing**

## Advantages

As compared to conventional drying methods, microwaves penetrate into much greater depths. This so-called volume heating has the following advantages:

1. a temperature gradient directed towards the surface, i.e. temperatures inside are higher than on the outside giving rise to a higher partial pressure that drives the evaporating liquid to the surface
2. consequently, the superficial layer does not dry out completely and the surfaces remain permeable
3. the liquid evaporating inside the product is emitted through the pore structure of the solid material's macro-capillary system, resulting in a high drying velocity
4. the heating of water and most organic solvents occurs selectively - due to the greater dielectric losses of water as compared to the product to be dried
5. swift and thorough drying of moist products with low thermal conductivity
6. stationary drying of thick layers without frictional losses
7. high total efficiency of energy application
8. high-speed control of the energy transport
9. short processing times, i.e. suitable for automated manufacturing

Together with the appropriate vacuum drying technology the following advantages for the product are available:

1. Low drying temperatures treat the product gentle
2. No oxygen attack for the product
3. Highly nutritious instant properties
4. Better flavour
5. Less hygroscopic final product
6. Minimal product losses
7. Static drying of thick layers without frictional losses, therefore no mechanic stress for the product

The microwave vacuum technology results in unique process technology giving the following benefits:

1. Local controlled energy zones with high controller speed
2. Defined and short residence times of the product to be dried
3. Cooling zone at the outlet for viscous products
4. Closed system
5. Simple setting of different plant operation modes for every product
6. Continuous operation mode possible
7. Short process times, high efficiency and full automated guarantees an economic operation
8. Service friendly because of modular system and full-automated cleaning

## Special Conditions for Vacuum Drying

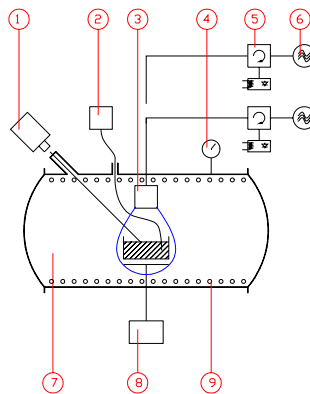
For integration of microwave components into a vacuum system, some important high frequency specific items have to be taken into consideration. These are in particular:

- Dielectric properties
- Installed transport systems
- Product throughput rate
- Drying parameter
- Used vacuum, in particular the depth of the vacuum

A homogenous microwave energy distribution over the cross section of the product bed is a significant requirement. Especially in applications of end-drying or in applications using products with poor dielectric losses, special microwave antenna systems are required in order to achieve even temperature and drying results.

Also peaks in the electric fields strength have to be avoided using high quality DC microwave power supplies, because the breakdown field strength is reduced by the vacuum. If the breakdown field strength is exceeded, the results are sparks and plasma.

The process parameters as well as the microwave applicator or microwave antenna system have to be evaluated using microwave vacuum trial plants. Figure 2 shows a microwave trial plant, which can be equipped with different applicators or antenna systems. Using an infrared and fibre-optical temperature measurement system, core and surface temperatures can be measured. Additional the weight loss, pressure and the absorbed microwave energy can be measured also.



### Legends:

- 1) Pyrometer (0..1000°C)
- 2) Fibre-optical temperature measurement system
- 3) Microwave antenna
- 4) Pressure measurement
- 5) Circulator (magnetron protection) incl. Measurement of the reflected power
- 6) 1.2kW/2450MHz Magnetron incl. DC high-voltage power supply.
- 7) Vacuum vessel ca. 200l
- 8) 10kg load cell, 10.000 digits
- 9) wall heating

**Figure 2.** Microwave Vacuum Trial Plant *μWaveVac0150*

In order to perform trials for evaporation of solvents, the vacuum vessel can be inert using nitrogen. The procedure of applying nitrogen is observed with an oxygen measurement device.

Based on the results of these trials an up-scale plant concept for the final production throughput rate can be made.

## Combination of an Air/Vacuum/Microwave 2.4qm Disk Dryer under Explosive Protection and cGMP

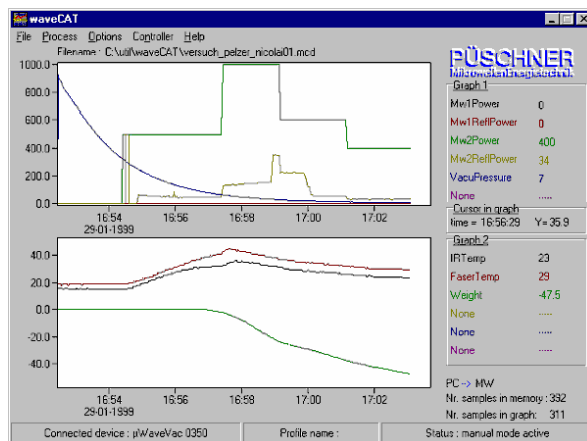
For drying of solvents in pharmaceutical products the microwave vacuum dryer ***µWaveVac0209*** was developed. As a 2.4qm disk dryer it can be used with circulating air heating as well as with vacuum. In order to cover the requirements of explosive protection, the procedure of applying nitrogen to inert the atmosphere inside the vacuum vessel is controlled with a redundant oxygen measurement system.

The evaporation of solvent e.g. ethanol is measured with an online infrared measurement system in order to avoid critical gas concentrations within vacuum vessel. An inbuilt online load cell as well as an infrared and a fibre-optical enable a controlled drying process using a PLC system. The microwave vacuum dryer fulfils the requirements of cGMP.

A flexible PLC with a processing control language guarantees the reproducibility of each drying batch. In end-drying the absolute deviations from all product positions on all disk layers are smaller than 0.2%. A typical drying curve is shown in Figure 4.



**Figure 3.** Microwave Disk Dryer for combined Air Circulation and Vacuum Mode ***µWaveVac0209***



**Figure 4.** Visualized Drying Curve of a thermo sensitive chemical Raw-material

## Continuous Vacuum Drying using the Microwave Belt Dryer $\mu$ WaveVac1290

Using conventional vacuum dryer the product is heated using contact heating within several heating zone. Heated plates are used as heat transfer medium using pressure water, steam, oil or electrical sources.

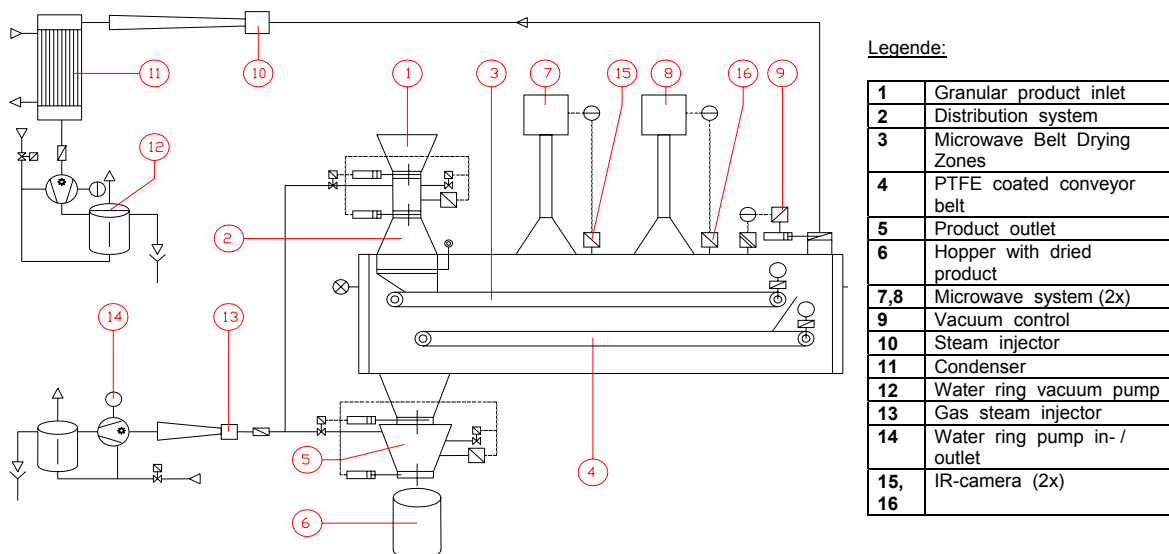
Compared to the above mentioned conventional heating systems microwave is the better alternative. Microwave vacuum belt dryers are used for the continuous and automatic drying of temperature sensitive products with low thermal conductivity such as herbal extracts, food, pharmaceutical and chemical products. The drying process is adapted to the desired dry product quality such as final solids content, solubility, density and others.

The feed of solids is effected by means of a suitably designed feeding device with attached and product orientated metering and distribution set-up in the dryer. When feeding wet product a metering pump and an oscillating feeder are allocated. The movement of this feeder is adjustable during operation. Therefore an easy optimising is possible.

Pump able products pass normally a high viscose and sticky phase during drying. Towards the end of the drying and influenced by the steam bubbles a dry cake is formed. This cake is brittle and is broken at the end of the belt and, if required, granulated.

The vacuum belt dryer consists of a casing with built in transporting belt. The belts are made from selected PTFE coated glass fibres. For the continuous and automatic operation of such a plant, a belt control device that runs reliably for a long period is imperative.

For the product quality to be achieved, the heating temperature during drying is of essential influence. In the tradition vacuum belt dryer normally the belts run over 3 or 4 heating zones and at the end through a cooling zone. These are fed with hot water, steam or thermo oil. With using microwave heating careful arranged microwave heater with infinitely variable power making sure an optimum energy transfer into the product. The temperature profile may be selected to the needs of the product. The product temperature will be controlled with infrared thermometers installed on top of the dryer. When the product temperature will climb up in a critical value, the microwave power will be reduced automatically. Microwave heater usage normally is 1.2/2/3/6kW-2450MHz. The design of the size will be according to the necessary water evaporation



**Figure 4. Process Flow Diagram  $\mu$ WaveVac1290**

The normal working vacuum pressure for drying is in the range of 10 to 50mbar abs. The outlet sluice chamber can also be used as a product buffer hopper. The material is discharged in a cyclic way during continuous operation. The vacuum for the inlet and outlet sluice chamber is provided by a separate vacuum system.

The complete microwave vacuum dryer is controlled by a PLC system. Data recording and process visualising as well as active process diagrams can be managed with a WINDOWS PC. Standard protocol interfaces based on RS232 or Ethernet are available. As a http-server the microwave plant can be set-up in a TCP/IP network and allow *web enabled engineering* as well as remote control access via Intra- and Internet.



**Figure 5.  $\mu$ WaveVac1290**

For cleaning two options are available:

- Integrated cleaning
- External cleaning by removing the complete transport system

The continuous microwave vacuum dryer provides optimal setting in order to achieve best drying results. The following parameters can be adjusted:

- Flow rate for granular or for viscous products
- Nozzle section, nozzle distance to belt, charge width of viscous products
- Foam of the product while charging the belt
- Closed product foam
- Inlet cycle time and product rate for granular at the inlet sluice chamber
- Belt speed
- Vacuum
- Microwave power
- Outlet cycle time

## Summery

Microwave Drying has a big advantages compared with conventional drying, because in microwave drying, heat is generated by directly transforming the electromagnetic energy into kinetic molecular energy, thus the heat is generated deep within the material to be dried.

Especially in microwave vacuum drying this advantages has a big significance for viscous and bulk products with poor thermal conductivity.

The described microwave vacuum technology is used for high-end drying applications of thermo sensitive products in order to achieve higher product qualities and shorter drying times.

Using appropriate process control even applications with explosive protection can be managed.

As microwave vacuum technology always has to cover specific application requirements due to the transport system, microwave applicators, dielectric losses of the product, the effort on development work is quite high. Therefore this technology is mainly used for high value products.

## References

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- (2) A.R. von Hippel, Dielectric Materials and Applications, The Technology Press of. M.I.T., 1954