Temperature Distribution in the Humid Powder Beds Dried in Microwave Field and Warm Air

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Abstract* – *In an experienced system (2.45 Ghz,90W) have been monitored the internal temperature distributions of humid pharmaceutical powder samples, dried in microwave field and combined microwave-hot air. It has been found that temperature variation depends on material, dielectric physical and thermal properties of solvent and powder. The diagrams have been also dependent on the layer sizes (area and thickness), a rising of area size and a decrease of thickness causing lowed temperature of the product. The surface temperature remains lower than central temperature during all the processing period, differential on sample thickness rising with the humidity content reducing.

Keywords*: *Drying, Microwave, Hot Air, Powder.

I. INTRODUCTION

The drying is a complete operation in pharmaceutical powder manufacturing. Inside the parcel are used many types of driers, however recently has been renewed the interest in microwave application during drying processing. Generally, the products are thermo-labile, such a degradation of material being unacceptable. Therefore, the control of heating uniformity and thus the product temperature, is of great importance for successful application of microwave energy.

The temperature distribution in a product exposed to microwaves is governed by the interaction and radiation absorption by the dielectric environment, and by accompanied transport processes due to electromagnetic energy dissipation in heat (*Oliveira*, *2002*, [4]). This is controlled by a number of product and process variables related, including the penetration depth, dielectric and thermal properties of material and microwave field distribution in oven; these are only partly understood. *Roques and Zagrouba* (1992, 1997) has determined experimentally, and modeled the convention heat transfer diagrams using microwaves for polymeric non-porous gels [5]. Authors have considered that the process must be almost entirely controlled by heat producing and heat transfer rate. Especially the presence of short accelerated period, superposed on a drying curve (which positioning has been dependent on sample nature and geometry) was attributed to heat wave reaching the surface.

Tulasidas et. al. (*1995*) have examined the effect of processing parameters, including air speed and temperature on behavior at combined microwave-hot air drying of grapes. The drying at higher air temperatures produced higher temperature of the material , which have risen air flows producing rapid heat transfer at the surface and therefore the fruit temperature lowered [7].

Lu et. al. (*1999*) determined experimentally and developed a half-empirical model to describe the temperature variations during microwave of food slices [11]. Three distinct phases has been noticed:

(1) the rising of initial temperature due to microwave power absorption;

(2) the constant temperature period, with significant reducing of humidity (all absorbed electromagnetic power contributes to water evaporation);

(3) temperature rising phase with humidity reduction. The maximal temperatures values has been found dependent on microwave power -mass ratio and sample thickness.

Jumah (*2001*) has experimentally and numerically investigated the heat transfer phenomena during combined microwave- hot air drying of a moist layer of wheat grain. Temperature variations in wheat particles has been found dependent on air characteristics. An air temperature rising produced the sample temperature rising, while speed rising has facilitated a temperature decrease [2].

For adequate design of dryer and process control it is necessary to know the material temperature and humidity amount and the dependence on process parameters. Therefore, the aim of this work is to examine the effect of produce parameters and of the process itself on internal temperatures of pharmaceutical powders exposed to microwaves- hot air drying.

II. MATERIALS AND METHODS

A. Materials

The powders which are going to be analyzed are Aspirin (AP), Lactose (LT), and Paracetamol (PM).These has been chosen in order to represent a large range of materials found in pharmaceutical product manufacturing, and solvents like Water (W), Methanol (M), Acetone (A).

B. Experienced equipments

The microwave drying system used in this work is composed of a microwave applicator (the strictly speaking classical oven) provided inside with a turning/fix plate made of Teflon, linked directly to an electronic balance (Figure 1). The microwave generator control unit transmits the function control to microwave generator working on a frequency of 2.45 GHz and variable power up to 1000W. The waves transmission from magnetron to cavity is realized by a square wave guide. The installation is protected by possible accidental leakages of reflected power by a connection to a water network. Through the interface circuit the experimentally obtained data are transmitted to a computer to be stocked and processed, later being printed.

For a maximal efficiency of the drying process in microwave field, the shown installation includes also hot air electrical generator type BOSCH PHG 600-2 CE, with a maximum power of 2000 W and a air flow of (300÷500) l/minute used to combined microwaves-hot air heating. The air pipe precisely dimensioned, it 's attached to the side part of oven. The air temperature is between the interval (20-100 ± 5)°C.

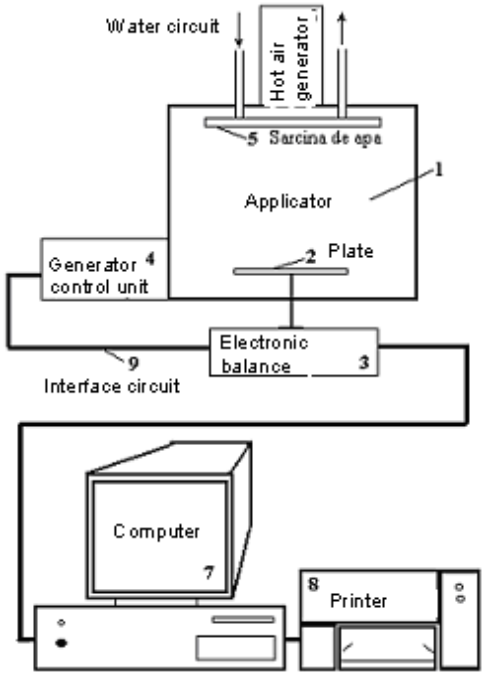

Fig.1. Block diagram of test stand

The electronic balance model, SCALTEC SPO 61, is provided for a maximum weight of 3.1 kg with a precision of 0.1 g and a conversion analogical output of digital signal to be transmitted to computer in order to be processed and interpreted.

C. Experienced procedure

The internal material temperature has been determined during the microwave processing only (90W) and by microwaves-hot air. Before the development of each experiment, the microwave oven has been preheated to nominal power (650W) for 5 minutes using 500ml of water charge (*Lu et. al.1999)* and the hot air system has been left to stabilize itself, in selected conditions for ten minutes. A water charge of (75g) has been placed in the microwave cavity to provide a heating load sufficiently to protect the magnetron of over-heating, especially during the last drying phases. For each experiment has been prepared a sample of 100g (dried based, water), using a specific powder and solvent, which later has been placed in oven. At period of five minutes in the entire drying process (till the material has lost 95% of humidity) the sample has been removed, the temperature and weigh measured and then stirred for 15s. The temperature has been measured in the sample geometrical center using an infrared radiation thermometer.

This procedure has been applied to study the effect of product characteristics and processing on central temperature. The samples humidity amount has been determined at the start and end of each experiment.

III. RESULTS AND DISCUSSION

A. Processing conditions and material characteristics

A typical curve of central temperature for a sample lactose-water is shown in Figure 2. The data are graphically presented with rate curve corresponding to drying to allow the examination of temperature diagram in different drying phases. Independently of processing technique (microwaves or microwaves-hot air) and the powder type, the generalized temperature curve is composed of three phases. At start a short period of pre-heating or 'heating' can be observed. A high amount of microwave energy is absorbed by the mixture due to a high presence of solvent inside the sample, with a loss factor relatively higher than that of powder. All electromagnetic power absorbed is transformed in thermal energy and consequently a quick temperature rising is noticed. Once the humidity steams pressure in material exceeds that of the environment, the humidity is removed, but at a relatively limited rate.

During the next phase " constant speed" the temperature remains relatively constant and the quick loss of humidity is observed. This suggests that the absorbed energy is balanced by energy losses associated to humidity evaporation and convention cooling of surface. The microwave component produces the sample temperature rising over the air temperature in humid state (the superior limit pre-established during only convection drying), as it results from the experienced data.

In a complementary study have been examined the loss factors in dielectric of some powder systems. Particularly, the value of dielectric properties for lactose have been found greater to a critical content of humidity than at higher humidity levels (*Mc Loughlin et. Al.,* 2003, [3]). This would determine an absorbed power rising and

followed by sample temperature rising observed in this humidity area.

During all period of rate decrease, the sample temperature gradually decreases. This shows the reduced power absorption due to solvent decreasing in mixture, the absorbed energy being less than losses due to cooling by evaporation and convention. The temperature decrease shows the rate decrease which emphasizes the fact that the presence of solvent is the control factor in power absorption and that the under the critical humidity content the dielectric loss factor doesn't significantly vary with humidity content.

Approaching to the end of drying process, the sample temperature decreases to a constant value which amount is dependent on processing conditions. This is a little bit higher than hot air temperature, showing that the mixture still absorbs microwaves. The observed temperature trend is important for product quality, being more probably the degradation takes place in initial phase of processing.

The general shape of temperature diagram is comparable with that noticed by *Roques and Zagrouba* (*1992*) and *Tulasidas et. al.* (*1995*) during the microwave convection drying of gelatin and grapes respectively [5] ,[6], [7].

A specifically feature of temperature curve of lactose is that at medium humidity content in rate decrease regime, it appears an obvious sample temperature rising and humidity removing rate rising (Figure 2). This may show the variation of physical lactose sample characteristics which endure a transition from a muddy paste to a more humid one which tends to agglomerate in "a solid ball" even at stirring and which allows a heat accumulation. *Roques and Zagrouba* (*1997*) have noticed also a quick temperature rising associated with a pick superposed on the side of decreasing rate side of drying characteristic during the microwave convention drying o poliacrilamidic gel.

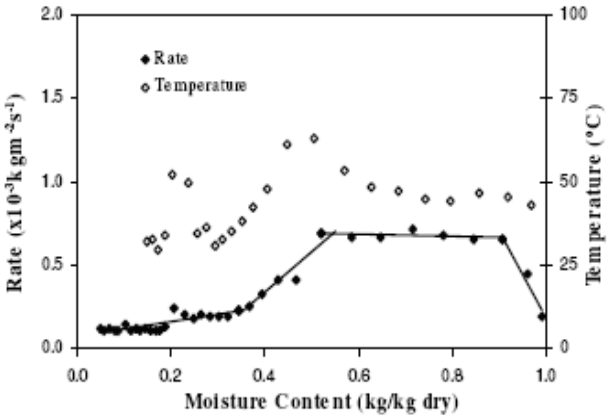

Fig. 2. Dependence temperature-humidity for a lactose sample dried in microwaves.

The temperature in the center of lactose-water samples submitted to microwave-hot air drying are compared with diagrams of only microwave drying in Figure 2, and in Table 1 is given a summary of medium and central temperatures. The insert of environment air over the sample surface produces the material temperature decrease during entire drying process. This suggests that prevails the combined effects of cooling by evaporation and heat transfer from sample to air (T_{sample} higher than T_{air}), the system air being used as effective cooling environment. However, pre-heating the air, the sample temperature decreasing becomes less obvious than temperature during microwave drying. This is due to reduced convection heat lost by air temperature rising. Independently of convection air temperature the value of temperature decreasing is more significant in constant rate regime. In fact, the temperature curves during the decreasing rate period in microwaves and microwaves-hot air (60 °C) drying are comparable. As expected, the air pre-heating induces also the rising of maximum material temperature (Table 1). *Tulasidas et. al.*(1995) and *Jumah and Raghavan* (*2001*) noticed higher product temperature at combined drying at higher air-temperature [2], [7].

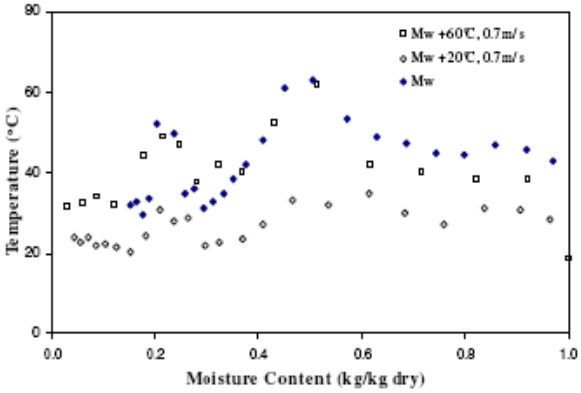

Fig. 3. Temperature distribution in center of lactose-water sample dried in microwaves-hot air.

The pharmaceutical industry requires the powder and solvent processing with some physical and thermal properties of dielectric.

The central temperatures in the selected powder-solvent systems have been monitored during the microwave drying. A summary of central medium and maximum temperatures is given in Table 1. The study of temperature variation, i.e. constant temperature amount, the humidity content at which for which the 'maximum' temperature takes amplitude and 'pick' duration, the temperature decreasing rate in phase of rate ace, the decreasing.

At the final material temperature shows that it is specific to system [8]. In later valuations of temperatures of different powder -solvent system suggests that the amount is more dependent on solvent, namely on dielectric properties (loss factors) and thermal (latent heat of vaporization, boiling point) (*Perry and Green, 1994*) than the powder. No matter the solid, the mixture water content involves the highest temperature rises, followed by samples damped with ethanol and acetone. Except the first samples, the temperatures remain under the acceptable limit of 60°C during the processing [1]. See Table bellow.

Table 1. A summary of central medium and maximum temperatures

Powder	Solvent	Microwave		Microwave-Convective (20°C)		Microwave-Convective (60°C)	
		T_{avg} (°C)	T_{max} (°C)	T_{avg} (°C)	T_{max} (°C)	T_{avg} (°C)	T_{max} (°C)
Paracetamol	Water	39.4	76.1	26.9	43.1	35.8	54.1
	Methanol	30.8	47.3	22.9	35.5	31.0	43.8
	Acetone	29.1	49.0	24.3	28.9	28.8	39.5
Aspirin	Water	46.4	75.6	27.4	55.8	34.0	46.2
	Methanol	29.8	42.8	18.2	20.5	21.4	25.1
	Acetone	28.3	43.3	14.9	16.8	19.5	25.8
Lactose	Water	42.0	63.1	26.3	34.9	40.0	61.7
	Methanol	29.8	55.3	17.8	25.1	22.8	28.5
	Acetone	23.1	38.9	12.9	14.7	17.5	20.1

IV. CONCLUSIONS

During the drying operation, the sample and equipment sizes may vary. No matter the sample geometry, the temperature variation follows the general pattern discussed previously. For both drying procedures, a sample surface rising is accompanied by an internal temperature decreasing. This suggests that if the diameter is bigger the evaporation cooling rises (due to the bigger evaporation surface with a rate higher) than the internal heat generation. The container diameter increasing is accompanied also by a decreasing of surface flow [9]. As expected, the air insertion in microwave system facilitates a sample temperature decreasing, however the decreasing amount depends on air properties and the sample surface size.

Jumah and Raghavan (2001) noticed at microwave-hot air drying of wheat grains internal temperatures higher than at the surface. In addition, Chamchomg and Datta (1999) communicated that the temperature amount and sample temperature difference vary with load geometry [2]. At high humidity contents, the dielectric loss factor of material is higher, following a less microwave penetration depth and consequently, a decreasing of temperature gradient (considering that total sample thickness ≤ penetration depth). As the material dries the penetration depth rise ,however, with the temperature difference rising [10].The air flow insertion in microwave system decreases the sample internal temperature, decreasing amount being determined by air characteristics and the layer geometry.

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