# The Influence of Granulometric Properties and Drying Conditions on the Drying Kinetics of a Nonhygroscopic Material

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Original scientific paper
Received: December 3, 2008
Accepted: June 9, 2009

The influence of granulometric properties of the material and of the operational conditions on the drying kinetics of nonhygroscopic material was studied. Four fraction sizes of dolomite were chosen and dried in four dryers by different heating methods. Dolomite was dried in a microwave dryer under different microwave heating powers. During convective drying the effects of temperature and superficial air velocity were monitored. Vacuum drying was carried out at different temperatures and pressures.

Page's model has shown to be successful in describing the drying kinetics throughout the entire drying operation, irrespective of the heating mode.

Heat intensity ($\vartheta$, $P_m$), superficial air velocity and particle size had influence on the drying kinetics. The process was facilitated by higher temperatures and superficial air velocity, higher intensity of microwave radiation and bigger particles. Change of pressure in the vacuum dryer had no significant effect on the drying kinetics under the chosen operating conditions.

*Key words:*
Convective drying, drying kinetic, mathematical models, microwave drying, vacuum drying

## Introduction

Drying is a very important thermal separation operation applied by the majority of industries. Currently there is almost no product which has not been dried at least once in its lifetime, either as a raw material or as a final product.[1] Given the difference in the drying materials, especially when it comes to transport properties of the wet materials, the dryers appear in various types, which imply their design and transfer of heat to the wet material. Heat is transferred to the wet material either in a convective or a conductive mode or by radiation (microwave radiation, IR radiation, etc.).[2] Various modes of heat transfer during drying are being combined recently[3–11] in order to shorten drying time and to achieve uniformity of the material properties (uniform moisture content).

According to Faust[12] wet solids are classified in two basic groups. The first one comprises particle or crystalline solids, which retain moisture in the open pores between the particles. During the operation of drying, moisture removal has no effect on the material properties. Consequently, selection of a drying mode and conditions has no significance for the finished product quality. Such materials dry very quickly to low moisture content. The second group of wet solids comprises "fibrous, amorphous and gel-like" materials that are either dissolved or that trap the moisture within the very tiny pores. Properties of such materials are very much affected by drying because of the occurring condensation. Moisture and temperature gradients may cause deformities or solidification of the surface. In other words, the materials dried are either nonhygroscopic or hygroscopic.[13] In the view of the previously mentioned, it is very important to meticulously choose drying conditions.

When it comes only to the properties of a dried material, the mode of heat transfer to the wet material and various processes occurring during drying (transfer of momentum, heat and mass, and physical changes), this operation is clearly a very complex one.[1] Its mathematical modelling is extremely complex. Hence, the laboratory-scale experiment and data collection are indispensable for the selection and design of the scale-up equipment.

Presently an increasing number of authors have been researching the effects of various heating modes[14–19] on the drying kinetics and on the consequential qualities of the finished material being dried. This work was also aimed at choosing the optimal drying method for a nonhygroscopic material, dolomite, of different granulometric properties.

### Convective drying

This type of drying is still most frequently applied irrespective of its being least convenient from the energy and ecological aspects.[1–2] It requires large

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amounts of energy to heat the air, whereas humid and warm air at a dryer outlet is usually released into the atmosphere. During convective drying the heated air transfers heat to the wet material. Heat is used for evaporation/drying of the material's surface moisture. In this process, the air carries heat and draws off moisture. Such dryers are called direct dryers. Heated air usually flows above the surface of the wet material. The operation can, however, be accelerated by passing the air through the bed of particles or by increasing the superficial air velocity so as to achieve fluidization which speeds up mass and heat transfer between the wet material and air.

## Vacuum drying

Vacuum drying is applied to the materials sensitive to high temperatures. Vacuum draws off moisture from the materials and simultaneously prevents explosion or oxidation, which may occur if some of the materials or solvents come in contact with air. Taking into account the requirement for the system to be closed when the process takes place at low pressure, one of the advantages of this drying is the use of hazardous materials (toxic materials and explosive solvents). It is also applied to recover the solvents and to dry to very low moisture content. In addition, drying at low pressure is less energy- and product-consuming, attributed to contamination, oxidation, and change in colour or chemical composition.[4]

Unlike convective dryers in which the material is placed in a current of hot air, vacuum dryers are indirect drying applications. In other words, heat is transferred to the wet material by conduction over the carrier's surface and radiation. Knowledge of the heat transfer mechanism is crucial for understanding the advantages and disadvantages of vacuum drying and selection of a dryer. Unlike with the convective dryer, by pressure control in a vacuum dryer it is possible to increase the efficient temperature difference of a given operation, which shortens the drying time.

## Microwave drying

High-frequency drying enables mild drying of the materials and avoidance of deformities to the products.[17–18] Therefore, it is applied to high-quality materials such as wood, ceramic products, foodstuffs and pharmaceutical products. Microwave drying yields the product of uniform moisture distribution and, consequentially, of better quality. The process is fast and gives a sterile dry product. With conventional drying methods, drying time is limited by the rate of heat flow from the material's surface to its interior, as defined by its properties (heat capacity, thermal conductivity, density and viscosity). Surface heating is slow but also uneven, the exposed surface being significantly warmer than the interior, which has direct effect on the product's quality. With microwave heating, the whole volume of a wet material is being heated at almost an equal rate. Energy is transferred through the material by electromagnetic waves, rather than as heat flow, and so the heating rate is not limited while heating uniformity is improved. Drying time is reduced significantly compared to the conventional drying methods. Given the fact that volumetric heating does not depend on the convective or conductive heat transfer, microwave heating is applied whenever conventional methods are unsuitable. Microwave energy is absorbed at the points of higher moisture content, which yields a uniform distribution of temperature and moisture content. In addition, microwave dryers are environmentally friendlier. Their fundamental disadvantage, in addition to higher investment costs, is the penetration depth achievable with microwave energy, which depends on the frequency, temperature and dielectric properties of the material.

## Mathematical models

Drying kinetics of dolomite was described in the present work with the modified Page model.[20]

Page model:[21]

$$\frac{X - X_{eq}}{X_0 - X_{eq}} = e^{-k \cdot t^n} \tag{1}$$

Page's model is modified by introducing substitution:

$$k = \left(\frac{1}{t_k}\right)^n \tag{2}$$

$$\frac{X - X_{eq}}{X_0 - X_{eq}} = e^{-\left(\frac{t}{t_k}\right)^n} \tag{3}$$

This model was chosen because of its applicability to describe drying kinetics of various materials and the heating mode for the wet material.[16,21] The material used in this work was nonhygroscopic and it was interesting to see whether the model was suitable for the mentioned purpose.

The correlation index, $\rho$, and standard deviation, $\sigma$, were calculated by the equations:

$$\rho = \sqrt{1 - \frac{\sum_{i=1}^{N} (X_{calc} - X_{\exp})^2}{\sum_{i=1}^{N} (\bar{X} - X_{\exp})^2}} \tag{4}$$

$$\sigma = \sqrt{\frac{1}{N} \cdot \sum_{i=1}^{N} (X_{\exp,i} - \overline{X})} \quad (5)$$

$\overline{X}$ is the arithmetic mean of the values $X_{\exp,i}$, defined as:

$$\overline{X} = \frac{1}{N} \cdot \sum_{i=1}^{N} X_{\exp,i} \quad (6)$$

## Experimental setup

The measurements were carried out in the laboratory dryers (Fig. 1) under the drying conditions shown in Table 1. Wet samples were prepared with the addition of the preset water volume to a completely dry dolomite (dried at $\vartheta$ = 105 °C) so that the material's initial moisture content was $X_0$ = 0.1583 kg kg$^{-1}$. The measurements were being carried out until complete loss of moisture, given that the material was nonhygroscopic.

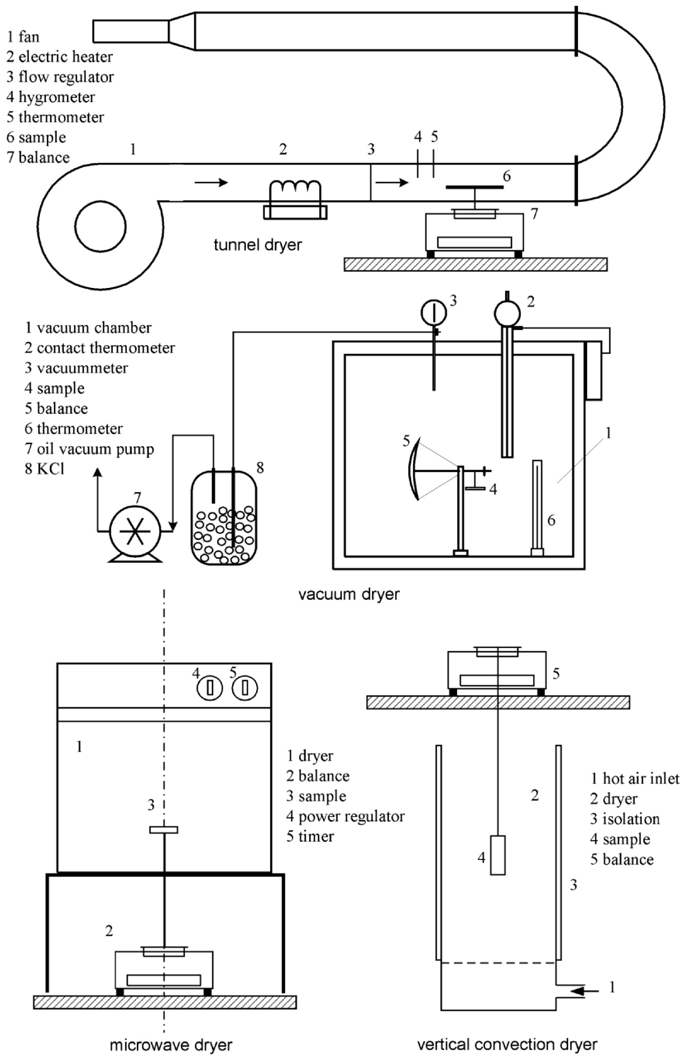



Fig. 1 – *Laboratory driers*

Table 1 – *Drying conditions*

| Dryer | $\vartheta$/°C | $p_{abs}$/bar | $v$/m s$^{-1}$ | $P_m$/W kg$^{-1}$ |
|---|---|---|---|---|
| vertical convection | 40 | 1 | 1.55 | – |
| | 60 | | 1.85 | |
| | 80 | | 2.00 | |
| | 100 | | 2.20 | |
| tunnel | 40 | 1 | 0.87 | – |
| | | | 1.09 | |
| | | | 1.24 | |
| | | | 1.55 | |
| microwave | – | 1 | – | 22.77 |
| | | | | 45.54 |
| | | | | 68.31 |
| | | | | 85.39 |
| | | | | 113.85 |
| vacuum | 40 | 0.3 | | – |
| | 60 | 0.6 | | |
| | 80 | 0.9 | | |
| | 100 | 1 | | |

### Material

Dolomite was chosen because of its good chemical and physical properties. This is known to be a nonhygroscopic water-insoluble material. Its dielectric constant ($\varepsilon$ = 6.8–8.0 F m$^{-1}$) is markedly lower than of water. Dolomite is non-porous and available in a wide range of particle sizes.

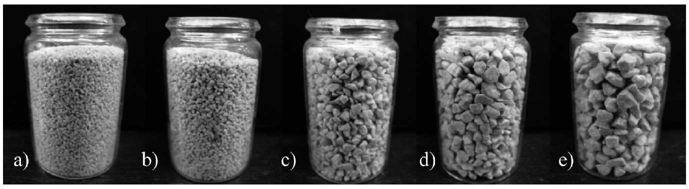



Fig. 2 – *Size fractions of dolomite*
*a) 600–850 μm, b) 850–1180 μm, c) 1180–2360 μm, d) 2360–3350 μm, e) 3350–4500 μm*

Upon passing through the sieves of different mesh size five various particle sizes of dolomite were chosen (Fig. 2). The smallest fractions ($x$ = 600–850 μm) were dried only on the vertical convective dryer because after sifting there was scarcity of the original sample for the experiment in other dryers. Porosity of the particles layer was calculated from the available data on real and bulk density (Table 2).

Table 2 – *Porosity of the fixed bed of particles*

| $x$/$\mu$m | $\varepsilon$ |
|---|---|
| 600–850 | 0.4856 |
| 850–1180 | 0.4822 |
| 1180–2360 | 0.4631 |
| 2360–3350 | 0.4398 |
| 3350–4500 | 0.4377 |

## Results and discussion

Drying kinetics of a fixed bed of particles of a nonhygroscopic material, dolomite, was studied in the laboratory convective (vertical and tunnel), vacuum and microwave dryers under different operation conditions. The fractions of dolomite were medium-size i.e. $x$ = 725, 1015, 1770, 2855 and 3925 μm.

The initial moisture content ($X_0$ = 0.1583 kg kg$^{-1}$) and mass of the dry samples were equal in most of the experiments to enable finally the comparison of various heating modes of the material during its drying. Some slight deviations occurred, however, with the largest fractions dried in the vacuum and vertical convective dryer due to water escape through the porous carrier. Carriers of the samples in the microwave dryer and in the tunnel were non-porous. Due to the tunnel design and possible overheating of the device there was no monitoring of temperature effect and all measurements were carried out at $\vartheta$ = 40 °C. Mass of the wet samples was also much higher in the tunnel ($m$ = 294.22 g) than in other dryers ($m$ = 11.41 g). Therefore, specific drying rate, $R$, in kg m$^{-2}$ min$^{-1}$ was applied for comparison purposes.

Based on the measurement data about changes in the wet material mass during drying its moisture contents $X(t)$ were calculated and shown in relation to drying time of all dryer types, but under various process conditions (Figs. 3–6). Given nonhygroscopicity of the dolomite's particle layers, all moisture during drying was eliminated to the balanced moisture content of $X_{eq}$ = 0. Material moisture content changed linearly with time during almost the entire drying process of dolomite in the vertical convective dryer in all performed experiments. That meant that during the entire process the conditions for maintenance of constant drying rate were being satisfied. In fact, heated air in the vertical convective dryer was flowing through the layer of particles, by which air current was constantly transferring moisture to the evaporation surface. In the remaining three dryers, there were very short periods of a falling rate period during which the retarded small amounts of moisture were drawn off. That was definitely due to nonporosity of dolomite particles i.e. material's nonhygroscopicity and to the pores structure (intracellular space). All moisture was free and the pores were large enough not to exert significant resistance to the flow of moisture. However, due to particles being of an extremely irregular shape (Fig. 2) there was a risk of formation of denser local packing (occasional appearance of markedly tinier pores) which would slow down drying rate. Given the fact that energy in microwave dryers is channeled to a material of higher dielectric constant i.e. to water, moisture content in the material under drying is being gradually decreased. Consequently, heating intensity of the particle layer is smaller, which results in the falling rate period. In view of the slight deviations in the initial moisture content, especially in the largest particles, dependencies of moisture content in the material are shown in the dimensionless form ($X(t)/X_0$).

Fig. 3 shows the effect of heating intensity on the drying kinetics of dolomite $x$ = 1180–2360 μm fraction in the vertical convective (Fig. 3a), microwave (Fig. 3b) and vacuum (Fig. 3c) dryers. The rising heating intensity (temperature in the convective and vacuum dryer and power in the microwave dryer) increases driving force of heat transfer which speeds up moisture transfer and shortens drying time. It is assumed that in all heating modes moisture travels through the fixed bed of particles as a liquid and evaporates at the surface because under all operating conditions (except vacuum drying at $\vartheta$ = 100 °C), the operating temperature is below the boiling point of water. The effect of temperature is more marked in convective drying (Fig. 3a) since the process is faster due to flow through the layer of particles (mass and heat transfer occur in the same direction). Generally, vacuum drying is slower than convective drying. At $\vartheta$ = 40 °C it is faster because of the lower drying temperature. Actually, the outlet of a vertical convective dryer is open and so the relative amount of thermal losses is larger at lower drying temperatures. On the other hand, the vacuum dryer is a closed system. At $\vartheta$ = 100 °C more favorable conditions are re-established in a vacuum dryer rather than in a conventional one, because of the drying temperature being higher than the water boiling temperature at a given absolute pressure (Fig. 3c). In that case, a part of the water evaporates already in the pores and the moisture moves partly as vapor. The inner pressure gradient facilitates the drying process. As already mentioned, absorption of the microwave energy is reduced with the reduced moisture content of a material. Therefore, the drying process is slowed down. In our work, this was most clearly demonstrated at two lowest heating intensities, when drying time was significantly longer (Fig. 3b).

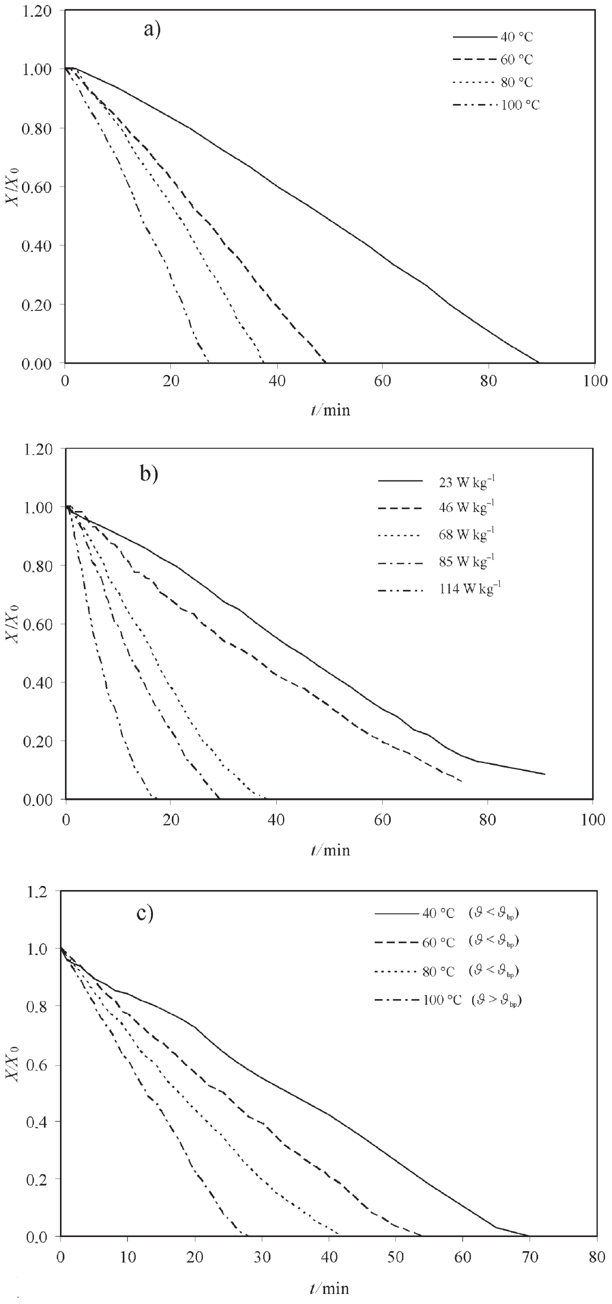



Fig. 3 – *The influence of the heating intensity ($x$ = 1180–2360 μm) a) Convective drying ($v$ = 1.55 m s$^{-1}$), b) Microwave drying, c) Vacuum drying ($p_{abs}$ = 0.9 bar)*

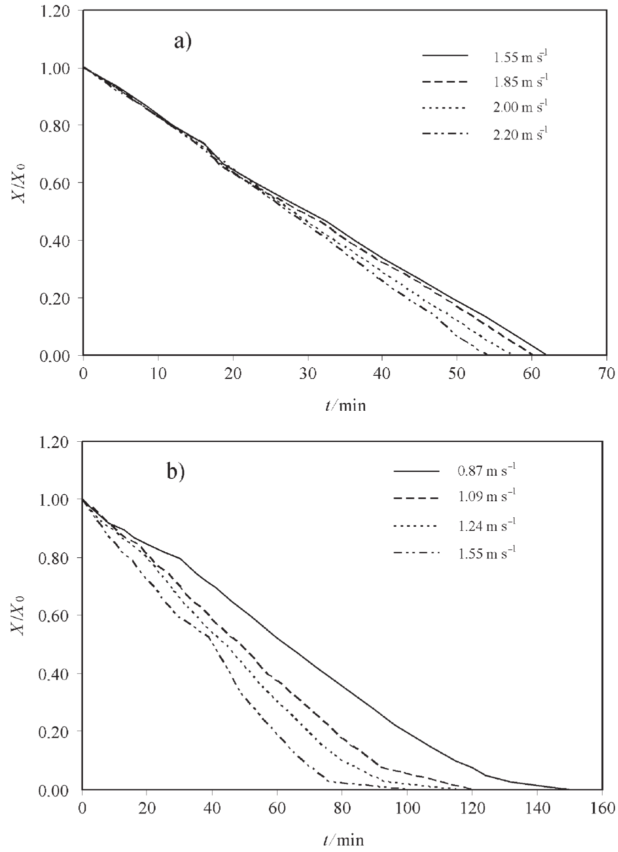
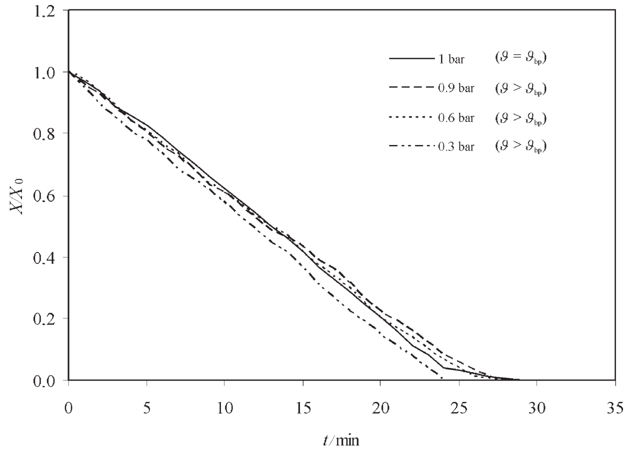



Fig. 4 – *The influence of the hot air superficial velocity a) Convective drying ($x$ = 600–850 μm; $\vartheta$ = 60 °C), b) Tunnel drying ($x$ = 1180–2360 μm; $\vartheta$ = 40 °C)*

Fig. 4 shows the effect of the superficial air velocity on the drying kinetics in the convective dryers (vertical and tunnel). Drying rate increased with the increased superficial air velocity. At higher superficial air velocity hydrodynamic conditions were improved, i.e. the resistances to mass and heat transfer were reduced. In the convective vertical dryer (Fig. 4a) the air flowed through the fixed bed of particles, thus creating more favorable drying conditions than those in the tunnel where the air flowed above the material surface and the transfer of mass and heat was reversed. Irrespective of a markedly bigger volume of the sample, however, drying time in the tunnel was not significantly longer ($t$ = 10 min) under equal operational conditions ($v$ = 1.55 m s$^{-1}$, $\vartheta$ = 40 °C, $x$ = 1180–2360 μm) (Fig. 4b, (tunnel dryer) and Fig. 9a (vertical convective dryer), also evident from Fig. 7). Volume of the sample was compensated with a larger evaporation surface. Besides, some heat was also transferred to the wet material over the carrier with good thermal conductivity and, actually, it was also heated in the direction of moisture flow through the porous structure.

The results showed that under the studied operational conditions the pressure changes during vacuum drying did not have significant effect on the drying kinetics because of dolomite being non-pressurizable, nonporous and nonhygroscopic (Fig. 5). However, reduction of the absolute pressure in the dryer slightly increased the drying rate. All dependencies were linear except under the atmospheric pressure, when there was a falling rate period. At the temperature of $\vartheta$ = 100 °C and the pressures below atmospheric, when the drying temperature was above boiling point, the flow of generated moisture was determined by the pressure gradient. The falling rate period under equal operating conditions was not recorded in the convective dryer

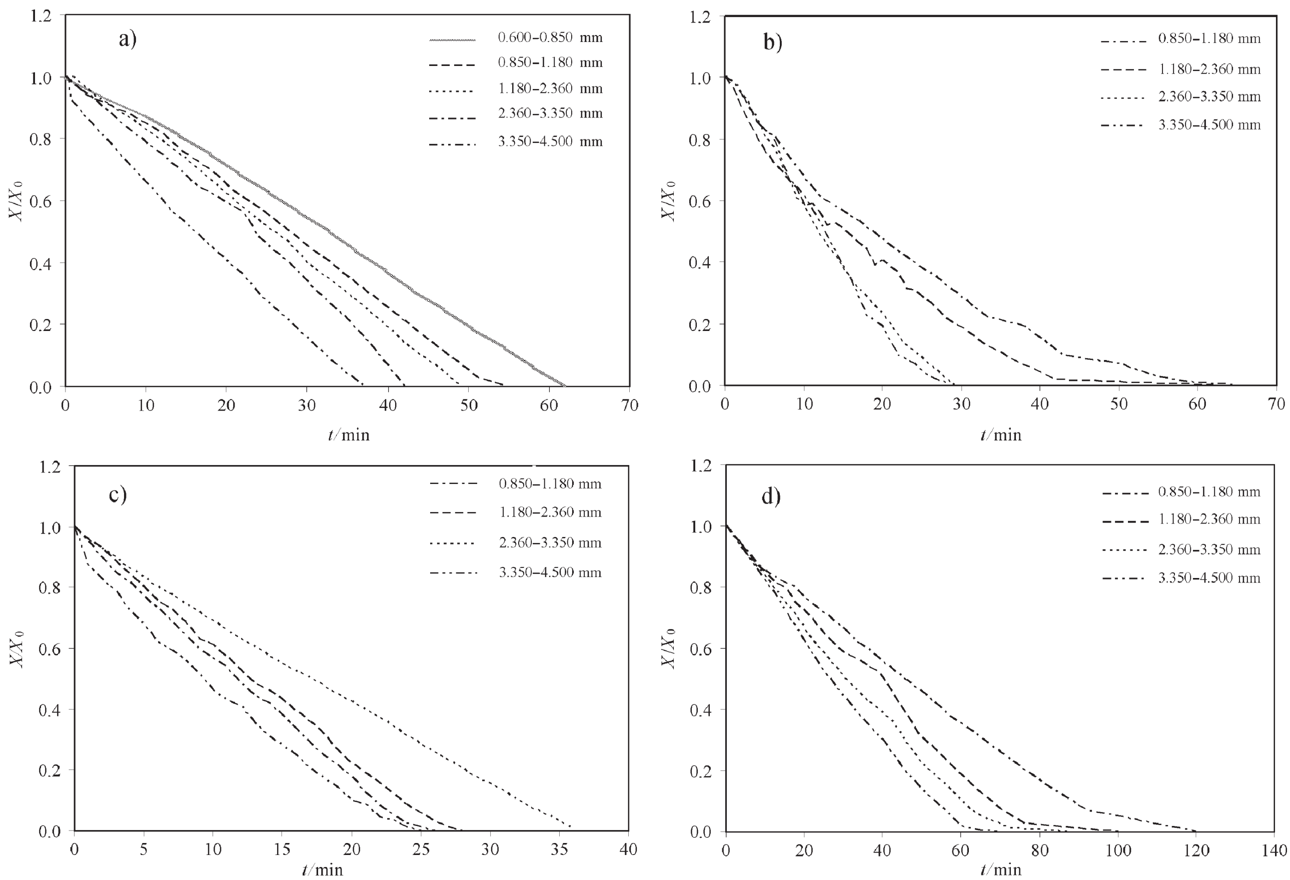



F i g . 5 – *Vacuum drying: the influence of the pressure in the drying chamber (x = 1180–2360 μm; ϑ = 100 °C)*

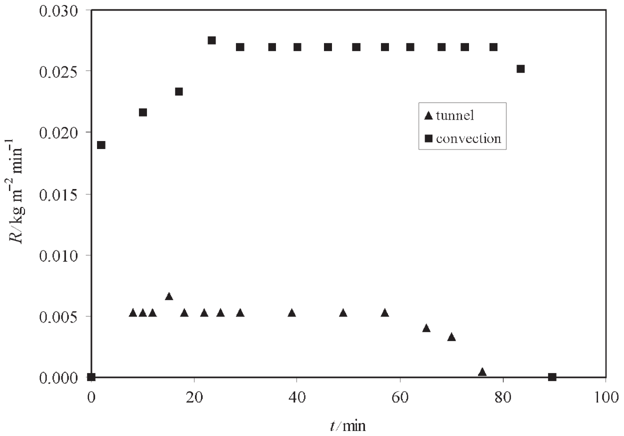
because of the way air came in contact with the fixed bed of particles.

The effect of particle size on the drying kinetics of dolomite is shown in Fig. 6. With the increase in particle size, the drying time in the convective (Fig. 6a), microwave (Fig. 6b) and tunnel (Fig. 6d) dryer was reduced. Because every dryer was fed with equal initial mass of a dry material, bigger particles were "packed" in one layer only, whereas the smallest ones were creating several layers. That resulted in the trap of some moisture between particle layers and consequential increase in the resistance to mass transfer on the side of the material. Fig. 6c shows the effect of particle size on the dolomite drying kinetics in the vacuum dryer. The effect was clearly opposite to that in other dryers. Actually, as drying in the conduction dryers was controlled by heat transfer rather than by mass transfer, as in convective drying, the surface of the material through which heat was brought played an important role. Smaller particles are known to have larger specific surface area which results in faster moisture transfer. Consequently, smaller particles dry fastest.

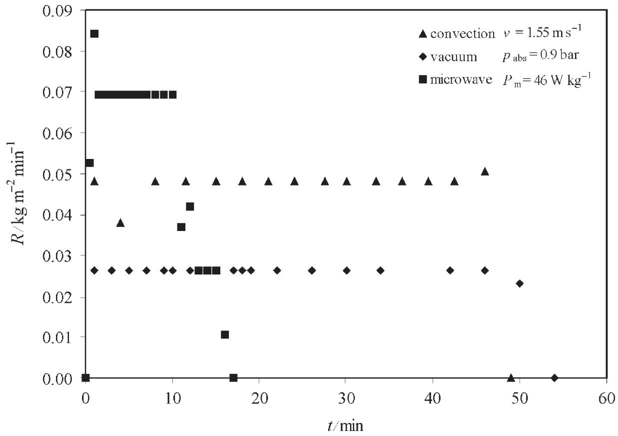
To present how the most favorable dryer for a fixed bed of dolomite particles was chosen, Fig. 7 and 8 are shown, comparing corresponding drying rate curves obtained under equal external conditions. Fig. 7 shows two convective dryers, and Fig. 8 shows various heating modes of the material (convective, vacuum and microwave). The obtained curves drive to the conclusion that the period of constant drying rate lasted almost until the very end of the process, and that the falling rate period was almost absent. The results show that the drying operation was controlled by external conditions through-




F i g . 6 – *The influence of the particle size: a) Convective drying ($v$ = 1.55 m s$^{-1}$; ϑ = 60 °C), b) Microwave drying ($P_m$ = 85 W kg$_{dm}^{-1}$), c) Vacuum drying ($p_{abs}$ = 0.9 bar; ϑ = 100 °C), d) Tunnel drying ($v$ = 1.55 m s$^{-1}$; ϑ = 40 °C)*



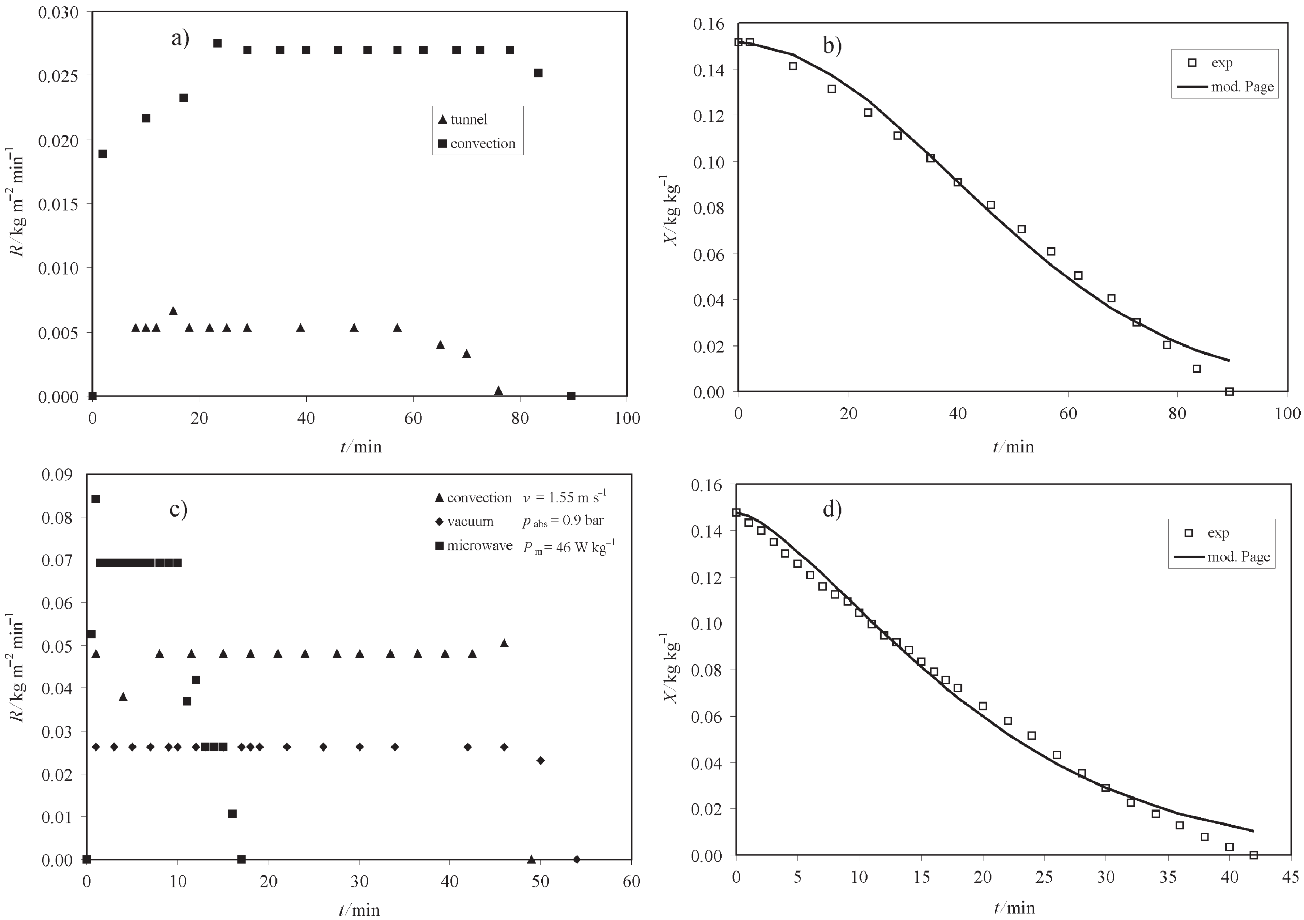

F i g . 7 – *Drying rate curves for convective drying ($v$ = 1.55 m s$^{-1}$, $\vartheta$ = 40 °C, $x$ = 1180–2360 μm) (vertical and horizontal (tunnel) driers)*




F i g . 8 – *The effect of the heating mode on the drying kinetics ($x$ = 1180–2360 μm, $\vartheta$ = 60 °C)*

out the whole experiment. The surface of the material retained a thin layer of moisture during entire drying, whereas the material's resistance was negligible. In the vertical convective dryer the achieved drying rate was far higher than in the tunnel due to the air stream through the fixed bed of particles. The vertical convective dryer was superior to the vacuum dryer for the same reason. Microwave drying was optimal for drying of the fixed bed of the nonhygroscopic dolomite particles.

Measurement data regarding dependence of the material's moisture content upon time were approximated to the modified Page's model (Fig. 9,




F i g . 9 – *The applicability of the modified Page model: a) Convective drying ($\vartheta$ = 40 °C, $v$ = 1.55 m s$^{-1}$, $x$ = 1180–2360 μm), b) Tunnel drying ($\vartheta$ = 40 °C, $v$ = 1.55 m s$^{-1}$, $x$ = 3350–4500 μm), c) Vacuum drying ($\vartheta$ = 80 °C, $p_{abs}$ = 0.9 bar, $x$ = 1180–2360 μm), d) Microwave drying ($P_m$ = 68 W kg$_{dm}^{-1}$, $x$ = 1180–2360 μm)*

Table 3 – *Average values of the correlation indexes and the standard deviations for the Page model*

| Dryer | $\rho$ | $\sigma \cdot 10^3$/kg kg$^{-1}$ |
|---|---|---|
| vertical convection | 0.979–0.995 | 4.40–6.60 |
| tunnel | 0.991–0.998 | 1.85–6.70 |
| microwave | 0.987–0.998 | 2.78–5.20 |
| vacuum | 0.986–0.998 | 4.31–6.80 |

Table 3). It could be concluded that the chosen model could be applied in describing the drying kinetics. However, physical relevance of parameter $t_k$ mentioned in previous studies showed[20,22] becomes insignificant in the experiments with only the period of constant drying rate. Hence, it could be concluded that the drying kinetics of nonhygroscopic materials could be described by the original Page's model (eq. (1)) rather than the modified one (eq. (3)). Deviations between the experimentally obtained and calculated data were due to the prolonged periods of constant drying rate during which the function $X(t)$ was linear. These deviations were smaller under mild drying conditions when the falling rate period was somewhat longer, and also when the process stabilization period was prolonged.

The effect of the process conditions on the parameters of Page's model, $k$ and $n$, in all the experiments are shown in Fig. 10. The parameter $k$ increased with the drying rate. The increased intensity of heating (Fig. 10a) i.e. temperature in convective and vacuum drying and of the microwave heating power during microwave drying, the increased superficial air velocity (Fig. 10c) and lower pressure (Fig. 10d) resulted in the increased parameter $k$. During vacuum, convective (vertical and tunnel) and microwave drying the parameter $k$ changes with the medium size of the particles, in a same way as the drying curves (Fig. 10b). Higher values corresponded to the higher drying rates. Parameter $n$ was affected by the mode in which heat was transferred to the wet material during drying.[16] Other stated process conditions (superficial air velocity, particle size and pressure) only slightly influences its value.

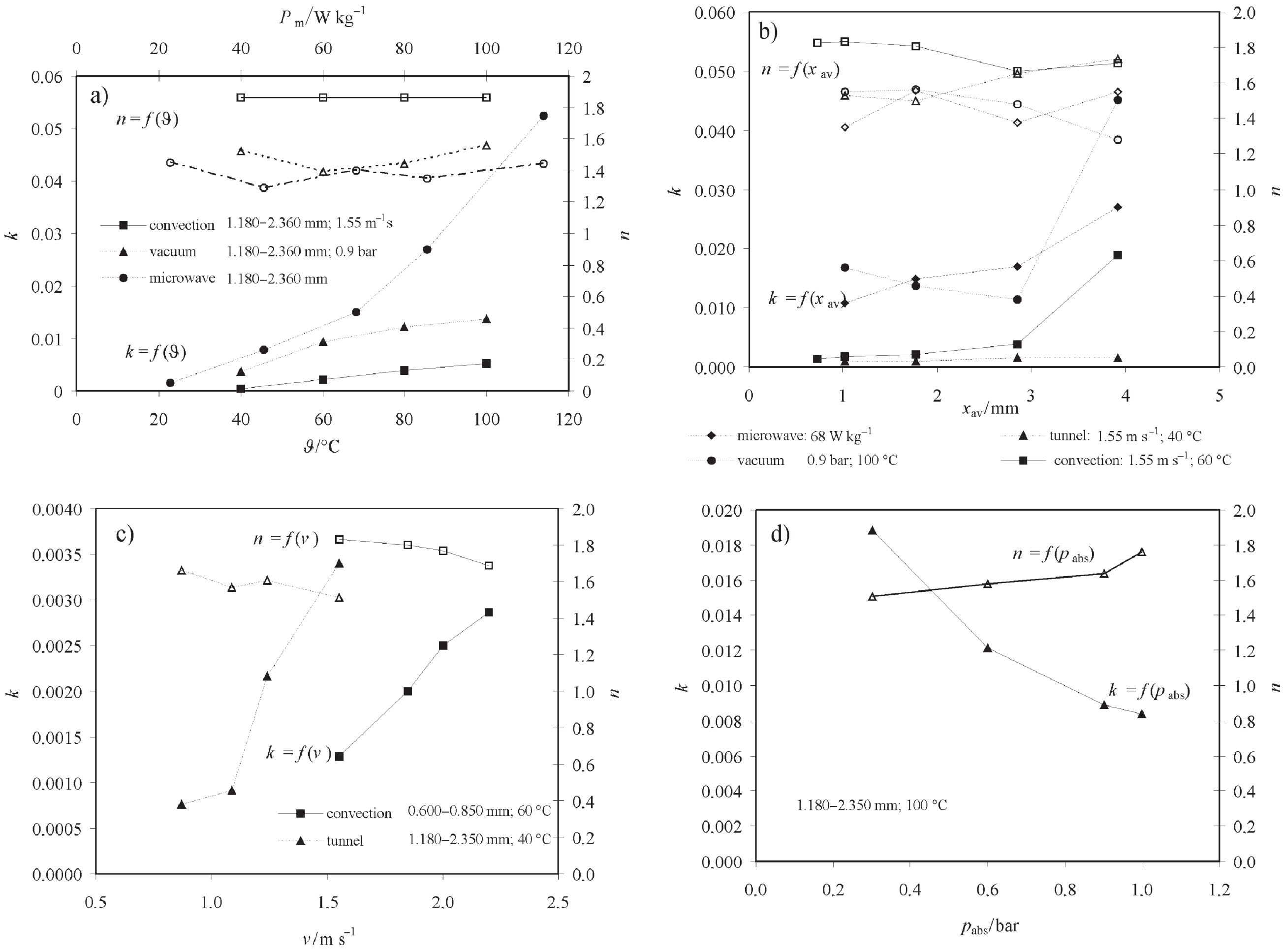



Fig. 10 – *The influence of the operational conditions on the Page model parameters k and n: a) the influence of heating intensity, b) the influence of the particle size, c) the influence of the superficial air velocity, d) the influence of pressure*

## Conclusions

This work was aimed at investigating the influence of granulometric properties of the material and of the operating conditions on the drying kinetics of dolomite.

Drying kinetics depends on specific surface area i.e. particle size of the material and on the process conditions.

The increased intensity of heating i.e. temperature in convective and vacuum drying and of the microwave heating power during microwave drying, and the increased medium size of the particles and superficial air velocity result in the increased drying rate i.e. shorter operation. During vacuum drying, the drying rate decreases with the increase in the medium size of the particles, which is due to the decreased surface of heat and substance exchange. Changes in the pressure do not have any significant effect on its values.

Page's model has shown to be successful in describing the drying kinetics throughout the entire drying operation, irrespective of the mode in which heat was transferred.

A microwave dryer is optimal for dolomite drying because of the operating duration.

### Notation

$k$ – Page model parameter, $s^{-n}$
$m$ – mass, kg
$n$ – mathematical model parameter
$p$ – pressure, bar
$P_m$ – specific microwave heating power, W $kg^{-1}$
$R$ – drying rate, kg $m^{-2}$ $s^{-1}$
$t$ – time, s
$t_k$ – modified model parameter, s
$\vartheta$ – temperature, °C
$v$ – drying air superficial velocity, m $s^{-1}$
$x$ – particle size, m
$X$ – material moisture content, kg $kg^{-1}$
$\bar{X}$ – arithmetic mean moisture content, $m_{H_2O}/m_{dm}$, kg $kg^{-1}$
$X/X_0$ – dimensionless moisture content

### Greek letters

$\varepsilon$ – dielectric constant, F $m^{-1}$
$\varepsilon$ – porosity
$\rho$ – correlation index
$\sigma$ – standard deviation kg $kg^{-1}$

### Subscripts

0 – initial
abs – absolute
av – average
bp – boiling point
dm – dry material
eq – equilibrium
exp – experimental

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