Acta Agrophysica, 2008, 11(1), 81-90

DRYING KINETICS AND ANTIOXIDANT ACTIVITY OF OREGANO*

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A b s t r a c t. Oregano was dehydrated by three methods: freeze drying, convective drying and vacuum-microwave drying (VM). Freeze drying was conducted at temperature of -60°C and under pressure of 65 Pa. The temperature of heating plate was 30°C. Convective drying was performed at three temperatures: 50, 60 and 70°C. Hot air velocity was 1 m s^{-1}. In VM dehydration the pressure in drying chamber was changing from 4 to 6 kPa and three microwave power levels were applied: 240, 360 and 480W. The decrease in moisture content of oregano dehydrated in convection was described by an exponential function, while in VM method by a linear function in the first drying period and by an exponential function in the second period. Moisture content at the critical point which divides drying curves into two periods was about 1.4 kg kg^{-1}dm regardless of the drying temperature used. The study revealed that the increase in drying temperature (convective dehydration) and in microwave power (VM) resulted in decreasing of drying time. Freeze drying was the least destructive method of drying because of the antioxidant activity. The most harmful method was convective drying at temperatures of 60 and 70°C. It was observed that higher phenolic content in dried oregano may be reached by reduction of temperature in convective method or by increasing the microwave power in V-M method.

K e y w o r d s : oregano, drying kinetics, antioxidant activity, polyphenols

INTRODUCTION

Oregano (*Origanum vulgare*) is an important herb rich in phenolic compounds with strong antioxidant (Capecka *et al*., 2005) and antibacterial (Sagdic and Ozcan, 2003) activity. The antioxidant activity depends on the amount of compounds that can

* This work was supported by the Polish State Committee for Scientific Research, under grant No. N312 031 32/2036 in the years 2007-2009.

delay or prevent the oxidation of lipids and other molecules. Zeng and Wang (2001) reported that the oregano herb had 3-20 times higher antioxidant activity than other herbs studied according to ORAC method, and higher total phenolic content. Additionally, oregano has 42 times more antioxidant activity than apples, 30 times more than potatoes, 12 times more than oranges and 4 times more than blueberries (AChSNS, 2002). It is well established that diet rich in fruits, vegetables and herbs can inhibit the development of major diseases such as cardiovascular disease and cancer (Hamauzu *et al*. 2006; Kanazawa *et al.* 1995). It is proved that antimicrobial activity of oregano extracts plays a role in inhibition of *Helicobacter pylori* (Chun *et al*. 2005) and enhances permeability of some drugs (Laitinen *et al.* 2004). Diet containing dried leafs and flowers of oregano also improves reproductive performance of animals (Allan and Bilkei, 2005). Herbs have been widely used to extend the shelf life of foods as well (Botsoglou *et al*. 2004). Oregano is a native herb to the countries of the Mediterranean region. Nevertheless, the plant is successfully cultivated in Poland in glass houses all over the year. However, in the food industry the dry form of oregano is commonly used. Dehydration of herbs can be performed using different methods. The most popular method is convective drying. However, increase in the air temperature usually results in a decrease of the quality of dried herbs (Diaz-Maroto *et al*. 2002). Vacuum-microwave (VM) drying of food is getting more and more popular thanks to its advantages. In this method microwaves penetrate the interior of the material causing water to boil at a relatively low temperature. This creates a high vapour pressure in the centre of the material, allowing rapid transport of moisture out of the product (Sham *et al*. 2001). Parsley samples obtained by VM drying were better compared to those air dried in terms of colour, essential oil contents, aroma and off-flavour (Boehm *et al*. 2002). The relative content of major flavour volatiles, rehydration rate, colour and structural integrity determined for VM dehydrated Mexican oregano (*Lippia berlandieri Schauer*) were quite similar to those obtained in freeze-drying and much better than those obtained in air drying (Yousif *et al.* 2000). No work has been done so far to dry oregano (*Origanum vulgare*) using the VM method.

Therefore, the study was aimed at drying kinetics determination of oregano dehydrated in convection and by VM. The aim was also to estimate the effect of drying method on antioxidant activity of dehydrated oregano.

MATERIALS AND METHODS

Drying of fresh oregano

Fresh oregano plants of moisture content 10.5 kg kg^{-1} dm were tested. They were grown under greenhouse conditions. Three methods of dehydration were used: convection drying (convective dryer designed and made in the Agricultural Engineering Institute of Wroclaw), vacuum microwave drying (VM-200 dryer, Plazmatronika Wroclaw), and freeze drying (freeze dryer OE-950, Hungary). Convective drying was conducted at three temperatures: 50, 60 and 70^{o}C, air velocity was 1 m s^{-1}. During the M-V drying three levels of MW power were applied: 240, 360, 480 W. The pressure in the drying chamber varied between 4 and 6 kPa. During freeze drying the pressure was reduced to 65 Pa. The temperature in the drying chamber was –60^{o}C, while the heating plate reached 30^{o}C. Convection and VM drying curves were determined according to samples weight loss measured during drying. The process of dehydration using all the methods mentioned above was conducted until moisture content in dried samples was 0.05 kg kg^{-1}dm.

Estimation of antioxidant properties and total polyphenol content

Ground dry plant materials (1 g) were weighed in a test tube. A total of 10 ml of 80% aqueous methanol was added, and the suspension was stirred slightly. Tubes were sonificated twice for 15 min and one was left at room temperature (~ 20^{o}C) for 24 h. The extract was centrifuged for 10 min (10 min, 1500 x g), and supernatants were analysed.

Total polyphenol content was measured according to the Folin-Ciocalteu colorimetric method described previously by Gao *et al.* (2000). Plant extracts (100 μl) were mixed with 0.2 ml of Folin-Ciocalteu reagent and 2 ml of H_2O, and incubated at room temperature for 3 min. Following the addition of 1 ml of 20% sodium carbonate and the mixture, total polyphenols were determined after 1 h incubation at room temperature. The absorbance of the resulting blue colour was measured at 765 nm with a Shimadzu UV-VIS spectrophotometer. Quantification was done with reference to the standard curve of gallic acid. The results were expressed as gallic acid equivalents (GAE), milligrams per 100g. All determinations were performed in triplicates (n = 3).

The DPPH radical scavenging activity was determined using the method proposed by Yen *et al.* (1995). One hundred μM DPPH was dissolved in ethanol (96%). The radical stock solution was prepared fresh daily. The DPPH solution (1 ml) was added to 1 ml of polyphenol extracts with 5 ml of ethanol. The mixture was shaken vigorously and allowed to stand at room temperature in darkness for 10 min. The decrease in absorbance of the resulting solution was monitored at 517 nm at 10 min. The results were corrected for dilution and expressed in μM Trolox per 100 g. All determinations were performed in triplicates (n = 3).

Radical scavenging activity (RSA) was expressed as the percentage of DPPH elimination after 1 and 10 min since the start of reaction, calculated according to the following formula:

$$DPPH = \frac{\text{absorbance of control sample - absorbance of herb extract}}{\text{absorbance of control sample}} \times 100\% \quad (1)$$

RESULTS AND DISCUSSION

Drying kinetics

Based on the measuring points obtained during convective drying it was determined that the loss of water content may be described by means of an exponential function (Fig. 1):

$$Mc = A \cdot e^{-\frac{t}{B}} + C \quad (2)$$

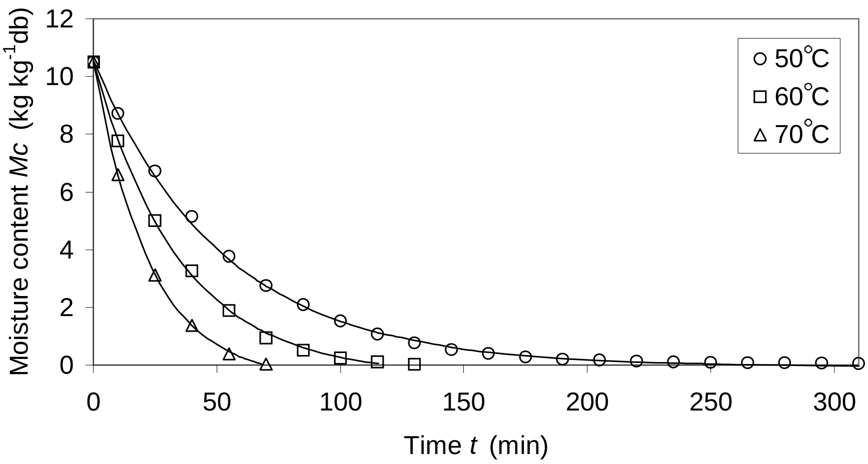

Fig. 1. Drying kinetics of oregano for convective method

During the VM method (Fig. 2) two periods divided by the critical point K were observed (Pabis and Jaros 2002). Water content at the critical point was about 1.4 kg kg^{-1} db regardless of the temperature used. The decrease in moisture content during the first period (till the moment of reaching the critical point K) was described by a linear function. During the second period water loss was described by an exponential function (Fig. 2).

$$Mc = D \cdot t + E \quad (3)$$

Table 1 presents the parameters A, B, C, D and E of the functions which describe the drying process of both methods.

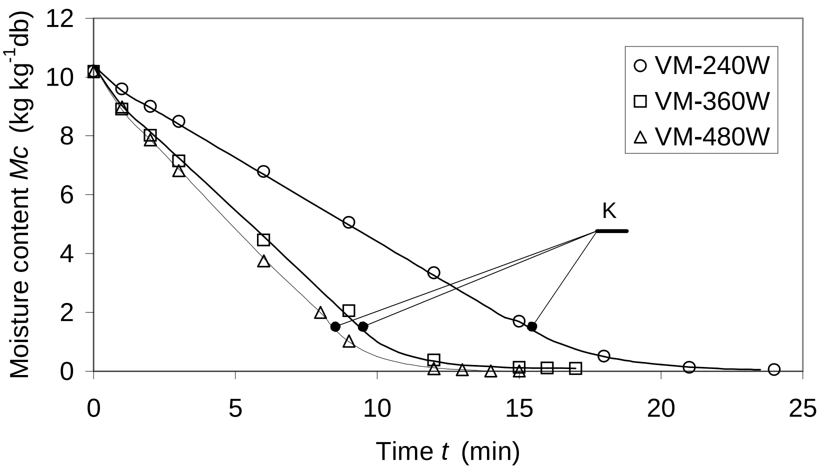

Fig. 2. Drying kinetics of oregano for vacuum-microwave (VM) method, K – critical point

Table 1. Parameters A, B, C, D and E of functions which describe drying process using convective and vacuum-microwave (VM) methods. Coefficient of determination R^2 is over 0.99 in every case

Drying process variables settings	Equation parameters				
	A	B	C	D	E
		Convective method			
Temperature (˚C)					
70	11.01	22.58	–0.50		
60	10.85	34.97	–0.35		
50	10.60	52.33	–0.05		
		Vacuum-microwave method (VM)			
Microwave power (W)					
480	447.96	1.48	–0.02	–1.008	9.98
360	606.28	1.57	0.086	–0.888	9.91
240	732.54	2.47	–0.003	–0.57	10.1

As it was to be expected, the VM method was faster than convection. During the analysis of convective drying it was observed that temperature increase from 60 to 70°C caused shortening of drying time from 310 to 70 minutes, while during the VM method microwave power increase from 240 to 480 W resulted in time shortening from 24 to 15 minutes.

Derivation of the functions describing the oregano drying process permit determination of the drying rate. The kinetics of convection and VM drying are shown in Figure 3.

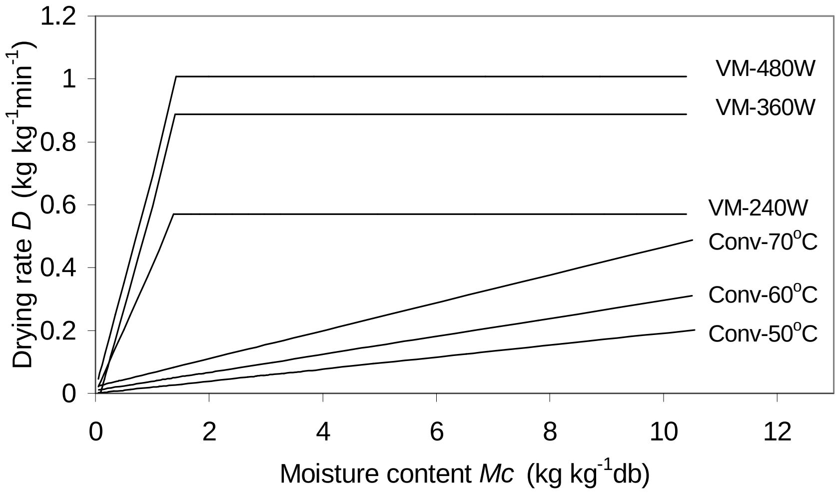

Fig. 3. Drying rate during dehydration of oregano using convective (Conv) and vacuum–microwave (VM) method

Antioxidant properties and total polyphenol content

The total phenolic content in methanol extracts was analysed by the Folin-Ciocalteu method. In Table 2 the total phenolic content of oregano is also reported, the content varying from 394.06 to 63.96 mg GAE 100 g^{-1}. In dried plant material significant losses of polyphenolic compounds were found in comparison to the fresh herbs.

After the drying process the phenolic content measured was over 50% lower with the relation to control (regardless of the method used). The lowest phenolic content was determined in samples that were dried using the convective method with the temperature applied 60 and 70^{o}C (63.96 and 64.68 mg GAE 100 g^{-1}, respectively), while the general phenolic content in samples dried at the temperature of 50^{o}C was 138.04 mg GAE 100 g^{-1}, even though the process was conducted for 310 minutes (Fig. 1). Phenolic compounds degrade when high temperature is applied (even when the process is short). This fact may influence the content of phenolic compounds in the dried material. Bigger amount of phenolic content was gained during the VM method. That let us observe that the bigger power of microwaves applied the bigger the phenolic content in general. Higher microwave power caused shortening of drying time (Fig. 2). It also resulted in shorter time of sample exposure to the destructive effects of temperature. Temperature should be dependent on reduced pressure used in the chamber during careful drying.

Table 2. Total phenolic content, antioxidant activity (DPPH) and radical scavenging activity (RSA) of fresh oregano and oregano dehydrated using three different processing methods

Drying method	Total phenolic (mg GAE 100 g^{-1})	DPPH (μM Trolox 100 g^{-1})	RSA (%)
Fresh sample	394.06	362.49	64.1
Freeze drying	168.87	286.00	50.6
VM – 240W	85.14	100.34	17.7
VM – 360W	100.48	129.82	23.0
VM – 480W	112.11	148.15	26.2
Convective – 50˚C	138.04	168.07	29.7
Convective – 60˚C	63.96	75.64	13.4
Convective – 70˚C	64.68	15.88	2.8

The highest phenolic content was determined during freeze drying. Those samples were characterized with phenolic content higher by 18.22% and 33.61% than those dried using convective drying at 50^{o}C or VM method at microwave power of 480W.

In general, measured phenolic content is connected with the activity of dried material. The percent inhibition of DPPH was from 64 to 6% and depended on the method of drying (Tab. 2). The ability of reduction in synthetically generated radical 2.2-diphenyl-1-picrylhydrazyl in fresh samples was 362.49 μM Trolox 100 g^{-1} and it was lower in samples dehydrated by other drying methods by 76.49 μM Trolox (freeze drying), 214.34 μM Trolox (VM at 480W) and by 194.42 μM Trolox 100 g^{-1} (convection).

High content of phenolics affected also the strong capability of the species examined to deactivate free radical DPPH, especially fresh oregano. Lagouri and Boskou (1996) and Kähkönen *et al.* (1999) stated that among plant phenolics responsible for antioxidant activity a prominent role was played by phenolic acids and flavonoids. Chun *et al.* (2005) found five major phenolic metabolites in oregano: rosmarinic, caffeic, coumaric and protocatechuic acid and quercetin. A very important compound in herbs of *Lamiaceae* family is rosmarinic acid, showing high scavenging DPPH potential (Chen and Ho, 1997), this being related to the presence of four hydroxyl groups in its molecule (Fecka *et al.*, 2002). The rosmarinic acid content in oregano extracts is very high (Chen and Ho, 1997).

CONCLUSIONS

1. The loss of moisture content in oregano samples dried in convection may be described by an exponential function, while in those dried using the VM method by a linear function during the first period and by an exponential function during the second period.
2. Increase in temperature from 50 to 70°C in the convective drying causes shortening of the time from 310 to 70 minutes. Increasing the microwave power in the VM method from 240 to 480 W results in shortening of drying time from 24 to 15 minutes.
3. Reduction of antioxidant activity during dehydration is the smallest while using the freeze drying method. It is also higher when the microwave power increases and the biggest during convective drying at the temperature of 60 and 70°C.
4. Lowering the temperature of drying in convective method and increasing the microwave power in VM leads to gaining higher phenolic content.

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KINETYKA SUSZENIA ORAZ WŁAŚCIWOŚCI PRZECIWUTLENIAJĄCE OREGANO

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Streszczenie. Oregano wysuszono przy użyciu trzech metod: sublimacyjnej, konwekcyjnej i mikrofalowo-próżniowej (VM). Suszenie sublimacyjne przebiegało w temperaturze –60°C, przy ciśnieniu 65 Pa i temperaturze płyty grzejnej 30°C. Suszenie konwekcyjne przeprowadzono w trzech tempe-raturach: 50, 60 i 70°C. Prędkość powietrza suszącego wynosiła 1 $m \cdot s^{-1}$. W suszeniu metodą VM ciśnienie w komorze suszenia wahało się w przedziale od 4 to 6 kPa, a moc mikrofal wynosiła: 240, 360 i 480 W. Zmniejszenie zawartości wody w oregano suszonym metodą konwekcyjną opisano przy użyciu funkcji wykładniczej, natomiast w odwadnianym metodą VM przy użyciu funkcji liniowej w pierwszym okresie suszenia i funkcji wykładniczej w okresie drugim. Zawartość wody w punkcie krytycznym, który dzieli krzywe suszenia na dwa okresy wynosiła blisko 1,4 $kg \cdot kg^{-1}$ sm niezależnie od zastosowanej temperatury suszenia. Badania wykazały, że zwiększenie temperatury w suszeniu konwekcyjnym i mocy mikrofal w suszeniu VM powodowało skrócenie czasu suszenia. Najmniej destruktywną metodą suszenia oregano ze względu na właściwości przeciwutleniające okazała się liofilizacja a najbardziej szkodliwą była konwekcja w temperaturze 60 i 70^{o}C. Stwierdzono, że większą zawartość polifenoli w wysuszonym oregano można osiągnąć redukując temperaturę suszenia w metodzie konwekcyjnej i zwiększając moc mikrofal w metodzie VM.

Słowa kluczowe: oregano, kinetyka suszenia, aktywność przeciwutleniająca, polifenole