JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

Effect of Drying Temperature on Alkylamide and Cichoric Acid Concentrations of *Echinacea purpurea*

DOUGLAS L. STUART* AND RON B. H. WILLS

School of Applied Sciences, University of Newcastle, PO Box 127, Ourimbah, NSW 2258, Australia

Root and aerial sections (flower, stem, and leaf) of *Echinacea purpurea* were dried with hot air at temperatures in the range of 40−70 °C, and the concentrations of alkylamides and cichoric acid were determined after drying. Increasing drying temperature decreased from 48 h at 40 °C to 9 h at 70 °C but resulted in a decreased concentration of cichoric acid in all plant sections with a greater loss from aerial plant parts than from the root. There was, however, no significant difference in the concentration of the alkylamides at any drying temperature. Establishment of operational parameters for the drying of echinacea must therefore be structured around the more labile cichoric acid.

KEYWORDS: ***Echinacea purpurea*; drying; alkylamides; cichoric acid**

INTRODUCTION

Echinacea purpurea (L.) Moench is a native North American perennial medicinal herb that has gained popularity internationally in recent years through claims that it beneficially stimulates the human immune system (*2*). It has been established that the chemical composition of *Echinacea* spp. is complex, but the alkylamides and caffeoyl-phenols are commonly used as markers to determine the medicinal quality of plant material and herbal extracts (*1*). There is an increasing pressure from the community for medicinal herb preparations to be standardized and thereby provide products that contain stated levels of active constituents (*6*). Surveys of retail products have shown that the levels of alkylamides and cichoric acid present vary greatly with many products containing very low levels (*8*, *10*). A survey of dried *E. purpurea* grown and marketed in Australia also revealed a large range in the levels of both groups of constituents (*10*), indicating that the material supplied to manufacturers is at least part of the problem. It is not known whether this variation is due to inconsistent quality of harvested raw material or from degradation during drying. Drying with hot air that is fan-forced across plant material is the common commercial method of drying. The effect of air-drying temperature has been assessed on the cichoric acid level in flowers by Kim et al. (*3*) and in the root material by Li and Wardle (*4*) with both groups reporting cichoric acid susceptible to drying at higher temperatures. The temperature range examined was 40−70 °C by Kim et al. (*3*) and 35−45 °C by Li and Wardle (*4*). There is no published information on the effect of drying temperature on the alkylamides. This study investigated the effects of hot air-drying in the temperature range of 40−70 °C on alkylamide and cichoric acid concentrations in root and aerial plant sections of *E. purpurea*.

* Corresponding author. E-mail: ftdls@cc.newcastle.edu.au.

MATERIALS AND METHODS

Mature *E. purpurea* plants for the first experiment were harvested after one season of growth from a commercial farm on the Central Coast of New South Wales, Australia in March 1998, and the below ground sections were immediately cleaned with running water. For the second experiment, plant material was obtained from the Northern Tablelands of New South Wales in April 2001. The roots were separated from the aerial plant sections, and both were placed in a hot air-dryer (G. T. D. Sydney, Australia) with hot air at 40, 50, 60, and 70 °C forced across the plants. At each temperature, there were six pairs of two plants. The change in weight of each sample was recorded at regular intervals to determine the rate of moisture loss, and drying was terminated when the sample weight between recordings was constant. The dried plant was then sectioned in root, leaf, stem, and flower, and the sample from each plant section was crushed (<200 μm) and analyzed for alkylamides and cichoric acid content as described by Stuart and Wills (*11*). All values were determined on a dry weight basis with the water content of the ground powder determined by drying in a vacuum oven for 12 h at 100 °C. A second drying study on drying temperature was conducted using air at 40, 55, and 70 °C with roots and flowers from plants that were divided after harvest into six sets of three plants per temperature. The flowers from each set of three plants were mixed together before being randomly separated into three subsamples, while the roots were cut with secateurs into three sections with one randomly chosen section from each plant placed into each subsample. One subsample was removed when dry, and the remaining two were left in the drier for a further 20 and 40% time increase to simulate overdrying.

RESULTS AND DISCUSSION

The drying curves of the plants held at each temperature in the initial drying experiment are given in **Figure 1**. The time to reach constant weight was taken as the drying time and decreased with increase in drying temperature. The drying time over the temperature range showed a 6-fold difference, being 48 h in air at 40 °C and 9 h in air at 70 °C. There was no difference in the appearance of plant material dried at the various temperatures. The average total loss in weight, which equates

10.1021/jf026213k CCC: $25.00
Published on Web 01/31/2003

Table 1. Concentration of Alkylamides and Cichoric Acid in *E. purpurea* Dried at Different Temperatures

temperature (°C)	Concentration (mg/g) root	flower	stem	leaf
	Alkylamides			
70	8.8	2.7	0.31	0.07
60	7.3	2.0	0.35	0.10
50	7.2	1.7	0.39	0.06
40	6.8	1.5	0.40	0.07
$LSD_{(P=0.05)}$	±2.26	±1.09	±0.26	±0.06
	Cichoric acid			
70	17.2	11.4	1.4	16.8
60	19.2	17.3	2.3	24.6
50	24.6	22.2	4.0	33.4
40	22.1	26.7	7.6	32.5
$LSD_{(P=0.05)}$	±4.2	±5.0	±2.8	±5.4

to water loss, of roots at constant weight was 74 g/100 g fresh material, while for the aerial sections the average loss in weight was 78 g/100 g. Subsequent analysis for moisture on the dried material showed that the moisture content remaining in all samples ranged between 8 and 12 g/100 g of the dried material. Thus, the total moisture content was about 80 g/100 g fresh weight for aerial sections and 76 g/100 g for root material.

The data in **Table 1** gives the concentration of alkylamides and cichoric acid in dried root and aerial sections dried at 40, 50, 60, and 70 °C. There was no significant difference in the concentration of alkylamides in root, stem, and leaf material dried at the various drying temperatures. There was, however, a significantly higher ($P < 0.05$) level of alkylamides in flower dried at 70 °C as compared to that dried at 40 °C. The concentration of cichoric acid was found to significantly decline in root, flower, stem, and leaf material as the drying temperature increased. There was a differential proportional decrease in cichoric acid level between plant sections with the level in root dried at 70 °C being about 80% of that present in root dried at 40 °C, whereas the corresponding proportions in flower, stem, and leaf were about 40, 20, and 50%, respectively.

To confirm the validity of these findings, a second study on drying root and flower was conducted. The results presented in **Table 2** show different levels of alkylamides and cichoric acid to the previous study because of the plant material originating from a different location and year of growth. There was no significant difference in the concentration of alkylamides in either plant section dried at 40, 55, or 70 °C, but the concentration of cichoric acid in root and flower decreased as the drying temperature increased. As in the previous study, there was a similar greater loss of cichoric acid from flower (about 45%) than from root (about 75%). Analysis of samples that were

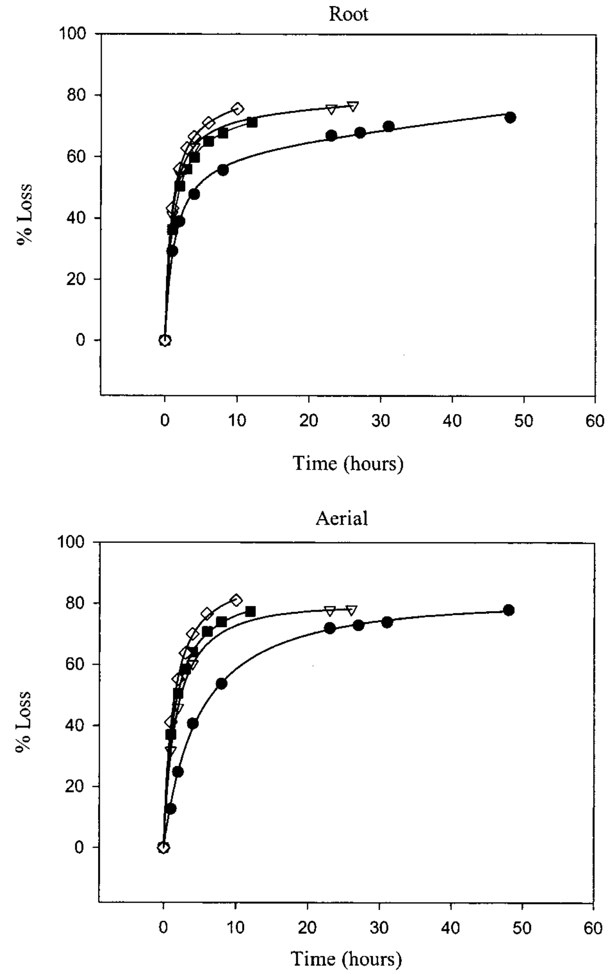

Figure 1. Drying curves for root and aerial plant sections of *E. purpurea* in a hot air-dryer at temperatures from 40 to 70 °C. (40 °C ●; 50 °C ▼; 60 °C ■; and 70 °C ◇).

left in the drier for an additional 20 and 40% of time after drying showed no decrease in alkylamides or any further decrease in cichoric acid. The values for alkylamides are the sum of 15 alkylamides within the root and three alkylamides within the aerial samples (*9*), and there was no change in the relative proportions of individual alkylamides in root and aerial material at different drying temperatures.

The findings in this study are the first reported data on the effect of drying temperatures on the alkylamides in echinacea and indicate considerable stability in the temperature range of 40−70 °C. However, the alkylamides are not fully stable as they have been shown to decline when dried material was held

Table 2. Concentration of Alkylamides and Cichoric Acid in *E. purpurea* Dried at Different Temperatures and Times

temperature (°C)	Concentration (mg/g) Alkylamides 40	55	70	$LSD_{(P=0.05)}$	Cichoric acid 40	55	70	$LSD_{(P=0.05)}$
				Root				
dry	4.7	4.8	4.4	±1.39	41.7	39.8	30.6	±6.8
dry + 20% OD	4.7	4.3	4.2	±1.96	42.3	36.7	34.2	±5.7
dry + 40% OD	4.6	4.3	4.3	±1.48	39.8	37.2	32.2	±4.8
$LSD_{(P=0.05)}$	±1.38	±2.14	±1.22		±6.0	±5.9	±5.5	
				Flower				
dry	2.1	1.7	1.7	±0.64	24.7	19.5	11.1	±4.5
dry + 20% OD	1.7	2.1	1.6	±0.54	28.3	20.0	11.5	±2.8
dry + 40% OD	2.0	1.8	1.7	±0.54	26.6	20.5	14.4	±4.5
$LSD_{(P=0.05)}$	±0.77	±0.42	±0.33		±4.3	±4.1	±3.6	

for a prolonged period at temperatures from 20 to 40 °C (*5*, *7*, *9*). The data showing the loss of cichoric acid confirm the results obtained by Kim et al. (*3*) for echinacea flower dried in over the same temperature range and by Li and Wardle (*4*) for root dried at 35−45 °C but extends these findings to echinacea leaf and stem. The enhanced loss of cichoric acid at higher temperatures indicates cichoric acid could be volatile in these conditions or that it is undergoing oxidative degradation. Since there was a much lower proportional loss of cichoric acid from root material than from aerial plant parts, it is suggested that cichoric acid within root material is more effectively compartmented and thus protected from these processes. Further investigations would be required to confirm this hypothesis.

A faster drying time for echinacea is more desirable from an industry efficiency perspective, and this can certainly be achieved by increasing the air temperature from 40 to 70 °C. However, since any increase in temperature will result in enhanced loss of cichoric acid, such an operational change can only be utilized if the alkylamides are the primary quality factor required in the end product. In most circumstances, the greater instability of cichoric acid dictates that the establishment of drying conditions for echinacea must be structured to ensure retention of cichoric acid.

ACKNOWLEDGMENT

We thank Rural Industries Research and Development Corporation and MediHerb Pty Ltd for the research grants and assistance for this study and Prof. Cliff Ellyett for advice and plant materials.

LITERATURE CITED

(1) Bauer, R.; Wagner, H. Echinacea Species as Potential Immunostimulatory Drugs. *Econ. Med. Plant Res.* **1991**, *5*, 253−321.

(2) Hobbs, C. *The Echinacea Handbook*; Eclectic Medical Publications: Portland, Oregon, 1989.

(3) Kim, H.-O.; Durance, T. D.; Scaman, C. H.; Kitts, D. D. Retention of caffeic acid derivatives in dried *Echinacea purpurea*. *J. Agric. Food Chem.* **2000**, *48*, 4182−4186.

(4) Li, S. C.; Wardle, D. A. Effects of Root Drying Temperature and Moisture Content on the levels of Active Ingredients in *Echinacea* Roots. *J. Herbs, Spices, Med. Plants* **2001**, *8*, 15−22.

(5) Livesey, J.; Awang, D. V. C.; Arnason, J. T.; Letchamo, W.; Barrett, M.; Pennyroyal, G. Effect of temperature on stability of marker constituents in *Echinacea purpurea* root formulations. *Phytomedicine* **1999**, *6*, 347−349.

(6) Maddocks, I. Alternative Medicine. *Med. J. Aust.* **1985**, *142*, 547−551.

(7) Perry, N. B.; van Klink, J. W.; Burgess, E. J.; Parmenter, G. Alkamide levels in *Echinacea purpurea*: effects of processing, drying and storage. *Planta Medica* **2000**, *66*, 54−56.

(8) Schieffer, G. W. Validated HPLC method for caffeic-acid derivatives and alkylamides in *Echinacea* solid dosage forms using single extraction procedure. *J. Am. Nutraceutical Assoc.* **2000**, *3*, 67−81.

(9) Stuart, D. L.; Wills, R. B. H. Alkylamide and cichoric acid levels in *Echinacea purpurea* tissues during plant growth. *J. Herbs, Spices, Med. Plants* **2000**, *7*, 91−101.

(10) Wills, R. B. H.; Stuart, D. L. Alkylamide and cichoric acid levels in manufactured *Echinacea* products. *Chem. Aust.* **1999**, 17−19.

(11) Wills, R. B. H.; Stuart, D. L. Alkylamide and cichoric acid levels in *Echinacea purpurea* grown in Australia. *Food Chem.* **1999**, *67*, 385−388.

(12) Wills, R. B. H.; Stuart, D. L. Effect of handling and storage on alkylamides and cichoric acid in *Echinacea purpurea*. *J. Sci. Food Agric.* **2000**, *80*, 1402−1406.

Received for review November 12, 2002.

JF026213K