Effect of Heat Pretreatment on the Yield and Composition of Oil Extracted from Corn Fiber

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Previously, hexane extraction of corn fiber was reported to produce a unique and potentially valuable oil that contained high levels of several phytosterols (which have been noted for their cholesterol-lowering properties). Current studies revealed that heat treatment (over the range of 100−175 °C) of corn fiber in either a convection oven or a vacuum oven caused only a modest reduction in the levels of the phytosterol components. However, these same heat pretreatments caused a considerable increase (up to 10-fold) in the levels (increasing from 0.34 wt % to a maximum of 3.64 wt % γ-tocopherol in the oil) and yields (increasing from 5.4 mg of γ-tocopherol/100 g of corn fiber to a maximum of 52.1 mg of γ-tocopherol/100 g of corn fiber) of γ-tocopherol in corn fiber oil. The main differences between the convection oven and vacuum oven pretereatments were associated with the disappearance of free fatty acids and free phytosterols at the higher temperature pretreatments in the vacuum oven, probably due to the lower boiling points of these lipids. Microwave pretreatment was also effective but caused a much smaller increase in the levels of γ-tocopherol.

Keywords: *Corn; Zea mays; tocopherol; fiber; ferulate*

INTRODUCTION

We have previously reported on the extraction and composition of corn fiber oil (Moreau et al., 1996) and found that it contains high levels of several phytosterol components (phytosterol fatty acyl esters, ferulate-phytosterol esters, and free phytosterols). Corn fiber (consisting of hull and other cell wall material) is an abundant byproduct of the wet milling of corn, and currently all of the corn fiber produced in the United States is blended into corn gluten feed. Dietary phytosterols, such as those in corn fiber oil, have been shown to effectively lower serum cholesterol when included in the diet at levels of ∼1−3 g per day (Ling and Jones, 1995). In their recent patent, Lane et al. (1997) reported that a heat pretreatment of rice bran increased the levels of extracted tocotrienols, oryzanols, and other components. The current study was conducted to investigate the effect of heat pretreatment of corn fiber on the yield and composition of the resulting corn fiber oil. If a simple heat pretreatment step significantly increases the levels of one or more valuable components in corn fiber oil, then it may be profitable to include it during the industrial processing of corn fiber oil.

MATERIALS AND METHODS

Corn fiber was a generous gift from Dr. Ting Carlson, Cargill Inc., Dayton, OH. It was freeze-dried, ground to 20 mesh with a Wiley mill (Thomas Scientific Co., Philadelphia, PA), and stored at −20 °C, until use.

For heat pretreatments, 4 g samples of ground corn fiber were placed in 25 mm × 150 mm screw-top vials. Duplicate samples of each were placed horizontally, without caps, for 1 h, in either a NAPCO Model 58301 vacuum oven (National Appliance Co., Portland, OR), operated at a vacuum of 28−29 in. of Hg, or a Freas Model 825 convection oven (Precision Scientific, Winchester, VA). Microwave pretreatment was similarly performed for either 2.5 or 5.0 min in an Amana Model R321T Radarrange operated at 1500 W (Amana Refrigeration, Inc., Amana, IA). After heating, the samples were cooled to room temperature (30−60 min) and immediately extracted by adding 40 mL of hexane, capping the tubes with Teflon-lined caps, and shaking the tubes horizontally for 1 h in a Burrell Model 75 wrist-action shaker (Burrell Inc., Pittsburgh, PA). After shaking, the hexane extract was filtered through a Whatman GF/A glass fiber filter (Whatman Laboratory Products, Clifton, NJ), a small aliquot was removed for HPLC analysis, and the rest was dried under a stream of nitrogen at room temperature for measurement of mass. Quantitative high-performance liquid chromatography (HPLC) analyses of the lipid classes were conducted as previously described (Moreau et al., 1996). The HPLC method employed an evaporative light-scattering detector, a DIOL column, and a 60-min binary gradient of hexane/2-propanol/acetic acid. Each heat pretreatment was repeated twice, with duplicate samples, and the data presented are the means ± standard error.

RESULTS AND DISCUSSION

Heat pretreatments, with either a convection oven or a vacuum oven, resulted in some small changes in the yield of oil from corn fiber (Table 1). With both types of ovens, there was a small progressive increase (totaling ∼4%) in yield at 100 and 125 °C, followed by a small progressive decrease (totaling ∼5 and ∼9% for convection oven and vacuum oven pretreatments, respectively, as calculated compared to the control) in yield at 150 and 175 °C. Observable charring of the corn fiber occurred at 150 and 175 °C, and pretreatment at 200 °C resulted in the production of smoke, extensive charring, and very low yields (<1%) of corn fiber oil (data not shown).

Analyses of the corn fiber oil extracted after various convection oven pretreatments revealed some interesting trends (Figure 1). Among the free fatty acids and the phytosterol derivatives (phytosterol fatty-acyl esters, free phytosterols, and ferulate-phytosterol esters) there

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Table 1. Yield of Oil from Heat-Pretreated Corn Fiber

treatment (EC)	yield of oil, g of oil/100 g of fiber (% change)
control (no heat treatment)	1.58 ± 0.01
convection oven	
100 °C	1.61 ± 0.00 (101.9)
125 °C	1.64 ± 0.00 (103.8)
150 °C	1.53 ± 0.01 (96.8)
175 °C	1.43 ± 0.06 (90.5)
vacuum oven	
100 °C	1.61 ± 0.04 (101.9)
125	1.63 ± 0.03 (103.2)
150 °C	1.60 ± 0.01 (101.3)
175 °C	1.50 ± 0.01 (94.9)
microwave oven (1500 W)	
2.5 min	1.63 ± 0.03 (103.2)
5.0 min	1.67 ± 0.02 (105.7)

were progressive decreases (the maximum decreases at 175 °C for each of the four lipids were 9, 19, 4, and 17%, respectively) in the levels of each as the temperature increased. However, with γ-tocopherol, levels progressively increased from 0.34 wt % in the control to 3.64 wt % for the 175 °C pretreatment. In addition to increasing the levels (wt %) of γ-tocopherol in corn fiber oil, the 175 °C heat pretreatment also cause a nearly 10-fold increase in the yield of γ-tocopherol (increasing from 5.4 mg of γ-tocopherol/100 g of corn fiber to a maximum of 52.1 mg of γ-tocopherol/100 g of corn fiber). Heat pretreatment effects were also considered for other tocopherols and tocotrienols, but α-tocopherol was the only other one detected. Heat pretreatment did cause an increase in the levels of α-tocopherol, but even after heat pretreatment, its levels never exceeded 0.1%.

Vacuum oven pretreatment caused even more dramatic effects in the composition of corn fiber oil, with the complete loss of free fatty acids at 150 and 175 °C and loss of 90% of the free phytosterols at 175 °C (Figure 2). It is likely that the disappearance of free fatty acids and free phytosterols under these conditions of vacuum and higher temperature is due to evaporation, because their boiling points are lower than those of the other lipids in corn fiber oil. The levels of phytosterol-fatty acyl esters were slightly increased at the 125 and 150 °C pretreatments, but the levels at 175 °C were nearly identical to those of the control. The levels of ferulate-phytosterol esters decreased gradually with increasing temperature. The levels of γ-tocopherol increased from 0.34% in the control to 2.50 and 2.98% at 100 and 125 °C pretreatments and then decreased at the two higher temperatures.

In addition to heat pretreatments with a convection oven (Figure 1) and a vacuum oven (Figure 2), corn fiber was also heat pretreated with a microwave oven. As noted for the above two oven pretreatments, microwave pretreatment of corn fiber also caused a small increase in the yield of corn fiber oil (Table 1). Although microwave pretreatments of corn fiber for 2.5 and 5 min caused visible browning, comparable to that achieved in the above oven pretreatments, the levels of γ-tocopherol in the corn fiber oil were increased much less (9 and 60% increases, respectively) than with convection oven and vacuum oven pretreatments (for which the maximum levels of increase were ∼1000%). Microwave pretreatment had minimal effects on the levels of ferulate-phytosterol esters (a 1% increase and a 4% decrease for 2.5 and 5.0 min treatments, respectively) in corn fiber oil.

These experiments reveal that heat pretreatment caused (a) a modest reduction in the levels of the phytosterol components and (b) a profound increase in the levels of γ-tocopherol. The differences between the convection oven and vacuum oven pretreatments reveal significant disappearance of free fatty acids and free phytosterols at the higher temperature under vacuum, probably due to their lower boiling points. Lane et al. (1997) offered a possible explanation for the large heat-induced increase in the levels of tocopherols and tocotrienols in rice bran—they suggested that a significant amount of these tocols are bound to proteins or linked to phosphate or phospholipid, and heat breaks these bonds. It is possible that a similar phenomenon is occurring in corn fiber but that only one bound component, γ-tocopherol, is released by heat pretreatment.

A saponification step, to release bound tocopherols, is often recommended as part of the routine extraction of total tocopherols (Kramer et al., 1997). We found that

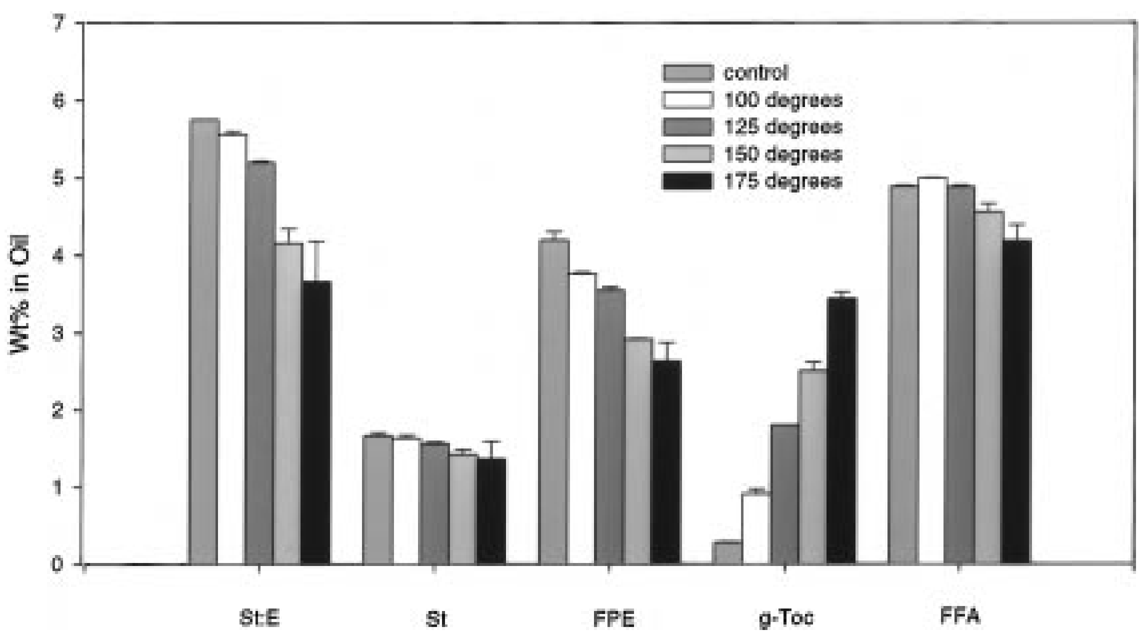

Figure 1. Effect of heat pretreatment of corn fiber in a convection oven on the composition of corn fiber oil: St:E, phytosterol-fatty acyl esters; St, free phytosterols; FPE, ferulate-phytosterol esters; g-Toc, γ-tocopherol; FFA, free fatty acids.

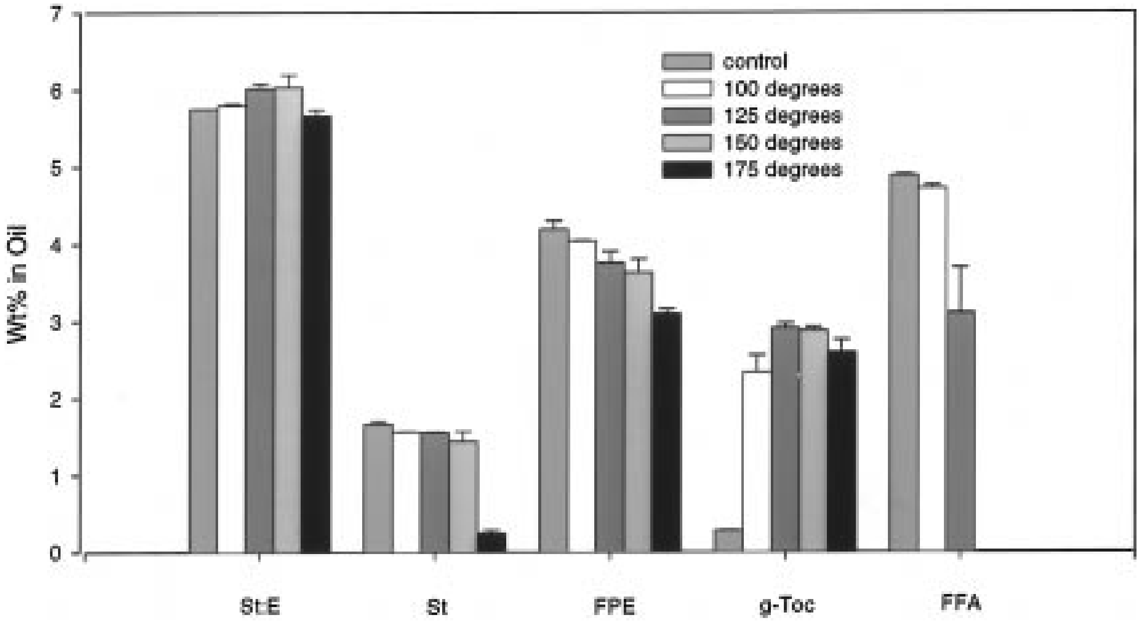

Figure 2. Effect of heat pretreatment of corn fiber in a vacuum oven on the composition of corn fiber oil. For abbreviations, see Figure 1.

saponification (alkaline hydrolysis) of corn fiber resulted in a 2−3-fold increase in the levels of extractable γ-tocopherol (data not shown). However, we are not able to explain why saponification released less bound γ-tocopherol than our heat pretreatments.

From an industrial perspective, these experiments have provided evidence that moderate levels of heat pretreatment of corn fiber could be used to dramatically enhance the levels of γ-tocopherol in corn fiber oil, without significantly decreasing its levels of valuable cholesterol-lowering phytosterol components. If this process was scaled to the pilot or industrial scale, further temperature optimization studies would need to be performed, but the trends revealed in this study provide a framework on which to start. These studies also reveal the general effects of convection versus vacuum oven pretreatments on the composition of the resulting corn fiber oil. Elimination of free fatty acid or the ability to use lower temperatures may justify the additional expense of vacuum oven pretreatment.

Two previous studies noted heat pretreatment effects for other seeds. Shin et al. (1997) noted a progressive decline in the levels of tocopherols, tocotrienols, and oryzanol in rice bran oil that was extruded at progressively higher temperature. Yoshida and Takagi (1997) reported that roasting (160−250 °C) of sesame seeds caused a temperature-dependent increase in the levels of sesamol and a decrease in the tocopherols sesamolin and sesamin.

The heat pretreatment effects observed in the current study differ in several ways from those reported by Lane et al. (1997). These authors noted that heat pretreatment of rice bran increased the levels of tocopherols, tocotrienols, and oryzanol (a ferulate-phytosterol ester). In contrast, heat pretreatment of corn fiber unexpectedly increased only the levels of one component, γ-tocopherol, and actually decreased the levels of ferulate-phytosterol esters. Lane et al. (1997) also noted that microwave pretreatment of rice bran was superior to other forms of heat pretreatment. However, in our experiments, microwave pretreatment of corn fiber caused only modest changes in the composition of corn fiber oil.

In conclusion, heat pretreatment appears to be a simple, inexpensive processing step that effectively increases the levels of γ-tocopherol in corn fiber oil from ∼0.3 to >3 wt %. Our previous studies have identified the unusually high levels of valuable phytosterols in corn fiber oil (Moreau et al., 1996). Increasing the levels of γ-tocopherol (a valuable natural antioxidant) to ∼3% should further increase the value of corn fiber oil. A logical and economical way to integrate a heat pretreatment step into an industrial process would be to modify the corn fiber drying step by optimizing the drying temperature/time parameters to achieve maximum levels of release of bound γ-tocopherol.

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