Effects of Heat Treatment and Dehydration on Properties of Cauliflower Fiber

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The effects of heat treatment and dehydration on fiber structure and hydration properties, using cauliflower floret/curd and stem tissues, have been investigated. No major changes in fiber composition resulted from sample treatments, but the degree of esterification of pectic polysaccharides, ~60% in fresh cauliflower, decreased by ~12% in samples heated at temperatures >40 °C. Enzymic activity was considered to be responsible, through pectin methyl esterase activity. De-esterification was temperature and moisture sensitive. Hydration properties were also affected by processing conditions. The solubility of nonstarch polysaccharides in fresh, freeze-dried, and 40 °C dried samples was ~6% but increased to 12% in boiled samples and decreased in samples dried at 75 °C. Similar behavior occurred for swelling and water retention capacity (WRC), with swelling and WRC highest for boiled samples and lowest for samples dried at 75 °C. Hence, a decrease in de-esterification was not directly responsible for changes in hydration properties. The results demonstrate the importance of processing history on functional properties and on the preparation of fiber-rich ingredients for successful incorporation into foods.

Keywords: *Fiber; cauliflower; esterification; dehydration; hydration*

INTRODUCTION

Dietary fiber is an important component of foods, with its chemical structure and physical properties considered important for functional and nutritional effects (Selvendran and Robertson, 1990). Ways to increase consumption of fiber can be through the supplementation of processed foods, but it is important to consider the type of fiber used as well as the amount present (Selvendran and Robertson, 1994). Most sources of dietary fiber, particularly for vegetables, are usually consumed after processing or cooking, and it should not be assumed that the fiber from the raw food is equivalent to that after cooking or processing. Processing, for example, drying/dehydration, can affect the structure and properties of foods, and, in particular, pectic polymers in the fiber matrix (Plat et al., 1988, 1991; Wu and Chang, 1990; Andersson et al., 1994). For example, blanching carrots leads to a de-esterification of pectic polysaccharides (Plat et al., 1991), and the change was considered to be enzyme-mediated rather than through an eliminative degradation (Albersheim et al., 1960).

During heat treatment, when moisture is not limiting, tissue softening occurs and is primarily due to a disruption of the constituent cell wall matrix. This involves the degradation and solubilization of pectic polysaccharides and may involve an eliminative degradation reaction (Van Buren, 1979). An increase in the apparent extractability of polysaccharides has been claimed to affect the dietary response to fiber (Shutler et al., 1987; Wolever, 1990), soluble fiber being promoted as having a positive effect. However, the insoluble uronic acid has also been suggested to correlate with dietary response (Wolever, 1990). It is therefore important to be aware of processing history when fiber concentrates for targeted use as food ingredients are developed. In particular, the ability of the fiber matrix to maintain its physical properties during cooking and processing may be more important than has hitherto been realized for the success of fiber incorporation into foods (Femenia et al., 1997). Similarly, the ability of pectic polysaccharides to resist degradation and solubilization may also be critical in the determination of a dietary response to fiber (Selvendran and Robertson, 1990; Wolever, 1990).

Work on the potential of using agrifood byproducts, such as cauliflower floret residues and stem sections, as fiber-rich ingredients in processed foods has involved investigation of the effects of processing on fiber properties. The objective of the current work was to determine how heat treatments and dehydration affected the fiber content in relation to the hydration properties of the cauliflower.

MATERIALS AND METHODS

Plant Material. Cauliflower (*Brassica oleracea* L. var. *botrytis*) plants were obtained locally, supplied as mature, market-ready complete plants. The root system and leaves were removed on receipt (~8 h postharvest) and discarded. The cauliflower head was removed from the main stem at the point where floret branching occurred, and the stem was dissected into upper and lower regions of approximately equal length. The lower stem was discarded, and the upper stem and the florets were retained as experimental material. Experimental material, stored overnight at 5 °C prior to processing or cooking, was considered equivalent to fresh cauliflower as purchased by the consumer and to material as supplied to a food processor.

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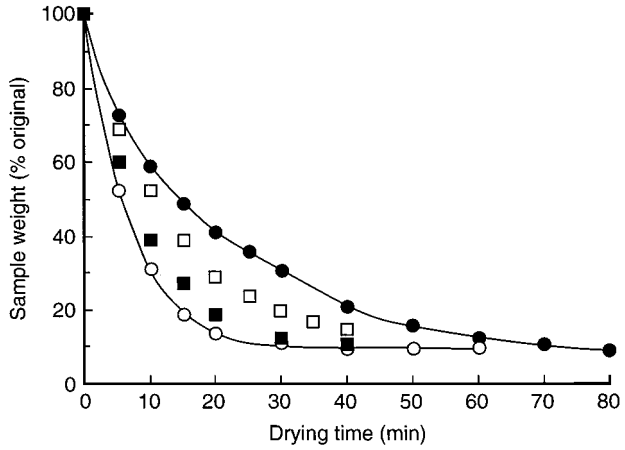

Figure 1. Effect of temperature on drying time: 40 °C (●); 50 °C (□); 60 °C (■); 75 °C (○).

Processing (Drying and Cooking). Fresh material was diced to ~25 mm^3 cubes, and samples (~100 g) were immediately frozen in liquid nitrogen and stored at −20 °C (fresh material × 5 batches), cooked (boiled material × 3 batches), or dried (dehydrated material × 5 batches for each temperature). Drying was undertaken using a laboratory scale fluidized bed drier (Catalytic Systems Division, Royston, U.K.) at temperatures controlled between 40 and 75 °C. The progress of drying at each temperature was monitored from sample weight loss at 5−10 min intervals (see Figure 1). Drying was continued until a constant weight was reached (fully dried) or until 50 or 80% of the moisture had been removed (partially dried). Partially dried samples were frozen in liquid nitrogen and stored at −20 °C until required for analysis. Fully dried samples were stored at −20 °C. Cooked samples were obtained from diced fresh material, and boiled for 15 min in distilled water (100 g/300 mL), with insoluble material recovered from the cooking liquor, frozen in liquid nitrogen, and stored at −20 °C until required for analysis.

Alcohol Insoluble Residues (AIRs). AIRs were prepared from stored samples by extraction in boiling ethanol [final concentration 85% (v/v) aq] and drying by solvent exchange (Waldron and Selvendran, 1990). Prior to further analysis, for each experimental treatment the individual AIR samples were pooled and milled to pass a 0.5 mm sieve using a laboratory grain mill.

Nonstarch Polysaccharide (NSP) Analysis. NSP was analyzed in duplicate for each experimental treatment. Sugars were released from AIR residues by acid hydrolysis. Samples (1−5 mg) were dispersed in 72% H_2SO_4 for 3 h followed by dilution to 1 M and hydrolyzed at 100 °C for 2.5 h. A 1 M H_2SO_4 hydrolysis (100 °C for 2.5 h) of water-insoluble residues was included to estimate noncellulosic glucose. Neutral sugars released were derivatized as their alditol acetates before analysis by GLC (Selvendran et al., 1979). Uronic acids were estimated colorimetrically, as total uronic acid (Blumenkrantz and Asboe-Hansen, 1973), using a 1 h 1 M H_2SO_4 sample hydrolysate.

Degree of methyl esterification (DE) of pectic polysaccharides was estimated in duplicate from methanol released after treatment of AIR with 1 M NaOH and its quantification by GLC using *n*-propanol as internal standard (Waldron and Selvendran, 1990).

Hydration Properties. *Extractability.* AIR samples (5 g), in duplicate, were suspended in distilled water (500 mL), with sodium azide (1 mg/mL) as bacteriostat, and stirred for 2 h at room temperature. The supernatants, recovered by centrifugation (12000 rpm, 20 min) and washings (two times) from the pellet, were pooled from each sample and freeze-dried (Gooneratne et al., 1994). Extracted NSP was estimated as the total sugars released by acid hydrolysis. The freeze-dried water-insoluble extracts were retained for the estimation of swelling and water retention capacity.

Swelling. Settled bed volume was used to measure swelling (Kuniak and Marchessault, 1972). Sample (~100 mg), in triplicate, was hydrated in excess (10 mL) distilled water, with sodium azide (1 mg/mL) as bacteriostat, for 18 h in a 10 mL graduated measuring cylinder. The settled volume of the sample was recorded as milliters of water per gram of residue.

Water Retention Capacity (WRC). Water retention was measured after centrifugation of the water-insoluble residue (Thibault et al., 1992). Sample (~ 5 g), in triplicate, was hydrated for 16 h in excess phosphate buffer (pH 6.3), with sodium azide as bacteriostat, in a weighed centrifuge tube prior to centrifugation at 2000*g* for 10 min. Excess supernatant was decanted and the pellet left to drain (15 min). The pellet fresh weight was determined prior to freeze-drying. Water retention was recorded as grams of water per gram of residue dry weight.

Changes in hydration properties, relative to the fresh material, were tested for statistical significance ($p > 0.05$) using the Student *t* test.

RESULTS AND DISCUSSION

Cauliflower samples were ~10% dry matter (Figure 1) (range 9.6−10.2%). The temperature used for drying had a marked effect on the rate of drying, >1 h being required for drying at 40 °C but only ~30 min for drying at 75 °C. Although not applied to the analysis of the drying behavior of the cauliflower samples, profiles of drying were similar to those expected from model systems developed to describe rates of water loss during drying (Rossello et al., 1992). From the relative times required to achieve drying at different temperatures, it was apparent that samples were exposed to variable periods of temperature treatment while at a relatively high moisture content, allowing the possibility of temperature-related modifications to NSP. Including a boiled sample treatment allowed some distinction to be made on the relative effects of enzymic activity and chemical modificaton on NSP and, from the freeze-dried sample, distinction between drying effects and temperature effects could be made. These effects are important when procedures to prepare fiber-rich ingredients from high-moisture content raw materials and to maintain or improve the properties of the fiber present are considered (Selvendran and Robertson, 1994).

The yield of AIR for fresh cauliflower samples was ~5% fresh weight (Table 1), representing ~50% of the cauliflower dry weight. AIR yield from dried florets was slightly lower, in particular the AIR of the sample dried at 75 °C (4.1%). Although this effect may be due to some loss of material, resulting from modifications to the sample during heat drying, it may also reflect changes in the affinity of polysaccharides for water during drying at increased temperature (40−75 °C). Novel associations between polysaccharides as heat drying proceeds may affect/remove water strongly bound by polysaccharides. Freeze-drying had no effect on AIR yield, but for boiled samples the AIR recovery was apparently higher. If account is taken of changes in dry weight, then the AIR yield of boiled sample was equivalent to ~5.6% of the fresh material. In this case the slightly higher yield might be ascribed to an increase in strongly bound water, associated with a partial degradation and increased porosity of the AIR from boiled material. All AIR preparations were shown to be effectively free of starch, by I_2/KI staining and from the low recovery of glucose following hydrolysis in 1 M acid.

Carbohydrate composition (Table 1) confirmed NSP was a major component of the AIR and that NSP was equivalent to ~2% of the cauliflower fresh weight in

Table 1. Effects of Drying on AIR Recovery and Polysaccharide Composition of Cauliflower Florets (Milligrams per Gram of Fresh Weight)[a]

sample	AIR (%)	Rha	Fuc	Ara	Xyl	Man	Gal	Glc	uronic acid	total mean	NSP SD
fresh	5.3	0.3	0.1	3.3	0.9	0.5	1.6	7.0	6.9	20.6	0.7
freeze-dried	5.1	0.3	0.2	3.2	0.9	0.5	1.5	7.1	6.9	20.6	0.9
dried at 40 °C	4.6	0.3	0.1	2.8	0.9	0.5	1.4	6.7	6.4	19.1	1.0
50 °C	4.4	0.3	0.1	2.9	0.9	0.5	1.5	6.6	6.3	19.1	1.0
60 °C	4.4	0.3	0.1	2.9	1.0	0.5	1.6	6.8	6.0	19.2	1.1
75 °C	4.1	0.3	0.1	3.1	1.0	0.5	1.7	6.9	6.2	19.8	0.9
boiled—as recovered	6.4	0.5	0.2	3.7	1.1	0.9	2.3	7.9	6.2	22.8	0.8
original fresh wt	5.6	0.4	0.2	3.3	1.0	0.8	2.0	7.0	5.5	20.2	0.8

[a] Values are the mean of determinations made in duplicate ± 1 SD where shown.

each sample treatment. On the basis of the experimental variation recorded (SD) a difference between samples of ~2.8 mg/g of fresh weight would be required for statistical significance ($p < 0.05$). Thus, on the basis of the original fresh weight there has been no significant change in fiber content due to experimental treatments. As noted for the AIR, the higher contribution of NSP from boiled material could be ascribed to changes in sample dry weight (fresh, ~10% dry weight; boiled, ~11% dry weight). There may also have been some loss of NSP into the cooking liquor, but from experiments involving cooked carrot (Ryden, 1995) loss of NSP into the cooking liquor was only ~4% of the total NSP and was not considered to make a major contribution to the interpretation of the experimental data.

The NSP content of the upper stem was ~3% sample fresh weight and the compositions were similar in the floret and upper stem samples (results for upper stem not shown). From the relative amounts of uronic acid, galactose, arabinose, and minor amounts of rhamnose, pectic polysaccharides were inferred to be the predominant type of cell wall polysaccharide present and accounted for ~50−60% of the total polysaccharides. From the relatively low amounts of glucose released by 1 M H_2SO_4 hydrolysis compared to Saeman hydrolysis, the bulk of the glucose in the AIR was inferred to be derived from cellulose, with some from xyloglucans. Carbohydrate analysis of the dried florets indicated that drying temperatures had no major effect on the composition or the NSP and that recovery of NSP was consistent with previous analysis of cauliflower (Englyst et al., 1988).

No major changes in the sugar composition of extracted material were noted due to drying or cooking, but the treatments did affect the DE of constituent pectic polysaccharides (Figure 2). The DE of the fresh cauliflower was 58% ± 1.5 for floret and for upper stem was 83% ± 1.9, but at drying temperatures >40 °C and after boiling, DE was reduced by ~12%. Freeze-drying had no effect on DE, and at drying temperature >50 °C there was no further decrease in DE, suggesting an enzymic rather than chemically mediated de-esterification had occurred. Pectin methyl esterase (PME) is known to remain active at increased temperatures (Cameron et al., 1994; Lopez et al., 1997), and activity can be affected by intracellular cations released during cell metabolism or following tissue disruption (Nari et al., 1991; Andersson et al., 1994; Alonso et al., 1997). PME is the most likely candidate to promote the de-esterification and has been implicated in the control of texture or tissue firmness in vegetables (Wu and Chang, 1990) and modification to carrot pectic polysaccharides during blanching treatments (Plat et al., 1991). It can be proposed that cooking-related tissue disruption begins to occur at temperatures >40 °C and hence that

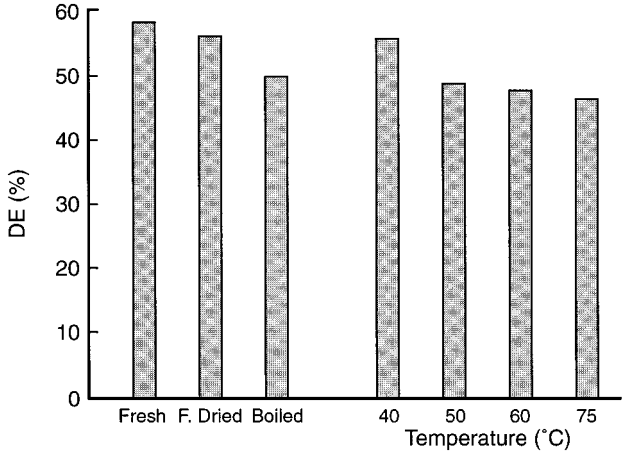

Figure 2. Degree of methyl esterification (DE %) of pectic polysaccharides from cauliflower floret. Results are the mean of determinations made in duplicate.

PME becomes activated. Thereafter, high PME activity can continue until either moisture becomes limiting or temperature becomes critical for enzyme denaturation, for example, boiling. These effects of processing have implications for the preparation of fiber concentrates that may be used as ingredients in processed foods. Because the decrease in DE was consistently ~12%, the mobility of PME in the cell wall and/or accessibility of methyl-esterified pectic polysaccharides may also be important in controlling the extent to which pectic polysaccharides can be modified by PME in the cell wall matrix.

The profile of de-esterification obtained from analysis of samples of stem and floret partially dried at 50 °C (Figure 3) was similar to the drying profile at 50 °C. This suggested a direct relationship between de-esterification activity and available moisture rather than just a relationship between DE and de-esterification activity because the upper stem, with a high DE (Femenia et al., 1998), was de-esterified in proportion to de-esterification in floret. When change in DE is expressed in terms of moisture availability (Figure 4), it becomes apparent that little change in DE occurred when moisture content was >60%, but between 60 and 20% moisture content there was a notable drop in DE for both stem and floret tissues. Undoubtedly, temperature gradients set up by heat-drying procedures could result in a gradient of cell disruption across tissue samples, that is, leading to water loss from surface tissues while core tissues remain intact. Hence, water loss from peripheral tissues, which have a lower initial DE (Femenia et al., 1998), could occur before the more highly esterified core tissues become de-esterified, that is, at apparently reduced moisture content. By incubating samples at a constant temperature between 50 and

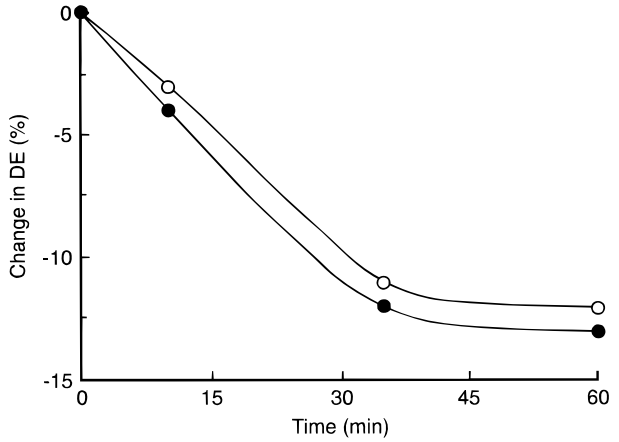

Figure 3. Time-related change in DE during drying at 50 °C. Change in DE is the loss in DE from time = 0, e.g., −10 = 48% DE for floret: floret (○); stem (●).

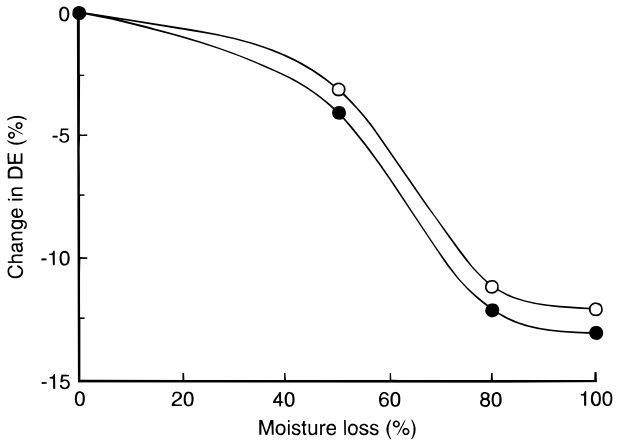

Figure 4. Moisture-related change in DE during drying at 50 °C. For change in DE, see the caption to Figure 3. Floret (○); stem (●).

75 °C, changes in DE could be monitored in the absence of a temperature gradient and when moisture was not limiting. Blanching broccoli within this temperature range (Wu and Chang, 1990) led to a firming of texture, considered due to PME activity and probably similar to the reason for DE changes in blanched carrot (Plat et al., 1991). However, as in the current study neither could resolve the extent to which changes in DE were limited by enzyme mobility or enzyme activity.

To determine whether hydration properties were affected by de-esterification or the method of drying and cooking, solubility, WRC, and swelling were measured (Table 2). The hydration properties can influence the success of incorporation of fiber-enriched ingredients into foods (Femenia et al., 1997). Solubility of NSP from floret and stem samples, as material extracted at 37 °C (Gooneratne et al., 1994), was similar, at ∼6% of the total NSP in fresh, freeze-dried, and dried at 40 °C samples. Solubility increased significantly ($p < 0.05$) to ∼12% of the NSP in boiled samples and decreased significantly ($p < 0.05$) to ∼4% in samples dried at 75 °C. Extracted NSP was mainly pectic polysaccharide in origin as indicated by the high proportions of uronic acid, arabinose, and galactose (data not shown). WRC and swelling were also similar for corresponding samples of floret and stem. Both WRC and swelling were significantly reduced ($p < 0.05$) in samples dried at 75 °C and significantly higher ($p < 0.05$) in boiled samples. WRC and swelling of samples dried at 40 °C were also

Table 2. Solubility and Hydration Properties of Cauliflower Floret AIRs[a]

	solubility (% total NSP)		WRC (g/g of insoluble AIR)		swelling (mL/g of AIR)	
sample	mean	SD ($n = 2$)	mean	SD ($n = 3$)	mean	SD ($n = 3$)
fresh	6.3	0.3	19.9	1.1	22.9	0.7
freeze-dried	6.2	0.3	18.7	1.0	19.4[c]	0.4
boiled	11.8[a]	0.7	24.6[b]	1.2	27.4[c]	0.8
dried at 40 °C	6.0	0.4	12.8[b]	0.3	16.9[c]	0.4
dried at 75 °C	3.8[a]	0.2	5.7[b]	0.5	4.2[c]	0.2

[a] Values are the mean ± 1 SD. NSP, nonstarch polysaccharide; WRC, water retention capacity. Values shown with a superscript are significantly different from the fresh material ($p < 0.05$) for each method used.

significantly lower than in the fresh material, and indeed WRC and swelling of samples dried at 75 °C were significantly lower than samples dried at 40 °C ($p < 0.05$). Hence, de-esterification was not directly responsible for changes in hydration properties, but DE may be involved by promoting the degradation and extractability of pectic polysaccharides and the generation of an increased pore volume during boiling. Conversely, a reduction in pore volume in dehydrated samples could result from close association between polysaccharides restricting the extent of rehydration. During boiling, β-elimiminative degradation of pectic polysaccharides is known to occur, catalyzed by several cations and anions (Albersheim et al., 1960; Keijbets and Pilnik, 1974; Sajjaanantakul et al., 1989), and will contribute to the extractability of pectic polysaccharides, as observed. Enzymic de-esterification may limit the potential for subsequent eliminative degradation and hence restrict the extractability of NSP from heat-dried samples. These results illustrate how processing history can affect the properties of fiber and hence influence the behavior and successful incorporation of fiber-rich ingredients into foods. In the preparation of fiber-rich ingredients it is therefore important to select processing conditions to control the type as well as the amount of fiber in foods.

CONCLUSION

Processing through heating or drying treatments to produce pectic polysaccharide-rich fiber supplements from vegetables can affect the structure and functional properties of the fiber, notably the DE of pectic polysaccharides and hydration properties. It can be proposed that heat treatment leads to an enzyme-mediated de-esterification and increased extractability of polysaccharides, whereas boiling may also involve increased extractability through an eliminative degradation of esterified pectic polysaccharides. The modification to structure and functional properties has implications for the successful exploitation of vegetable-based fiber products in foods.

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