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Spent residue from cumin – a potential source of dietary fiber

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Abstract

Cumin has total dietary fiber content (TDF) of 59.0%, insoluble dietary fiber (IDF) of 48.5%, and soluble dietary fiber (SDF) of 10.5%, while the spent residue from cumin (after oil and oleoresin extraction) was found to contain 62.1% TDF, 51.7% IDF and 10.4% SDF. The spent residue also contained 7.7% starch and 5% bound fat. Particle size analysis showed a direct effect on the hydration properties of the fiber. The spent residue exhibited 3.3 g/g water holding capacity, 4.0 g/g water retention capacity and 4.47 ml/g swelling capacity. Scanning electron microscopy revealed spherical starch granules embedded within cell wall material, which upon differential sedimentation gave differently sized starch granules (5.8 μm). Upon defatting the spent residue showed typically a 'honey comb' structure, almost devoid of starch granules. Thus, the spent residue from cumin, not having much commercial value at present, can be a rich source of useful dietary fiber and can find food applications. It can be an effective way of utilizing industrial waste from the point of view of environmental pollution from the residues of spice processing industries.

Keywords: Dietary fiber; Hydration properties; Soluble fiber; Insoluble fiber; Cumin; Cumin spent residue

1. Introduction

Dietary fiber consisting of non-digestible carbohydrates and lignin that are intrinsic and intact in plants, has received much attention due to its health benefits (Slavin, 2005). Soluble fiber lowers serum cholesterol and helps to reduce the risk of heart attack and colon cancer. It dissolves in the gut to form a viscous gel that lowers the absorption of released glucose. Consumption of dietary fiber reduces the risk of civilization diseases such as cardiovascular disease, colon cancer and obesity (Chau & Huang, 2004). Soluble and insoluble dietary fibers, are the storage and cell wall polysaccharides of plants that cannot be hydrolyzed by human digestive enzymes. They have been reported to decrease the absorption of carbohydrates and post prandial serum glucose level (Ou, Kwok, Li, & Fu, 2001). Cellulose, hemicellulose and lignin are the main components of insoluble fibre, which prevent or relieve constipation due to absorption of water from the digestive tract. Optimal intake of total dietary fiber reduces the risk of obesity, blood pressure, appendicitis and many other diseases (Zia-ur Rehinan, Rashid, & Shah, 2004). A fiber rich meal is metabolized more slowly and nutrient absorption occurs over a longer period (Jenkins et al., 1993). Further, a diet that provides adequate fiber is usually less energy dense and larger in volume and thus may bring a feeling of satiety sooner (Saris, 2003). Indeed the, National Advisory Committee in Great Britain has recommended a fiber intake of 25–30 g/day per person (Dashti, Al-Awadi, Khalafawi, Sawaya, & Al Amiri, 2003).

The total dietary fiber content of infant foods plays a central role in meeting the recommendations [19 g/d] as well as stabilizing the intestinal population by stimulating the proliferation of bacteria capable of digesting dietary fiber and lowering the colonic pH (Brooks, Mongeau, Deeks, Lampi, & Brassard, 2006). Spices are reported to contain 15–55% crude fiber and, except for a few, very little

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information is available on dietary fiber of spices. Cereal brans are used as a source of dietary fiber, but alternative sources of dietary fiber are also needed and data on nutritional input is also required.

Cumin (*Cuminum cyminum*) is commercially an important seed spice valued for its aroma, medicinal and therapeutic properties. Oil and oleoresin from cumin are value added products, which have export value. Spent residue from cumin, obtained after volatile oil extraction by steam distillation and resin extraction by solvents, is not commercially exploited for food application at present except in veterinary feeds to a small percentage. Currently around 400 tones of cumin spent residue are disposed of as waste every year. The amount of spent residue is calculated from the export figure of cumin oleoresin, and the yield of oleoresin from cumin (Mathew, 2004). It is highly desirable to find and use byproducts or wastes from the point of view of environmental protection by the effective utilization of industrial wastes. The aim of this study is to explore, the spent residue from cumin as a new source of dietary fiber, for its quality, physiochemical characteristics and application potential. No information is reported in the literature on the nature of dietary fiber and its characterization and also on the content and nature of starch in cumin. A systematic study has been carried out on the effect of residual fat and particle size on the hydration properties of the cumin fiber.

2. Materials and methods

2.1. Spent residue from cumin

Cumin seeds procured from local market were ground to 30-mesh size powder and the volatile oil was extracted by steam distillation. The resulting deoiled cumin powder (100 g) was taken in a glass column and extracted with acetone (200 ml) for 1 h contact time. The extraction was repeated five times and the pooled extract was subjected to desolventisation in a flash evaporator under vacuum. The desolventised material is the resin to which volatile oil is added to get oleoresin. The cumin powder after the solvent extraction was taken out from the column, air dried, and designated as spent residue.

2.2. Proximate composition

AOAC methods (1984) were followed for determining the proximate composition viz., moisture, protein, fat, and ash content of the spent residue.

2.2.1. Moisture

Homogenized spent residue from cumin was accurately weighed in a tared aluminum dish and dried overnight (about 16 h) in an oven. It was covered and cooled in a desiccator and then weighed. Sample was dried again for a further 2 h and reweighed until a constant weight was obtained.

2.2.2. Protein

The total nitrogen content in spent cumin sample was determined by micro-Kjeldhal method. A factor of 6.25 was multiplied by % N_2 to get % protein value.

2.2.3. Crude fat

The crude fat was determined by extracting the sample in a Soxhlet apparatus for 16 h using petroleum ether (40–60 °C), evaporated and the residue was weighed to get fat content.

2.2.4. Total ash

This was determined by igniting the spent residue from cumin until white ash was obtained followed by further ashing in a muffle furnace at 550 °C. The weight was recorded after cooling. The difference in weight was expressed as total ash content.

2.3. Carbohydrate profile

The fiber fractions were acid hydrolyzed followed by alditol acetate derivatization and subjected to GC analysis on 3% OV-225 (Chromosorb W, 100–120 mesh) in a Shimadzu gas liquid chromatograph equipped with FID detector at 200 °C (Sawardeker, Slonecker, & Jeanes, 1965).

2.4. Starch content

To the spent residue (0.5–1.0 g), dispersed in 50 ml water, was added heat stable alpha amylase (0.1 ml), then kept in a boiling water bath for 10 min and acetate buffer (pH 4.6) was subsequently added to 0.05 M concentration and equilibrated at 60 °C. To this glucoamylase (50 mg) was added and incubated in a shaking water bath at 60 °C for 2 h. The solution was filtered and made up to a suitable volume and the liberated glucose was determined by the TGO (Tris Glucose Oxidase) method. The glucose value multiplied by a factor 0.9 gave the starch content (Hassid & Neufeld, 1964).

2.5. Dietary fiber

The TDF, a measure of the sum of insoluble and soluble dietary fibers, based on digestion of food samples (1 g) with enzymes, was determined as described by Asp et al. (Asp, Johnos, Hollmer, & Slijestrom, 1983).

2.6. Determination of hydration properties (Robertson et al., 2000)

2.6.1. Water holding capacity

Water holding capacity, defined by the quantity of water that is bound to the fiber without the application of any external force (except for gravity and atmospheric pressure), was determined by accurately weighing dry sample (1 g) into a graduated test tube, and adding around 30 ml of water, and it was allowed to hydrate for 18 h at

ambient temperature. The supernatant was removed by passing through a sintered glass crucible (G4) under vacuum. The hydrated residue weight was recorded and it was dried at 105 °C for 2 h to obtain the residue dry weight.

$$\text{Water holding capacity (g/g)} = \frac{\text{Residue hydrated weight} - \text{Residue dry weight}}{\text{Residue dry weight}}$$

2.6.2. Water retention capacity

Water retention capacity, defined as the quantity of water that remains bound to the hydrated fiber following the application of an external force (pressure of centrifugation) was determined by accurately weighing dry sample (1 g) into a graduated centrifuge tube, adding 30 ml of water and it was hydrated for 18 h, centrifuged (3000*g*, 20 min) and the supernatant solution was removed by passing through a sintered glass crucible (G4) under applied vacuum. The hydrated residue weight was recorded and then sample was dried at 105 °C for 2 h to obtain its dry weight.

$$\text{Water retention capacity (g/g)} = \frac{\text{Residue hydrated weight} - \text{Residue dry weight (After centrifugation)}}{\text{Residue dry weight}}$$

2.6.3. Swelling capacity

Swelling capacity is defined as the ratio of the volume occupied when the sample is immersed in excess of water after equilibration to the actual weight. Accurately weighed dry sample (0.2 g) was placed in a graduated test tube, around 10 ml of water was added and it was hydrated for 18 h, and the final volume attained by the sample was measured.

$$\text{Swelling capacity (ml/g)} = \frac{\text{Volume occupied by sample}}{\text{Original sample weight}}$$

2.7. Isolation of starch

Starch from raw cumin powder (before steam distillation) was isolated by steeping in water followed by centrifugation (Tharanathan, 1995). The crude starch isolate was purified by repeated washings with sodium chloride (0.1 M)-toluene (10:1, v/v) and later by differential sedimentation in water.

2.8. Fourier transform infrared spectroscopy

FTIR spectra of native cumin starch were measured in a Nicolet 5700 FTIR spectrophotometer (from 400 to 4000 cm^{-1}) under dry air at room temperature using KBr pellets. Sample (4 mg) was mixed thoroughly with 200 mg solid KBr, from which 40 mg were taken for pellatization. Reproducibility of the data was verified on two preparations.

2.9. Scanning electron microscope

The dry sample, spread on a double sided conducting adhesive tape, pasted on a metallic stub, was coated (100 μ) with gold in a sputter coating unit for 2 min and observed in a LEO-435-VP electron microscope (LEO electron microscopy Ltd., Cambridge, UK) at 20 kV.

3. Results and discussion

3.1. Proximate composition

Proximate analysis (Table 1) of the cumin spent residue revealed 5.0% residual fat and higher protein content (21.0%) compared to whole cumin (17%). The fiber fractions from the spent residue were isolated as per the scheme shown in Fig. 1. It was found to contain TDF of 62.1%, of which IDF was the major constituent (51.7%). The SDF value (10.4%) of spent cumin was comparable to that of the whole cumin (10.5%). The spent residue had a starch content of 7.7%.

3.2. Effect of particle size on hydration properties

The hydration properties viz., water holding, water retention, and swelling capacity of the fiber actually determine its optimal usage levels in various processed foods for a desirable texture as well as beneficial physiological-functional characteristics (Raghavendra, Rastogi, Raghava Rao, & Tharanathan, 2004). The hydration properties of the spent residue increased with decrease in particle size (Fig. 2). The particle sizes studied were from sieve 18 to 30 (−850 – +699 microns to −500 μ). The increase in the hydration properties with decrease in particle size was due to the higher packing density by the smaller fiber particles, which enhances the surface area for better water absorption and higher swelling capacity.

3.3. Hydration properties of defatted spent

The results (Fig. 3) show that the hydration properties of defatted spent were much better compared to spent res-

Table 1
Proximate composition (% dwb) of spent cumin (dry weight basis)

Components	Cumin spent (%)	Whole cumin (%)
Crude fat	5.0	13.1
Proteins	20.3	16.0
Ash	5.0	6.0
Starch	7.7	8.3
Insoluble dietary fiber	51.7	46.5
Soluble dietary fiber	10.4	10.5
Total dietary fiber	62.1	57.0

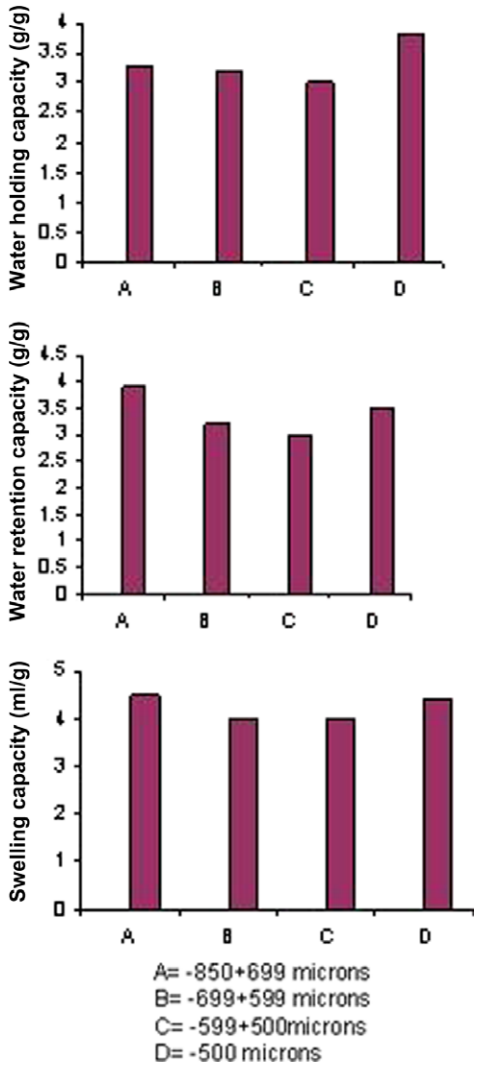

Fig. 1. Effect of particle size on the hydration properties of spent residue from cumin.

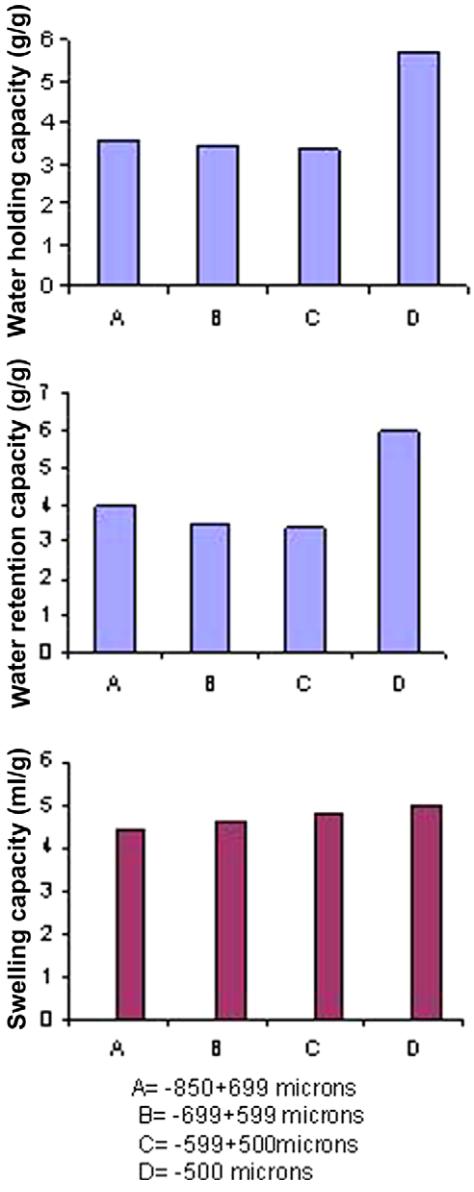

Fig. 2. Hydration properties of defatted spent residue from cumin.

idue with fat (Fig. 2). This may be due to the residual oil getting trapped inside the fiber matrix, thus restricting the entry of water molecules and resulting in decreased hydration properties.

3.4. Analysis of sugars in fiber fractions

By GC analysis, it was found that all six sugars viz., rhamnose, arabinose, xylose, mannose, galactose and glucose were present in both SDF and IDF fractions of spent cumin (Table 2). In cumin spent SDF, arabinose was present in highest concentration 52.3% followed by mannose 31% and rhamnose 6.5% and in IDF mannose was highest 47.0% followed by 44% arabinose and 6.7% xylose. In defatted cumin spent SDF arabinose was 78.8% followed by galactose 6.9% and mannose was highest 46.1% in deffated spent IDF with 37.8% arabinose. Glucose content was almost same in SDF and IDF before and after defatting the value ranging from 1.04 to 1.6%.

3.5. FTIR studies

In the FTIR spectrum of the starch isolated from cumin the –OH stretch appeared as a broad peak at ~3440 cm^{-1} and C–H stretch at around 2800–3000 cm^{-1}, the absorption peak at around 1648 cm^{-1} indicated relatively a higher degree of crystallinity.The peak at 1019 cm^{-1} was attributed to C–O–H bending and deformation, while that at 1640 cm^{-1} was due to O–H related vibration. The broad band at 3400 cm^{-1} was due to unsubstituted hydrogen bonded hydroxyl groups which was attributed to complex vibrational stretches associated with free inter- and intramolecular bound hydroxyl groups. The band at ~2934 cm^{-1} was indeed characteristic of C–H stretches associated with the ring hydrogen atoms. The sharp peak appearing at around 890–900 cm^{-1} was indicative of α-anomeric configuration of the glucan chains. The

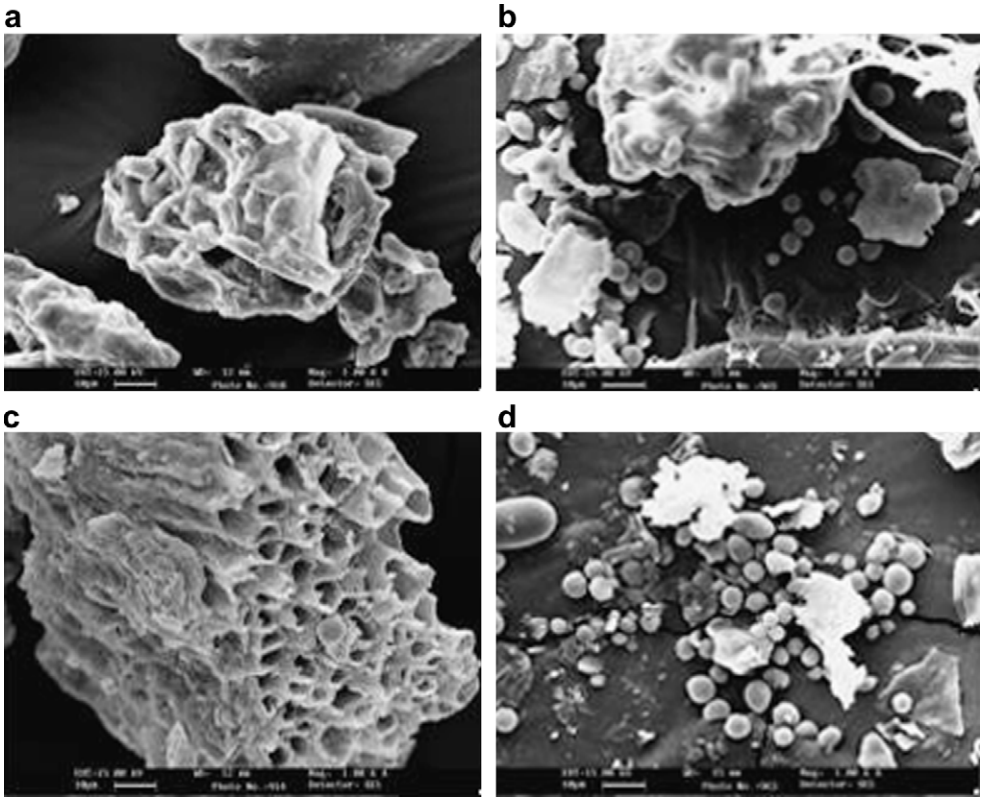

Fig. 3. Scanning electron micrographs of cumin and isolated starch: (a) cumin (native), (b) cumin spent, (c) cumin spent (defatted) and (d) isolated starch from cumin (native).

Table 2
Sugar profile in fiber fractions of spent cumin

Sample		Sugars (g/100 g)					
		Rhamnose	Arabinose	Xylose	Mannose	Galactose	Glucose
Cumin spent	SDF	6.5	52.3	4.8	31.1	3.8	1.4
	IDF	0.3	44.1	6.7	47.0	1.0	1.0
Defatted cumin spent	SDF	2.0	78.8	4.6	6.1	6.9	1.6
	IDF	3.1	37.8	11.4	46.1	0.5	1.2

methylene stretching ($-CH_2$) intensity was seen at 2928 cm^{-1} and carboxyl (–C=O) absorption at 1640 cm^{-1}. The absorption at ~1245 cm^{-1} was indicative of the presence of acetyl group substitution of some of the –OH groups (Mathlonthi & Koenig, 1986).

3.6. Scanning electron microscopy studies

For starch isolation, the solvent defatted spent residue was not found to be suitable, as the isolated 'so-called starch granules' were found to be partially swollen and highly covered (embedded) with fat, cell wall material and fiber matrix. This was probably attributed to the effect of steam distillation, allowing the starch granule hydration with better adhesion to the fiber matrix (may be because starch gelatinizes with steam). On the contrary, starch isolation from the raw cumin powder with successive sedimentations from the aqueous medium furnished spherically shaped starch granules (5.8 μm) in large numbers, attributed to the presence of some cell wall debris. Starch content in the cumin spent residue was 7.7%.

From the SEM studies, it was clear that the native cumin showed very few starch granules embedded well inside the fiber matrix (Fig. 3a), which showed distinct spherical starch granules (Fig. 3b), the residue left after volatile oil and oleoresin extraction showed partly gelatinized starch matrix with a few spherical starch granules, which upon further defatting by treatment with solvent the fiber matrix appeared to have a typical 'honey comb' structure, almost devoid of starch granules (Fig. 3c). Nevertheless, upon further purification by repeated differential sedimentation of spent residue, well separated spherically shaped starch granules of various sizes (5.8 μm) were clearly visible (Fig. 3d). The starch granules appeared to have smooth surface without any roughening, as seen in some legume starch granules (Tharanathan, 1995). Black pepper starch was shown to be unusually small sized (2–2.5 μm) and hav-

ing polygonal shape (Ramadas Bhat & Tharanathan, 1983).

4. Conclusions

Interest on health foods and focus on the health benefits of dietary fiber in the human diet invites the speculation that the spent residue from cumin could provide a new source of inexpensive dietary fiber in selected food products. The dietary fiber content of the spent cumin residue (62.1%) was much higher than that of many fruits and vegetables, the fiber content of which varies from 6 to 17%, viz., barley (12.5%), oats (16.9%) and carrot (58%) (Robertson et al., 2000). This study has revealed that the spent residue from cumin is a rich source of useful dietary fiber. It is also found to be rich in protein (20.3%). Utilization of the spent cumin residue in various functional food formulations augments sufficient value addition for an otherwise waste byproduct from the spice oleoresin industry.

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