JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY
ARTICLE

DOI:10.1021/jf901687r

Preparation of Dietary Fiber Powder from Tiger Nut (*Cyperus esculentus*) Milk ("Horchata") Byproducts and Its Physicochemical Properties

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"Horchata" is a vegetable milk obtained from tiger nuts. The solid waste from horchata production was analyzed for physicochemical and microbial properties, aiming to determine its potential use as a fiber source for the food industry. The solid waste contains a high proportion of total dietary fiber (59.71 g/100 g), composed mainly of insoluble dietary fiber (99.8%). It has a high water-holding capacity (8.01 g/g) and oil-holding capacity (6.92 g/g) and a low water absorption (1.79 g/g) and water adsorption (0.23 g/g) capacities, in comparison with other dietary fiber sources. The emulsifying ability was 70.33 mL/100 mL, and the wastes showed high emulsion stability (100 mL/100 mL). The physicochemical properties indicate that tiger nut byproducts are rich in fiber and may be considered a potential ingredient in a healthy diet. However, the microbial quality was poor, meaning that it must be pasteurized prior to its addition to any food product.

KEYWORDS: Tiger nut (*Cyperus esculentus*); byproduct; horchata; dietary fiber; physicochemical properties

INTRODUCTION

Food-processing byproducts may still contain many valuable substances, such as dietetic fiber, pigments, organic acids, flavors, and antibacterial or antifungal substances (*1*−*4*). Some of these products have been recognized by several organizations (FDA and EFSA) to possess proven health benefits. Public institutions are supporting the food industry in the implementation of new technologies to recover valued-added ingredients from byproducts. Depending on the availability of an adequate technology, these byproducts can be converted into commercial products either as raw materials for secondary processes (intermediate foods ingredients), as operating supplies, or as ingredients of new products. Numerous valuable substances in food production are suitable for separation and recycling at the end of their life cycle, even though the available separation and recycling technologies are not necessarily cost efficient.

Tiger nuts or "chufa" (*Cyperus esculentus* L. var. *sativus* Boeck.) are tubers mainly used to produce "horchata de chufa" (tiger nut milk), yielding a high quantity of byproducts (**Figure 1**). The development of optimized systems for the recovery of valuable compounds will assist in the reduction of wastes. However, as a previous step, these byproducts should be characterized to optimize the recovery-processing techniques. The presence of valuable compounds in horchata de chufa [Protected Designation of Origin (PDO) "Chufa de Valencia"] is not well-documented, although "horchata" has been used in traditional medicine for its antihepatotoxic, choleretic, diuretic, hypocholesterolemic, and antilipidemic properties; none of these properties has been attributed to specific compounds (*5*).

Spain is one of the main producers of horchata de chufa. The annual value of tiger nut production is close to 5 million euros (*5*). In recent years, the popularity of horchata has been extended to other countries, such as the United Kingdom. However, PDO horchata de chufa must be distinguished from different types of horchata based on rice and vanilla, which are from Central and South America. Chufa (or tiger nut) is also used to obtain other nonalcoholic beverages in African countries, such as Mali, where it is known as "kunnu".

Horchata byproducts can form up to 60% of the harvested plant material, the management of which represents an additional problem for the industry (*5*). Until now, the most common disposal method of horchata byproduct (solid and liquid) has been its use as an organic mass for combustion, composting, and animal feed. The term "byproduct", which is common in industry, suggests that these wastes might in fact be usable and have their own market value. Food byproducts can be used for the production of food ingredients, for example, polyphenols, protein isolates, and dietary fibers (DFs) (*3*). Fibers extracted from some grains and seeds exhibit physiological and functional properties that make them promising ingredients for the food industry and for health applications. Food researchers are looking for novel raw materials that meet these needs, with a particular focus on the byproduct or other components from raw materials as legumes (*6*). There is a parallel interest in new sources of DF with a similar profile to those of cereal and legume byproducts

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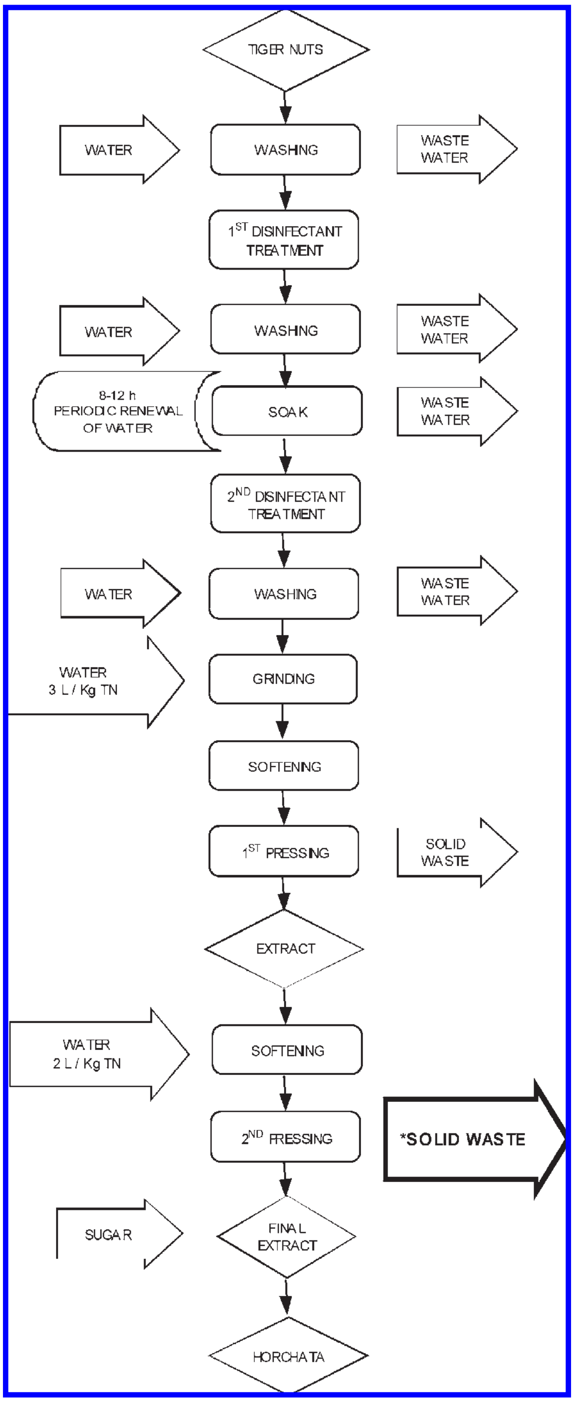

Figure 1. Flowchart of horchata de chufa D.O. Chufa de Valencia production (TN, tiger nut; *solid waste studied).

such as wheat, rice, and oat bran, lupine, etc. (*7*). Fiber source research has focused on tubers, cereals, seeds, vegetables, fruits, and algae, all of which are characterized by their high DF content with low digestibility and low caloric content (*2*, *7*−*20*). Byproducts from tiger nuts (*C. esculentus*) milk production might have similar characteristics. Thus, the aim of this work was to study the properties of horchata solid byproducts, as potential fiber sources for food enrichment.

MATERIALS AND METHODS

Standards and Reagents. All reagents were of analytical grade purity: sulphuric acid, Kjeldahl catalyst (Cu−TiO_2) tablets, boric acid, bromocresol green, methyl red, hydrochloric acid, and petroleum ether were supplied by Panreac (Barcelona, Spain), and sodium hydroxide and tryptone phosphate water (buffered peptone water) were from Scharlab, S. L. (Barcelona, Spain). A total dietary fiber (TDF) assay kit and all other chemicals were obtained from Sigma Chemical Co. (St. Louis, MO). Water was treated in a Milli-Q water purification system (Millipore, Billerica, MA).

Raw Material. The raw material was obtained from a local horchata producer, a member of the "Asociación de Horchateros Artesanales de la Comunidad Valenciana". This Association oversees the production of horchata from tiger nut PDO Chufa de Valencia. Four batches of byproduct were taken during June, July, August, and September, 2008, and transported to the pilot plant facilities of the IPOA Research Group at the Universidad Miguel Hernández (Orihuela, Alicante, Spain) in refrigerated conditions (4 °C). The whole waste was pressed to drain liquid waste and keep the solid residue for further analysis. All tests were carried out in triplicate (4 × 3 = 12 samples). Results are expressed as means ± standard deviations (SPSS 16.0 for Windows, SPSS Inc., Chicago, IL). **Figure 1** describes the flowchart of the horchata de chufa PDO Chufa de Valencia production.

Chemical Analysis. Moisture, ash, protein, and fat contents were determined by AOAC methods (*21*). Moisture (g water/100 g sample) was determined by drying a 3 g sample at 105 °C to constant weight. Ashing was performed on a 2−3 g sample after combustion in a muffle furnace at 550 °C for 8 h (g ash/100 g sample). Protein (g protein/100 g sample) was analyzed according to the Kjeldahl method, using a factor of 6.25 for the conversion of nitrogen to crude protein. Fat (g fat/100 g sample) was calculated by weight loss by extraction for 8 h with petroleum ether in a Sohxlet apparatus.

DF. TDF and insoluble dietary fiber (IDF) were determined following 985.29 AOAC method (*21*). Soluble dietary fiber (SDF) was calculated by subtracting the IDF proportion from the TDF.

Physicochemical Analysis. The pH was measured in a suspension resulting from blending a 10 g sample with 10 mL of deionized water for 2 min using a pH meter (model pH/Ion 510, Eutech Instruments Pte Ltd., Singapore). The water activity (A_w) was determined in a Novasina Thermoconstanter Sprint TH-500 (Pfäffikon, Switzerland). The color was studied in the CIELAB color space using a Minolta CM-2600d (Minolta Camera Co., Osaka, Japan), with D_{65} as an illuminant and an observer angle of 10°. Low reflectance glass (Minolta CR-A51/1829-752) was placed between the samples and the equipment. The CIELAB coordinates studied were lightness (L^*), coordinate red/green (a^*), and coordinate yellow/blue (b^*).

Functional Properties. The water-holding capacity (WHC) and oil-holding capacity (OHC) were determined according to Robertson et al. (*22*). The WHC was expressed as g of water held per g of sample, and the OHC was expressed as g of oil held per g of sample. The water adsorption capacity (WA_dC) and water absorption capacity (WA_bC) were determined according to Vázquez-Ovando et al. (*7*). The WA_dC was expressed as g of water adsorbed per g of sample, and the WA_bC was expressed as g of water absorbed per g of sample. The emulsifying activity (EA) and emulsion stability (ES) were evaluated according to Chau et al. (*23*) with slight modifications. One hundred milliliters of 2% (w/v) sample suspension in water was homogenized at 11000 rpm for 30 s using an homogenizer IKA T-25. One hundred milliliters of sunflower oil was then added and homogenized for another 1 min. The emulsions were centrifuged in 10 mL graduated centrifuged tubes at 1200 g for 5 min, and the volume of the emulsion left was measured. The EA was calculated as the volume of emulsified layer/volume of whole layer in centrifuge tube × 100. To determine the ES, emulsions prepared by the above procedures were heated at 80 °C for 30 min, cooled to room temperature, and centrifuged at 1200*g* for 5 min. The ES was calculated as the volume of remaining emulsified layer/original emulsion volume × 100.

Microbial Analysis. Serial dilutions of samples were prepared in sterile peptone water for most microbial determinations and in the De Man, Rogosa, Sharpe (MRS) broth for lactic acid bacteria. Total viable counts (TVC) were determined by plating the diluted samples on TVC 3 M Petrifilm plates followed by incubation at 37 °C for 48 h, lactic acid bacteria (MRS broth) and anaerobic bacteria on TVC 3 M Petrifilm plates incubated at 37 °C for 48 h in anaerobic conditions, psychotrophic bacteria on TVC 3 M Petrifilm plates incubated at 8 °C for 10 days, enterobacteria on 3 M Petrifilm plates for enterobacteria incubated at 37 °C for 24 h,

Table 1. Proximate Composition of Solid TNBP (Mean Values ± SD), Tiger Nuts, and Horchata de Chufa (g/100 g Fresh Weight)

component	TNBP	tiger nuts (*5*, *24*)	"Horchata de chufa" (*5*, *25*)
moisture	61.23 ± 4.12	6.72	88.00
protein	1.75 ± 0.12	5.05	0.91
fat	8.85 ± 1.11	24.50	3.09
ash	0.99 ± 0.24	1.43	0.25
TDF	59.71 ± 0.03	10.30	1.06

Table 2. TDF (g/100 g db) of TNBP (Mean Values ± SD) as Compared to Other DF Powders from Vegetable Processing Waste

product	TDF (g/100 g product db)
oat bran (*2*)	8.23
kivi (*2*)	25.80
rice bran (*2*)	27.42
peaches (*2*)	36.00
cabbage outer leaves (*2*)	40.50
pears (*2*)	43.90
apples (*2*)	44.00
carrots (*2*)	48.00
guava (*12*)	48.55
mandarin (*13*)	52.89
carob (*14*)	53.00
jack bean (*7*)	55.88
chia (*7*)	56.46
orange peel (*7*)	57.00
grapefruit (*16*)	58.60
TNBP	59.71
brewer's dried grain (*2*)	60.00
cocoa hulls (*15*)	60.54
coconut (*33*)	63.24
passion fruit (*38*)	63.25
lime (*11*)	64.30
cauliflower (*2*)	65.00
pineapple (*20*)	70.60
dates (*2*)	71.00
orange byproduct (*10*)	71.62
mango (*35*)	74.00
grapes (*2*)	77.89
olives (*2*)	80.00
chile (*2*)	80.41
peas (*2*)	82.30

Escherichia coli on 3 M Petrifilm plates for *E. coli* incubated at 37 °C for 24 h, *Staphylococcus aureus* on 3 M Petrifilm Staph express plates incubated at 37 °C for 24 h, *Clostridium perfringens* on SPS agar at 37 °C for 24 h in anaerobic conditions, and molds and yeasts on Rose Bengal plates with chloramphenicol incubated at 28 °C for 5 days.

RESULTS AND DISCUSSION

Solid Waste Yield. The average yield after liquid/solid residue separation was 47.12±1.97% for solid residue and 52.88 ± 1.97% wastewater (drained).

Chemical Analysis. The proximate composition analysis of solid tiger nut byproduct (TNBP) (**Table 1**) showed higher protein, fat, and ash contents than horchata de chufa but lower than tiger nuts (*24*, *25*). The moisture content of horchata de chufa was higher than that of the TNBP, which retains most of the solid components of the raw material (tiger nut).

TDF, IDF, and SDF. The term DF is used to denote plant substances (edible parts of plants) that are resistant to hydrolysis by digestive enzymes in humans and contains membrane components as well as endocellular polysaccharides. In chemical terms, the definition of DF refers mainly to the sum of nonstarch polysaccharides and lignin but also includes nonstructural components (gums and mucilages), as well as industrial additives (modified cellulose, modified pectin, commercial gums, and algal polysaccharides). Fiber is classified into soluble (pectins and gums) and insoluble groups (cellulose, lignin, and some hemicellulose) (*26*).

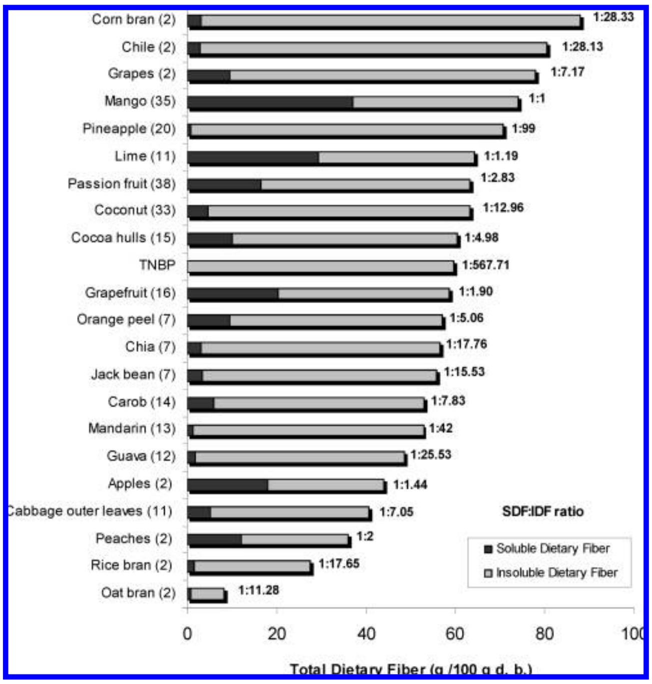

Figure 2. TDF (g/100 g db) and SDF:IDF ratio of TNBP as compared to other DF powders from vegetable processing waste.

Table 3. Physicochemical Properties of TNBP (Mean ± SD) as Compared to Other DF Powders from Lemon and Sugarcane Processing Waste

parameter	TNBP	lemon byproduct (*3*)	sugar cane bagasse (*32*)
water activity (24.9 °C)	0.99 ± 0.00	0.96 ± 0.01	
pH (15 °C)	6.73 ± 0.03	3.96 ± 0.01	
L^*	75.29 ± 1.04	66.98 ± 0.36	57.89
a^*	2.17 ± 0.32	−2.63 ± 0.22	3.47
b^*	17.11 ± 0.33	27.44 ± 0.52	15.60

Table 2 shows the TDF of TNBP as compared with other DF powders from vegetable processing wastes. The TDF content of the TNBP was 59.71 g/100 g ± 0.03 (**Table 2**), mainly IDF (59.610 g/100 g ± 0.08; 99.82% from TDF) and a little SDF (0.105 g/ 100 g; 0.17% from TDF) (**Figure 2**). **Figure 2** shows dietary fractions (insoluble and soluble) and the SDF:IDF ratio of TNBP as compared with other DF powders from vegetable processing wastes. As can be seen from **Table 2**, TNBP had more TDF than oat bran, rice bran, or orange peel (*1*, *2*, *10*) but less TDF than cauliflower, pineapple, or dates wastes (*2*, *20*, *26*).

The TNBP had a higher SDF/IDF ratio than that reported for fibrous residues from other DF byproducts (**Figure 2**). The high IDF content of TNBP points to a promising application in food products. IDF ingestion causes the sensation of satiety, since it absorbs water and increases bolus size. It also increases the volume and weight of the fecal bolus, promoting improved functioning of the digestive system and preventing disorders such as constipation and colon cancer (*7*). DF from several sources (vegetable, fruit, cereal, etc.) can increase the nutritional value of different foods like bread, cookies, muffins, cake (*27*), meat products (*1*), fish products (*28*), and milk products (*29*); thus, TNBP can be considered a potential ingredient for these types of food.

Physicochemical Analysis. Table 3 shows the physicochemical properties of TNBP as compared with other DF powders from

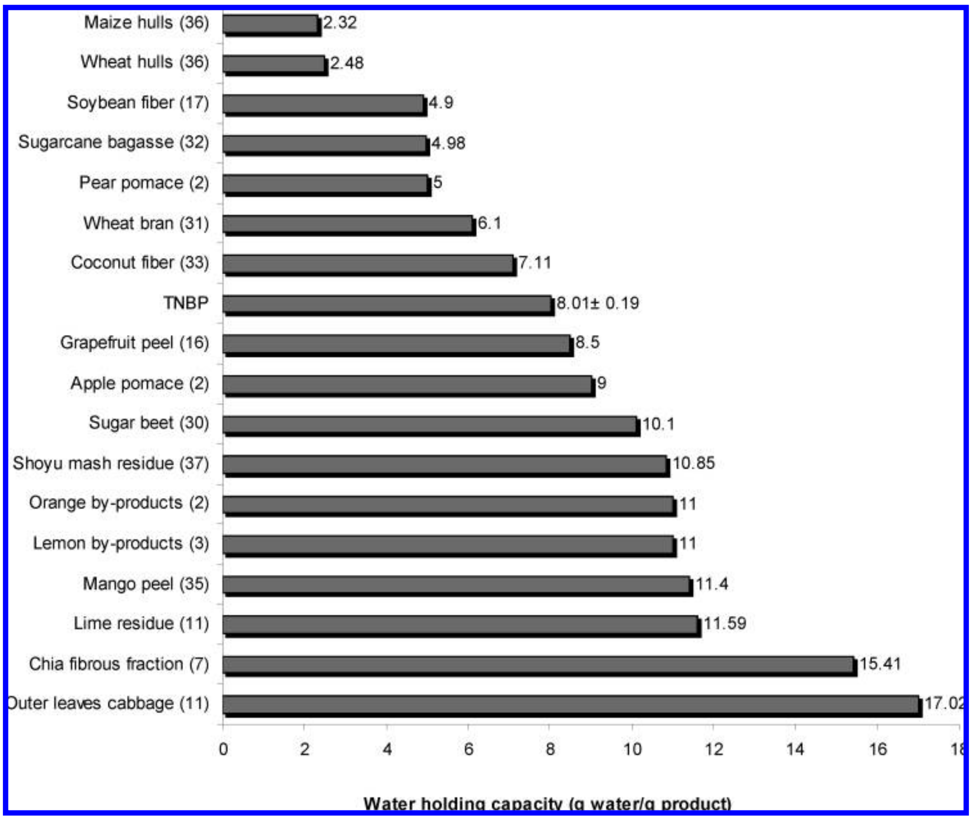

Figure 3. WHC of TNBP as compared to other DF powders from vegetable processing waste.

lemon and sugar cane processing wastes. The TNBP water activity and pH values were higher than those of lemon byproducts (**Table 3**), probably due to the higher moisture in TNBP. TNBP is a highly perishable product due to its high pH and A_w, which will favor microbial growth. TNBP may be a safe ingredient if its addition is followed by heat treatment (baking, cooking, etc.).

Color is one of the most important quality parameters in foods. Undoubtedly, TNBP addition to food would cause color changes in the product and so influence the organoleptic properties, which may limit its potential applications. TNBP color coordinates are similar to those of sugar cane, although the L^* is higher. The L^* values in TNBP samples were also higher than those of lemon byproducts (*3*) (**Table 3**), because of the higher moisture content in TNBP; this property suggests that the product is appropriate for incorporation in different foods. Some authors have reported that the increase in water in meat products results in lighter products (*1*). The low a^* and b^* values recorded showed that the incorporation of TNBP would not cause great modification of redness or yellowness in the product. In short, color properties of TNBP show their suitability as an ingredient in a large variety of food products, especially in meat and fish products, which may mask TNBP color and will benefit from the incorporation of moisture.

Functional Properties. *WHC.* The WHC is the ability of a moist material to retain water when subjected to an external centrifugal gravity force or compression. It consists of the sum of bound water, hydrodynamic water, and, mainly, physically trapped water (*7*). DF holds water by adsorption and absorption phenomena. Some water is also retained outside the fiber matrix (free water). Particle size, chemical composition, and structure of DF influence the WHC. The hydration properties of DFs determine their optimal usage levels in foods as they provide desirable texture properties (*30*). The WHCs of TNBP are presented in **Figure 3**, together with those of other DF powders from vegetable processing wastes. Tiger nuts exhibited a WHC 8.01 times its own weight (**Figure 3**). This is higher than that reported for fibrous residues from soybean (*31*), sugar cane (*32*) , pear (*2*), and coconut fiber (*33*) but lower than the fibrous residues of some fruits with a higher SDF content, such as citrus byproduct (*3*,*34*) . WHC depends on fiber processing and also on its chemical and physical structure, and it is also related to the SDF content (*3*) . TNBP had the lowest SDF content (**Figure 2**). This proves that even though the SDF content of TNBP was relatively low, it still had a high WHC, due to the high proportion of hemicellulose and lignin (both have certain WHC) in the TNBP that may have increased its WHC. The fiber structure may also increase WHC (*10*) . Some vegetable fibers with high WHC have been added to meat and fish products (*1*, *28*) . TNBP has potential applications in products requiring hydration, viscosity development, and freshness preservation, such as baked foods or cooked meat products.

OHC. **Figure 4** shows the OHC of TNBP as compared with other DF powders from vegetable processing wastes. TNBP had an OHC of 6.92 g oil/g product (**Figure 4**). This is similar to the OHC of pea fibrous residues (*2*). It is higher than most of the vegetable processing wastes found in the literature but lower than the OHC of asparagus (*9*) and date fiber (*26*). OHC is also a functional property related to the chemical structure of the plant polysaccharides and depends on the chemical and physical structure (*10*); it is also related with the IDF content (*30*). When IDF is added to any formulation, it can absorb the oil present, and the extent of this absorption is measured as the fat absorption capacity (*33*). OHC is important to flavor retention and product yield especially for cooked meat products, which normally lose fat during cooking (*30*). The WHC and OHC had similar values (**Figures 3** and **4**). It has been shown that other fruit fibers with similar WHC and OHC values are useful as emulsifiers for meat

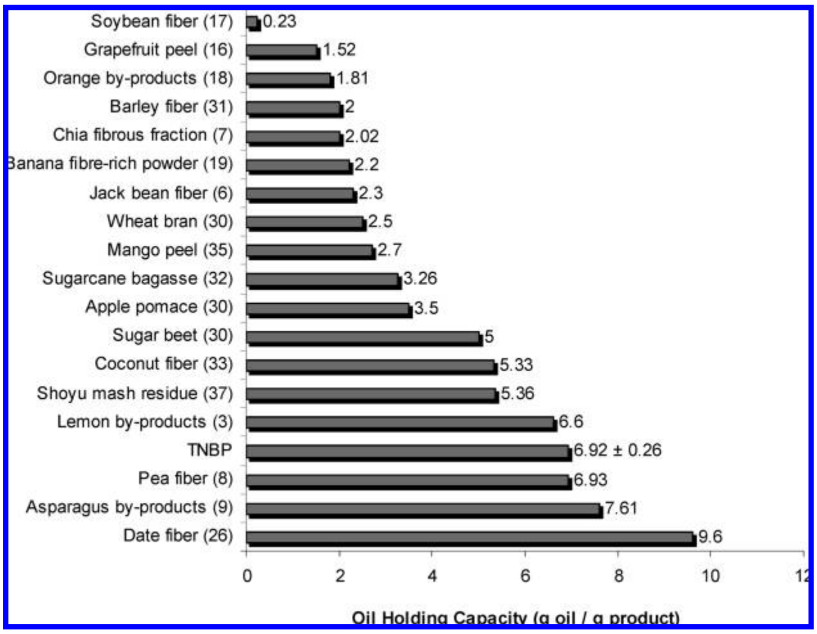

Figure 4. OHC of TNBP as compared to other DF powders from vegetable processing waste.

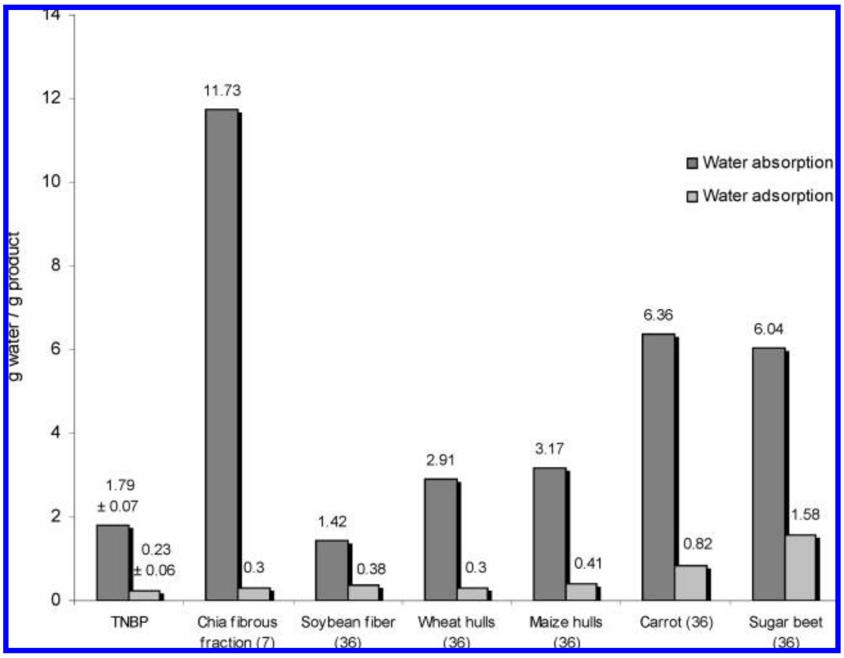

Figure 5. Water absorption (WA_bC) and adsorption (WA_dC) capacities of TNBP as compared to other fiber sources.

products and as thickening or bulking agents (*35*). The fiber particle size may influence OHC; smaller particles have relatively wider surface areas and therefore would theoretically be able to hold more oil than higher particles (*7*). In this sense, TNBP particles may be small enough to provide a high OHC. Because of its high OHC, TNBP is a potential ingredient for cooked meat products but not for fried products since it would provide a greasy sensation.

WA_bC and WA_dC. WA_bC is indicative of a structure's aptitude to spontaneously absorb water when placed in contact with a constantly moist surface or when immersed in water, while the WA_dC is the ability of a structure to spontaneously adsorb water when exposed to an atmosphere of constant relative humidity. It is initially a surface phenomenon, but at higher hydration levels, absorption can occur inside the structure, leading to swelling and eventual solubilization (*7*).

The WA_bC and WA_dC of TNBP as compared with other fiber sources are presented in **Figure 5**. The TNBP had a WA_bC of 1.79 g water/g product (**Figure 5**), which is almost 10 times lower than that of chia (*Salvia hispanica* L.) fibrous fraction (*7*) and three times lower that of carrot (6.36 g water/g sample) and beet (6.04 g water/g sample) (*36*) but similar to that of soybean fiber (*36*) (**Figure 5**). WAbC depends on the vegetable cell wall composition, meaning that the vegetable type affects this property. The SDF has good WA_bC (*7*) , so the low WA_bC of TNBP may be due to its low SDF content. A possible additional effect may be due to the low protein content of TNBP (1.75 g/100 g dry basis) (**Table 1**). In this case, the low degree of interactions among proteins and water may have accounted for the low WA_bC observed (*37*) .

The TNBP WA_dC was 0.23 g water/g product, which is substantially lower than that of carrot (0.82 g water/g product) and beet (1.58 g water/g product) (*36*) and similar to those of chia

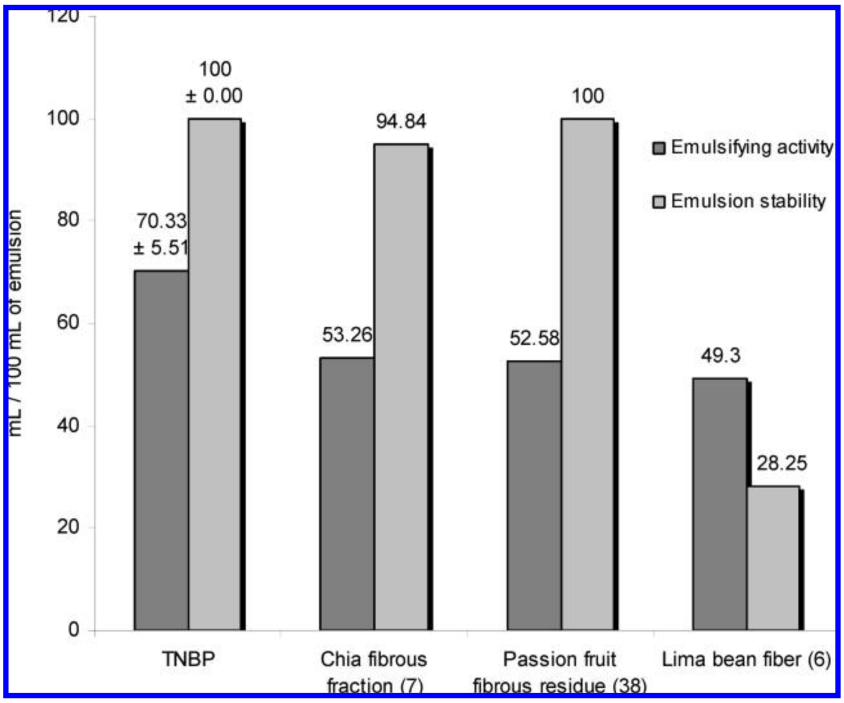

Figure 6. EA and ES of TNBP as compared to other fiber sources.

Table 4. Microbial Counts in TNBP [Log Colony-Forming Units (CFU)/g ± SD]

microorganism	log CFU/g ± SD
total viable counts	5.80 ± 0.28
molds and yeasts	4.95 ± 0.04
psychotrophic bacteria	4.32 ± 0.06
enterobacteriaceae	2.91 ± 0.08
anaerobic bacteria	5.43 ± 0.07
lactic acid bacteria	5.41 ± 0.10
C. perfringens	absence in 0.1 g
S. aureus	2.18 ± 0.04
E. coli	absence in 0.1 g

fibrous fraction (*7*), maize, wheat, and soy hulls (*36*) (**Figure 5**). The IDF is responsible for most WA_dC since it adsorbs water like a sponge; however, no direct relation between IDF and WA_dC was observed here or in commercial products with similar IDF contents (*38*).

EA and ES. The emulsifying capacity is a molecule's ability to act as an agent that facilitates solubilization or dispersion of two immiscible liquids, and ES is the ability to maintain the integrity of an emulsion. **Figure 6** shows the EA and ES of TNBP as compared with other fiber sources. The EA of the TNBP was 70.33 mL/100 mL, and its ES was 100 mL/100 mL. This EA is higher than that of chia (*7*), fiber-rich passion fruit powders (*38*), and *Phaseolus lunatus* fibrous residue (*6*) (**Figure 6**). The low protein content of TNBP (1.75 g/100 g db) would not explain its high EA and ES, since most proteins are strong emulsifying agents (*23*). However, it has to be noted that passion fruit fiber (*38*), which has lower EA than TNBP, but an identical ES, does not have a high protein content. Given that its ES is greater than 100 mL/100 mL, the TNBP studied here may be a good emulsifying agent. It can therefore be appropriated for foods requiring emulsifiers and those with long shelf lives that require long stability. The EA of a fibrous residue is also a good indicator of its ability to adsorb bile acids. This has potential health benefits since the fibrous component adsorbs bile acids and increases feces excretion, consequently limiting the absorption of these acids in the small intestine and therefore reducing blood cholesterol levels. Consequently, incorporation of TNBP into foods could have a potential hypocholesterolemic effect (*7*).

Microbiological Quality. **Table 4** shows the microbial counts in TNBP. TNBP microbial counts (**Table 4**) were high, as expected, because tiger nut is a small tuber and grows in the soil (*5*). Those values are higher than the microbial counts of other fruit byproducts (*3*, *10*) . However, the absence of *C. perfringens* and *E. coli* supports its possible use in food. Thus, from a microbiological point of view, TNBP must be pasteurized prior to its use in food product or either its addition should be followed by a heat treatment.

The results of this study indicate that tiger nut fiber-rich fraction may be considered a potential ingredient in food products, increasing their content of TDF and improving their technological properties. However, its microbial quality is poor, so it must be pasteurized before it can be added to any food product.

ACKNOWLEDGMENT

We thank Consejo Regulador de la Denominación de Origen Chufa de Valencia and Heladería-Horchatería Navia for kindly providing the vegetable residue samples for use in the study.

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Received May 20, 2009. Revised manuscript received July 21, 2009. Accepted July 21, 2009. We acknowledge the economic support from Caja Mediterráneo (CAM).