

Chemical, Physicochemical and Functional Characteristics of Dietary Fiber Obtained from Asparagus Byproducts (*Asparagus officinalis* L.)

Características Químicas, Físicoquímicas y Funcionales de la Fibra Dietaria Obtenida de los Subproductos del Espárrago (*Asparagus officinalis* L.)

Edith Lorena Agudelo Cadavid¹; Diego Alonso Restrepo Molina² y José Régulo Cartagena Valenzuela³

Abstract. Due to its mechanical and biochemical properties, dietary fiber is part of a healthy diet. Containing good levels of prebiotics, asparagus (*Asparagus officinalis* L.) produces healthy effects when incorporated into processed foods. The objective of the current study was to obtain fiber from asparagus byproducts and determine its chemical composition [Total Dietary Fiber (TDF), protein, moisture and ash] and physicochemical [pH, and water activity (a_w)] and functional characteristics [Water Absorption Index (WAI), Water Solubility Index (WSI), Oil Absorption Index (OAI) and Swelling Volume (SV)]. The plant material was treated through extraction and dehydration thermal treatments. A response surface model was applied to evaluate the effects of extraction and drying temperatures. The TDF ranged from 60.7 to 79%. Significant differences were only observed for TDF, moisture and a_w ($P \leq 0.05$). The WAI, WSI, OAI and SV were found to be within the range observed for similar plant materials. Due to their functional properties and elevated TDF content, asparagus byproducts can be used as active biological components in food production. This innovative utilization will contribute to reducing the environmental impact of the industrial processing of this vegetable.

Key words: Natural fiber, drying conditions, healthy food, product development.

Resumen. Por sus propiedades mecánicas y bioquímicas, la fibra dietética puede formar parte de una alimentación saludable. Al ser incorporado en los alimentos en el proceso de elaboración, el espárrago (*Asparagus officinalis* L.) tiene efectos benéficos para la salud del consumidor, debido a su contenido de prebióticos. El objetivo del estudio fue obtener fibra a partir de los subproductos del espárrago para determinar su composición química [Fibra Dietaria Total (FDT), proteína, humedad y cenizas] y sus propiedades físicoquímicas [pH y actividad del agua (a_w)] y funcionales [Índice de Absorción del Agua (IAA), Índice de Solubilidad del Agua (ISA), Índice de Absorción de Aceite (IAAC) y Volumen de Hinchamiento (VH)]. El material vegetal se sometió a tratamientos térmicos de extracción y secado. Se ajustó un modelo de superficie de respuesta para evaluar el efecto de las temperaturas de extracción y secado. La FDT estuvo entre 60,7 y 79%. Sólo los contenidos de FDT, humedad y a_w mostraron diferencias significativas ($P \leq 0,05$). Los valores de IAA, ISA, IAAC y VH estuvieron dentro de los niveles establecidos para otros materiales vegetales similares. Los subproductos del espárrago, dado su alto contenido de FDT y sus propiedades funcionales, pueden ser utilizados como elemento biológico activo en la elaboración de alimentos, contribuyendo a la reducción del impacto ambiental derivado del proceso agroindustrial de la hortaliza.

Palabras clave: Fibra natural, condiciones de secado, alimentos saludables, desarrollo de productos.

The food industry generates large amounts of solid and liquid waste products. This not only creates a contamination problem, but also the loss of valuable biomass and nutrients (Sánchez *et al.*, 2009). These waste products are usually used as animal feed or fertilizers. Nonetheless, aspects such as environmental contamination prevention, energy saving and the need for biodegradable materials have become more and more important in recent years. For this reason, food waste treatment, management methods and policies have been introduced for the recovery, bio-conversion and utilization of valuable components found in these materials (Laufenberg *et al.*, 2003; Tron, 2010).

The use of organic residues in the development of innovative products constitutes one of their promising possibilities. Dietary fiber (DF) can be used as an ingredient of functional foods, where it might perform as an additive with specific processing functions or final product benefits. Within a balanced diet, the combination of DF with at least one additional property can provide health benefits (Shieber *et al.*, 2001). The demand for fruit and vegetable products as sources of DF is currently increasing due to their high nutritional quality, total and soluble fiber contents, fermentation and water retention levels, higher antioxidant capacity and lower caloric content (Rodríguez *et al.*, 2006).

¹ Food Engineer. M.Sc. Consultant at Actuar Famiempresas. Medellín, Colombia. <edith79lo@yahoo.com>

² Associate Professor. Universidad Nacional de Colombia - Sede Medellín - Facultad de Ciencias Agrarias - Departamento de Ingeniería Agrícola y Alimentos. A.A. 17779, Medellín, Colombia. <darestre@unal.edu.co>

³ Full Professor. Universidad Nacional de Colombia - Sede Medellín - Facultad de Ciencias Agrarias - Departamento de Agronomía, A.A. 1779, Medellín, Colombia. <jrcartag@unal.edu.co>

Asparagus is a healthy and nutritious vegetable and, from an economic standpoint, an important crop worldwide. In some countries, it has been used as an anti-inflammatory (Jang *et al.*, 2004), anti-fungal (Negi *et al.*, 2010), and anticancer medicine (Zhao *et al.*, 2012). Asparagus stems are usually processed as canned foods, but about half the total length of each stalk is discarded, which represents an important loss for the producers. Nevertheless, these byproducts have a similar composition to that of the edible part of the plant, thus constituting a promising source of value adding new compounds (phytochemicals and fiber) (Nindo *et al.*, 2003).

DF structure and composition are species specific, depending on cell wall components (*e.g.*, hemicelluloses, hydrocolloidal celluloses and lignin) (Habibi, 2011) and complexity, ripening stage and the part of the plant that is used for consumption (García *et al.*, 2008). Furthermore, DF related processes may lead to meaningful changes in vegetable structure and properties (Mongeau and Brooks, 2003; Waldron *et al.*, 2003). Water retention capacity, particle size distribution and apparent viscosity are known to be important characteristics of DF, with significant influence on food technology. Hydration properties are important in the food industry because liquid retention influences ingredient functionalities, product yield and storage stability (Garau *et al.*, 2006; Femenia *et al.*, 2009).

The processing and dehydration of DF may cause irreversible modifications affecting its original structure and composition and, consequently, the final quality of the product (Garau *et al.*, 2007; Rosell *et al.*, 2009). In this context, the objective of the current study was to obtain DF from asparagus by-products, in order to determine its chemical composition, as well as its functional and physicochemical properties.

MATERIALS AND METHODS

Plant material. Raw material was provided by *Espárragos Erupción S.A.* (Manizales, Colombia). The stems were cut to obtain the upper 15 cm segment (the edible portion), while the remnant (12-15 cm spear) is considered a byproduct. Over the next 24 h, this material was preserved at 4 °C and sent to the laboratory.

Fiber extraction. The asparagus byproduct stems were initially selected, weighed and washed for 15 min at room temperature with a 2% (v/v) detergent

solution, after which they were disinfected for 10 min by immersion in a 200 ppm organic solution. Next, they were cut, homogenized and drained, and then subjected to particle size reduction in a cutting device (R302, Robot Coupe®) to obtain average sizes of 5-10 mm. Once cut, the material was homogenized with water at a 1:1 (solid: liquid) proportion (m/v) and treated at different temperatures (19.8 to 55.1 °C) for 60 min in order to extract the fiber; next the sample was centrifuged (Rotofix 32A, Hettich®). For 16 h, the resulting wet material was oven-dried (FD23, BINDER, Tuttlingen®) by forced air circulation at temperatures between 47.9 and 62.0 °C. After this period, a sample was taken to make sure the moisture was below 9% (dry base) (Santana, 2005). The obtained material was weighed with precision scales and packed in hermetically zippered polyethylene bags. This product was immediately taken to a hammer mill (Atlas®) to obtain a 0.12 to 0.25 mm particle size.

Experimental design. The Response Surface Methodology (RSM) was applied to evaluate the effects of extraction and drying temperatures (Torres *et al.*, 2003). The experiments were randomly conducted following a Rotatable Central Composite Design with a total of 12 combinations including four repetitions of the central points and two axial points. The design matrix was coded for the extraction (X_1) and drying (X_2) temperatures, respectively exploring the 19.8 - 55.1 °C and the 47.9 - 62.0 °C ranges. The action of these factors on the chemical composition and physicochemical and functional properties of the studied material was assessed by applying the second grade polynomial model expressed in the equation below (Piña *et al.*, 2006):

$$Y = \beta_0 + \sum_{i=1}^2 \beta_i X_i + \sum_{i=1}^2 \beta_{ii} X_i^2 + \sum_{i=1}^2 \sum_{j=1}^2 \beta_{ij} X_i X_j$$

Where Y is the estimated response; β_0 , β_i , β_{ii} and β_{ij} are constant coefficients of the regression model, and X_i , X_j and X_{ij} represent the linear and quadratic effects and the interaction of the independent variables, respectively. Table 1 presents the total number of experimental runs with their corresponding temperature combination treatments. The response surface diagrams were obtained from the regression equations, keeping the response variable on the Z axis and the independent variables on the X and Y axes.

At a 0.05 significance level, an Analysis of Variance (ANOVA) was conducted to evaluate the influence of the independent variables. All analyses were

conducted with the statistical software package Statgraphics Centurion XV (Version 2.15.06, Chicago, Illinois, USA). Additionally, the Lack of Fit test (LOF)

(Christensen, 2003) was employed to determine whether the selected model was adequate to describe the obtained data.

Table 1. Matrix of the rotatable central composite design coded to evaluate the processing of asparagus byproduct dietary fiber.

Treatment	Coded variable levels	
	X ₁ Extraction temperature (°C)	X ₂ Drying temperature (°C)
1	37.5	55.0
2	37.5	47.9
3	55.1	55.0
4	37.5	55.0
5	25.0	50.0
6	37.5	55.0
7	19.8	55.0
8	25.0	60.0
9	50.0	50.0
10	50.0	60.0
11	37.5	62.0
12	37.5	55.0

Chemical and physicochemical properties

Total Dietary Fiber (TDF). The non-enzymatic gravimetric method (AOAC 993.21 - 2005) recommended for analyzing the DF in foods and food products containing 2% starch was employed. One of the duplicates was analyzed for raw protein and the other one for ash.

Protein. was measured through the Kjeldahl method for nitrogen determination using the default protein factor of 6.25, following the AOAC 920.152 - 2005 method.

Moisture. was deduced from oven-dehydration at 103 °C, which was carried out until reaching constant weight, following the AOAC 925.45 - 2005 method.

Ash. was determined through dry-oven incineration at 550 °C, following the AOAC 942.05 - 2005 method.

pH. was measured with a Metrohm® 744 potentiometer in a mixture containing a 10 g sample

of the studied material with 100 mL of deionized water.

Water activity (a_w). was determined with an Aqualab® Decagon Devices Inc. 3.2 meter.

Functional properties

Water Absorption Index (WAI). One gram samples of the studied material were suspended in previously weighed 100-mL-capacity centrifuge tubes containing 25 mL of distilled water at 25 °C each. This mixture was stirred for 30 min and then centrifuged at 3,000 rpm for 10 min. The supernatant was transferred to a Petri dish of known weight and the centrifuge tube containing the pulp was weighed to determine the WAI (g of water/g of dry matter) (Fuentes *et al.*, 2009).

Water Solubility Index (WSI). Was obtained with the same methodology employed for the WAI. The supernatant was transferred to Petri dishes, which were oven-treated for approximately 24 h (Fuentes *et al.*, 2009).

Oil Absorption Index (OAI). Was determined under the same conditions of the WAI. The supernatant of each sample was disposed. The OAI was expressed in grams of oil/grams of dry matter (Fuentes *et al.*, 2009).

Swelling Volume (SV). Was measured with a graduated cylinder to which 1 g of the sample was mixed with an excess of distilled water. This suspension was agitated for 30 min in order to completely hydrate the sample, then allowed to stand for approximately 24 h. The volume measured after swelling was subtracted from that initially occupied by the mixture. The value was expressed in mL g⁻¹ of dry matter (Fuentes *et al.*, 2009).

RESULTS AND DISCUSSION

Chemical and physicochemical properties

The largest TDF content (about 79%) was measured on those samples subjected to the 55.1 °C extraction temperature and 59.5 °C drying temperature, probably as a consequence of soluble component depletion during the process (Table 2, Figure 1a). The ANOVA (Table 3) indicates that the fiber extraction temperature determined significant differences ($P \leq 0.05$). Neither the drying temperature nor the interactions showed any

significant effect on the response variable across the treatments. On the other hand, the lack of significance of the LOF test confirms that the proposed model accounts for the obtained data at a 95% confidence level. The lowest TDF count (less than 62%) was found in those samples extracted at 25 °C. The values found for this parameter in the present study, which ranged from 60.7% to 79.0%, are similar to those of Fuentes *et al.* (2009), and higher than the 49% record from previous analyses conducted on asparagus byproducts (Grigelmo and Martin, 1999). This contrast might be due to processing differences because the samples of the referenced research work had only been washed with water and then oven-dried. The TDF contents are usually ranked as low (30 to 50%); intermediate (50 to 70%); and high (70 to 90 %) (Jiménez *et al.*, 2001; Figuerola *et al.*, 2005; Rodríguez *et al.*, 2006). The current asparagus byproduct results corresponded to the intermediate rank.

The protein content varied from 9 to 11.7%, the most important value corresponding to those samples extracted at 19.5 °C and dried at 62.0 °C (Figure 1b). The average record of this parameter was lower than that obtained in another study on the same species (Fuentes *et al.*, 2009), but higher than that of other

Table 2. Chemical composition and physicochemical properties of the asparagus byproduct dietary fiber obtained through the different extraction and drying temperatures.

Treatment	TDF	Protein	Moisture	Ash	pH	a_w
	(%)					
1	70.9	11.7	5.83	4.04	5.85	0.371
2	73.5	9.9	6.13	2.85	5.81	0.376
3	78.0	9.0	5.94	1.96	5.76	0.323
4	74.5	9.0	5.67	2.74	5.73	0.375
5	71.5	11.0	6.17	3.85	5.87	0.375
6	75.5	9.0	5.55	2.73	5.73	0.363
7	72.9	9.8	5.53	3.55	6.05	0.335
8	60.7	11.8	5.74	4.20	5.89	0.343
9	72.2	10.9	5.94	3.34	5.71	0.373
10	72.6	10.6	5.40	3.08	5.85	0.334
11	72.9	10.6	5.33	3.16	5.77	0.321
12	72.4	10.1	5.77	2.94	5.75	0.362

*Percentage expressed in dry base

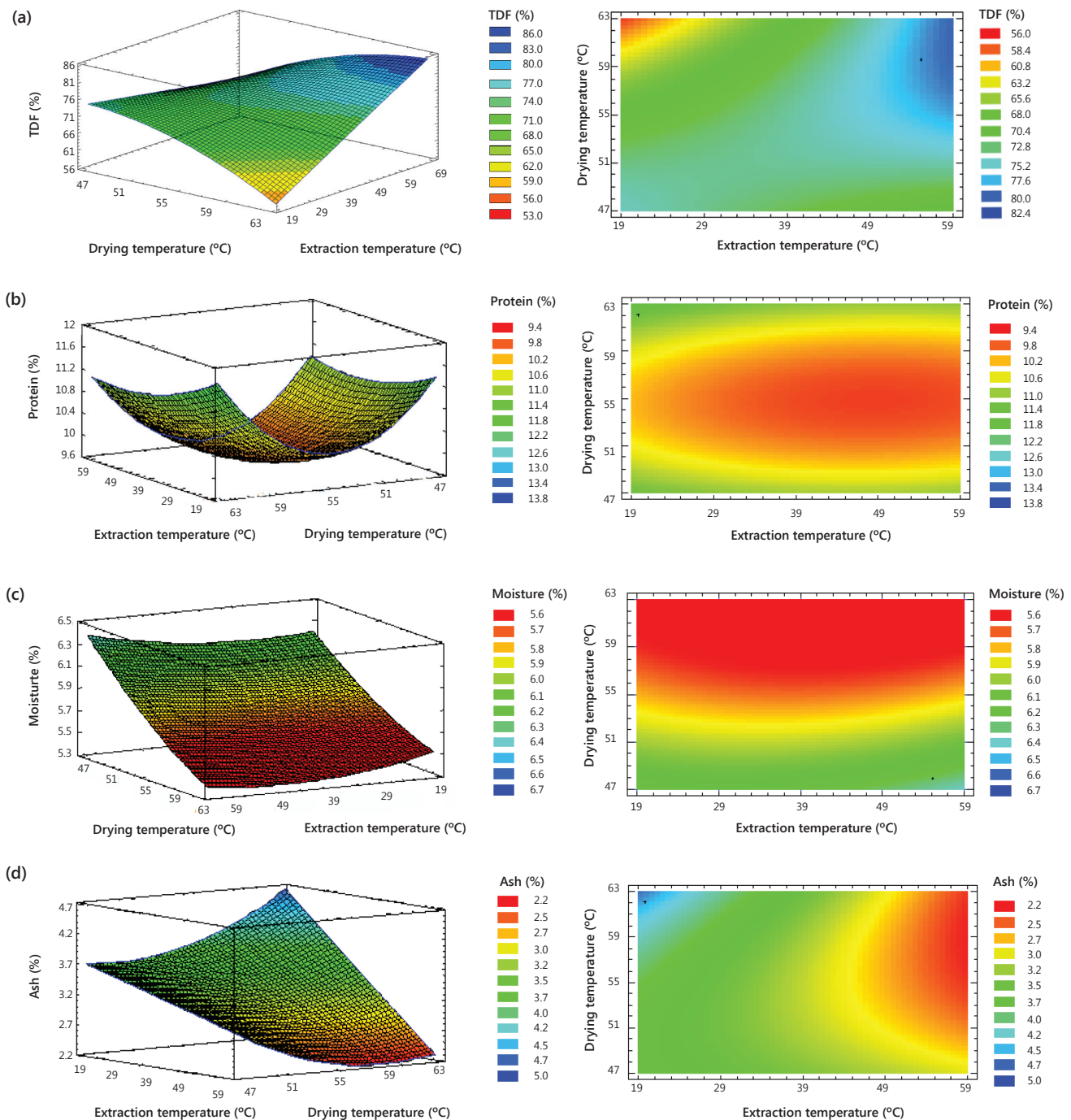


Figure 1. Response surfaces and contours estimated for the chemical composition of the asparagus byproduct dietary fiber as a function of extraction and drying temperatures. (a) Total dietary fiber, (b) Protein, (c) Moisture and (d) Ash.

agribusiness byproducts such as rice bran (16%) (Abdul-Hamid and Luan, 2000). The moisture ranged from 5.33% to 6.17%. The optimum temperatures for this parameter were 47.9 °C (extraction) and 55.1 °C

(drying) (Figure 1c). The ash content showed little variation among the treatments, which indicates the slight influence that the extraction and drying temperatures had on this component. Yet, the highest

Table 3. Analysis of variance for the chemical composition and physicochemical properties of the asparagus byproduct dietary fiber obtained at the different extraction and drying temperatures.

Source	TDF		Protein (%)		Moisture (%)		Ash (%)		pH		a_w	
	RC	P value	RC	P value	RC	P value	RC	P value	RC	P value	RC	P value
β_0	4.422	-	73.162	-	11.857	-	14.663	-	9.065	-	- 0.245	-
β_1 (Extraction)	- 2.242	0.036*	- 0.040	0.591	0.007	0.997	0.092	0.116	- 0.054	0.064	0.008	0.214
β_2 (Drying)	4.171	0.150	- 2.282	0.0850*	- 0.176	0.008*	- 0.470	0.784	- 0.082	0.574	0.020	0.003*
β_{11}	- 0.0001	0.983	0.0006	0.784	0.0002	0.522	0.0003	0.983	0.0002	0.131	- 0.0009	0.390
β_{12}	0.044	0.073	- 0.0004	0.956	- 0.0004	0.678	- 0.002	0.659	0.0004	0.362	- 0.0002	0.616
β_{22}	- 0.055	0.187	0.02099	0.213	0.001	0.551	0.005	0.634	0.0006	0.553	- 0.002	0.25
Lack of fit		0.169		0.468		0.136		0.506		0.608		0.103
Total (corr.)		199.3		6.556		0.085		1.175		0.063		0.017
R ²		0.5838		0.3130		0.7120		0.4768		0.7331		0.8543

RC: Regression coefficient; * Significance of $P \leq 0.05$.

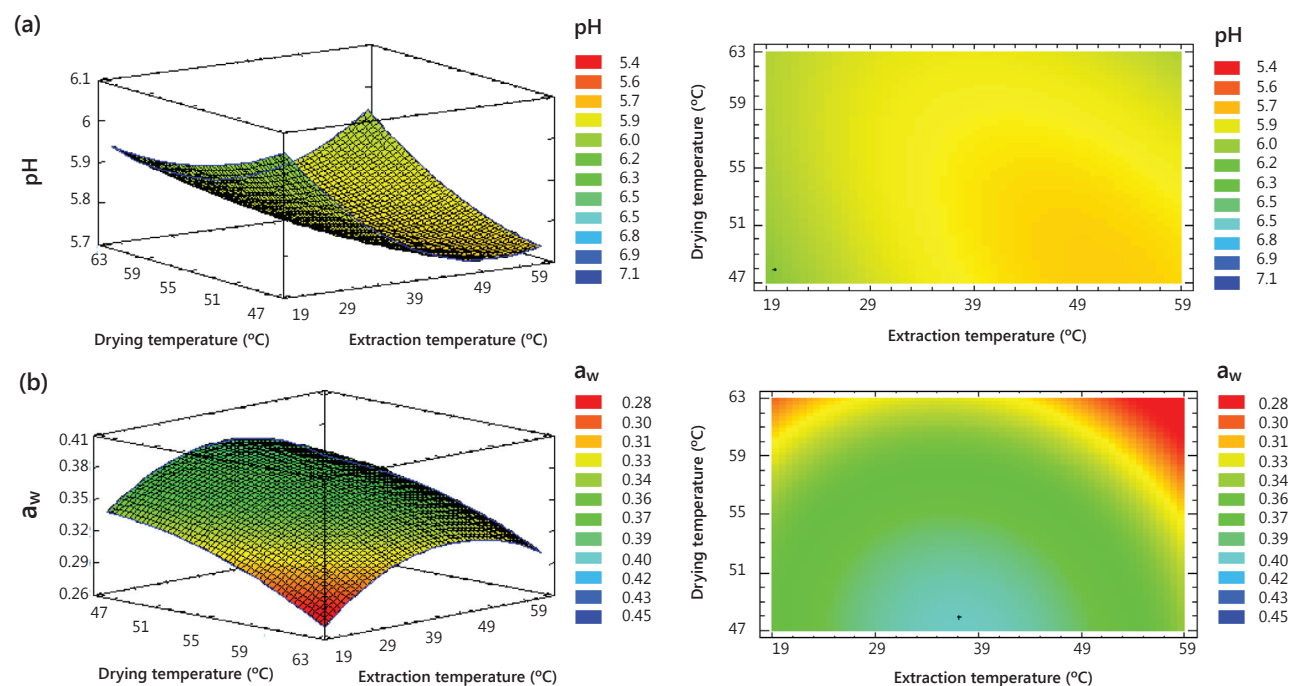


Figure 2. Response surfaces and contours estimated for the physicochemical properties of the asparagus byproduct dietary fiber as a function of the extraction and drying temperatures. (a) pH; (b) a_w .

ash content was obtained at the 19.8 °C and 62.7 °C extraction and drying temperatures, respectively (Figure 1d). The ANOVA (Table 3) only detected significant differences ($P \leq 0.05$) for the drying temperature, whereas neither the other variation sources nor their interactions or the LOF test showed any significance. These observations support the efficacy of the proposed model. The pH ranged from

5.71 to 6.05; the most remarkable contrast being that of the 19.8 °C (extraction) and 47.9 °C (drying) temperatures (Figure 2A). The slight influence of the extraction and drying temperatures on the pH is determining for the fermentation of fiber rich substrates, which is the case of the cell walls of the studied asparagus byproduct. In effect, this material contains elevated levels of cellulose and hemicellulose,

which are neutral sugar polysaccharides. The a_w was lower than 0.375 at the 37.0 °C (extraction) and 47.9 °C (drying) temperatures, being the most adequate ones (Figure 2B). If a_w decreases, micro-organisms with the ability to grow will also decrease. Each species of micro-organism (bacteria, yeast and mould) has its own minimum a_w value below which growth is no longer possible.

Functional properties. The results obtained for the WAI, WSI, OAI and SV are shown in Table 4. The extraction and drying temperatures had no significant effects on the functional properties or the LOF test (Table 5). WAI and SV are frequently used as water retention indicators, whereas WSI is related to soluble solid levels, which are, in turn, starch degradation and denitrification indicators (Dogan and Karwe, 2003). SV refers to the fiber's volume increasing capacity in the presence of an excess of water (Villarroel *et al.*, 2003).

The highest water absorption capacity was shown by the fiber extracted at 55.1 °C and dried at 47.9 °C (Figure 3A). From a technological and physiological standpoint, WAI is considered an important property (Fuentes *et al.*, 2009). High temperature drying is likely to alter the physicochemical properties of the product and, consequently, its functional properties too (Femenia *et al.*, 2000). Contrastingly, the extraction temperature is capable of modifying the structural characteristics of the fiber, which tends to facilitate water absorption (Figuerola *et al.*, 2005). The WAI and SV are related to the porous structure of the polysaccharide chain matrix, which bears the capability of attracting and retaining water through its hydrogen bonds. The WAI values found in the present study ranged between 9.92 and 14.32 g of water/g of asparagus fiber, which is close to the counts of other species, namely peach (fruit pulp fiber: 12.6 mL of water/g) (Grigelmo and Martin, 1999) and lemon (fiber in fruit juice: 11 mL of water/g) (Lario *et al.*, 2004). Lower values than these are usually found in cocoa shell (5 mL of water/g of fiber) (Lecumberri *et al.*, 2007). Based on the current results, the fiber obtained from the processing of the studied asparagus byproduct can be used in the formulation of new products as a texture and viscosity modifier and, collaterally, as a calorie reducer.

The WSI of the studied asparagus byproduct ranged between 10.99% and 15.73%; the lowest count being that obtained through treatment 3 (Table 4).

Additionally, the response surface model indicates that the region where the WSI was maximized corresponded to the 22.8 °C and 61.8 °C extraction and drying temperatures, respectively (Figure 3b). These WSI results broadly overcame the 4.38% to 7.0% range established for commercial corn flour, which is of common use in the human diet (Bressani *et al.*, 2001). The WSI was influenced by the drying conditions, which may contribute to modifying texture properties, preventing syneresis (the separation of the liquid component of a gel due to contraction) and improving shelf life (Mohamed *et al.*, 2011). Such results may have been determined by structural modifications taking place during fiber grinding, when particle size is reduced, thus exposing a larger surface area (and hence, more polar groups with water retaining sites) to the surrounding water (Rosell *et al.*, 2009).

OAI is the maximum amount of oil (in grams) that can be retained by 1 g of dry matter immersed in an excess of oil and under the action of a force (centrifugation); it represents the capacity of the fiber to absorb oil (Tamayo and Bermúdez, 1998). In the present case, the OAI ranged between 3.58 and 5.12 g of oil/g of asparagus by-product fiber (Table 4), while its optimum count regarding the extraction and drying temperatures corresponded to 41.6 °C and 51.9 °C, respectively (Figure 3c). The current OAI values exceed those recorded for apple pomace and orange peel (0.6 - 1.8 mL g⁻¹) by Figuerola *et al.* (2005), as well as unripe plantain (2 mL g⁻¹) (Rodríguez *et al.*, 2006), pea (1.06 -1.17 g g⁻¹) and pigeon pea (*Cajanus cajan* L.) (0.96 - 0.98 g g⁻¹) flours (Kaur *et al.*, 2007). It is worthwhile noting that the highest OAI value observed in the current study was close to 6.0 mL g⁻¹, which, measured in carrot pulp, is the largest record for this parameter to-date (Garau *et al.*, 2006). These results make the studied asparagus byproduct fiber a useful ingredient to be added to foods in which emulsifying properties are needed for flavor retention, palatability improvement and useful life increases. This is particularly true in the case of baked products and cut meats, where it is important to absorb large oil amounts (Alarcón *et al.*, 2013).

The SV of the studied asparagus by-product ranged from 1.0 mL g⁻¹ to 6.0 mL g⁻¹ (Table 4), the optimum record corresponded to the 55.1°C and 47.9°C extraction and drying temperatures, respectively (Figure 3d). Treatment 9 stood out for contributing

the highest SV value, corresponding to the 50 °C extraction and drying temperatures, corresponding with those employed to treat orange peel in other studies that obtained similar results (Hincapié *et al.*, 2010). Several of these SV records are within the range of many DF sources of commercial use (Valencia and

Román, 2006). Finally, the differences found in the values of this parameter may explain the chemical composition of the carbohydrates found in the byproduct fiber and the temperature effects during the sample extraction and dehydration (Nawirska and Kwasniewska, 2005; Rosell *et al.*, 2009).

Table 4. Functional properties of dietary fiber obtained from the asparagus byproduct treated with different extraction and drying temperatures.

Treatment	WAI (g/g)	WSI (%)	OAI (g/g)	SV (mL/g)
1	13.68	11.05	4.95	1.0
2	12.10	11.89	4.98	4.6
3	11.29	10.99	5.12	3.0
4	12.51	12.49	4.93	3.3
5	11.11	13.19	4.55	2.3
6	12.18	12.43	4.38	2.0
7	11.90	11.41	3.78	2.6
8	11.05	15.73	3.91	1.0
9	14.31	13.19	4.38	6.0
10	11.17	11.04	4.36	2.0
11	9.92	12.45	4.74	1.3
12	12.11	11.09	4.95	3.6

All values averages of triplicate tests.

Table 5. Analysis of variance for the functional properties of the asparagus byproduct dietary fiber treated with different extraction and drying temperatures.

Source	WAI		WSI		OAI		SV	
	RC	P value	RC	P value	RC	P value	CR	P value
β_0	- 87.293	-	47.489	-	0.761	-	21.423	-
β_1 (Extraction temperature)	0.877	0.318	0.924	0.103	0.012	0.074*	0.568	0.236
β_2 (Drying temperature)	3.175	0.055*	- 1.922	0.638	0.148	0.301	-0.859	0.063*
β_{11}	-0.002	0.299	0.0007	0.738	- 0.001	0.099	0.0009	0.784
β_{12}	-0.012	0.124	- 0.018	0.061	0.002	0.356	- 0.010	0.358
β_{22}	-0.026	0.108	0.024	0.154	0.002	0.627	0.009	0.670
Lack of fit		0.315		0.153		0.241		0.710
Pure error		1.596		1.945		0.241		4.555
Total (corr.)		15.674		20.722		2.083		24.919
R ²		0.7115		0.5554		0.6004		0.7263

CR: Regression coefficient; * Significance of $P \leq 0.05$.

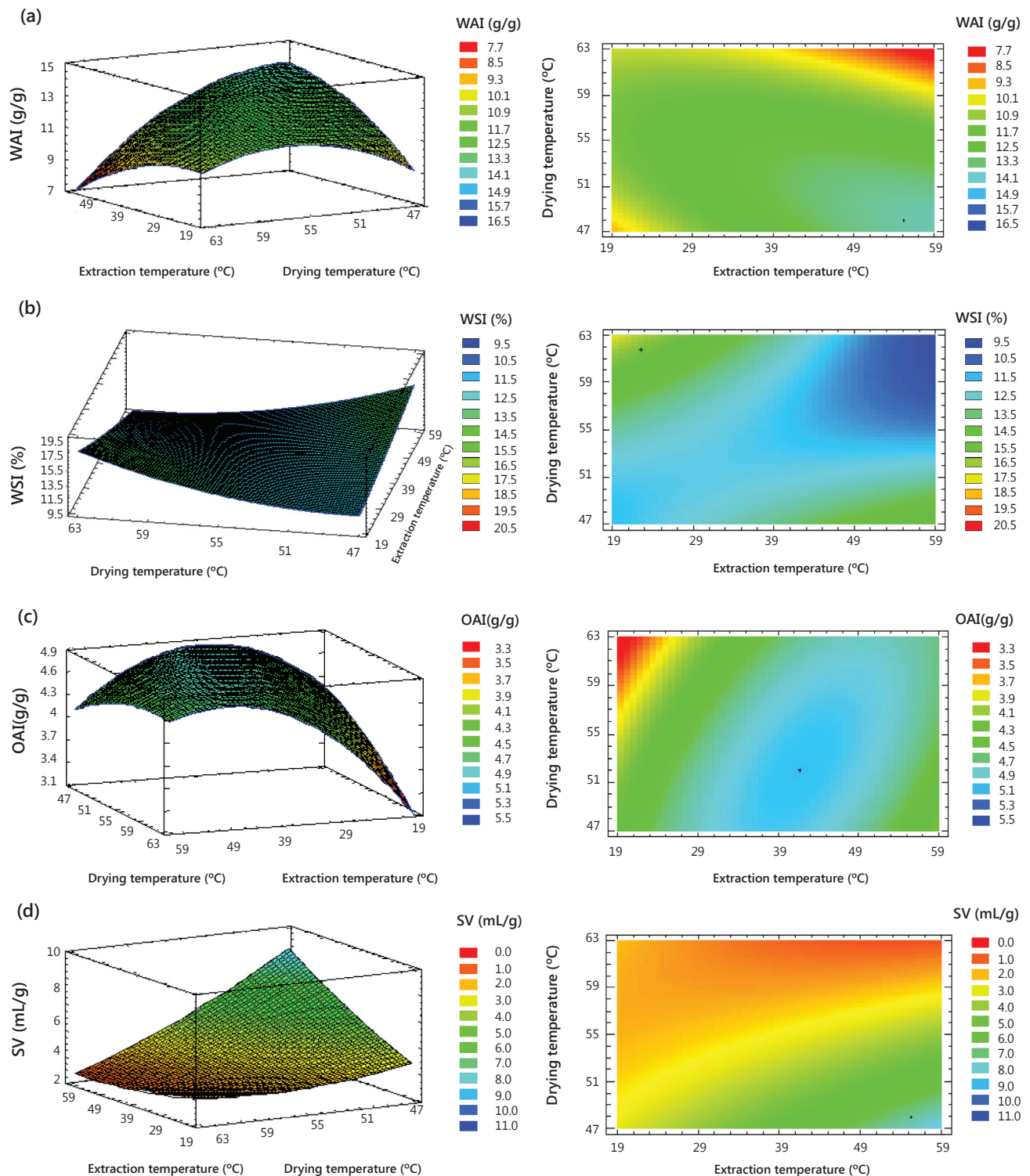


Figure 3. Response surfaces and contours estimated for the functional properties of the asparagus byproduct dietary fiber treated with different extraction and drying temperatures. (a) WAI, (b) WSI, (c) OAI and (d) SV.

CONCLUSIONS

The TDF content of the studied asparagus byproduct (60.7 – 79.0%) exhibited important functional properties – namely WAI (9.92 to 14.31 g/g), WSI (10.99 to 15.73%), OAI (3.78 to 5.12 g/g) and SV (1.3 to 6.0 mL/g) – all of which are useful in the food processing industry.

The chemical, physicochemical and functional properties of the studied asparagus byproduct make it a potential DF source, to be taken into account in the formulation of food products that are capable of satisfying the nutritional needs of the population and reducing the risk factor associated with non-traditional chronic diseases. Furthermore, this innovation may possibly contribute to a more environmentally-friendly asparagus agribusiness.

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