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Optimization of Dietary Fiber Extraction from Quince Peel †

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Abstract: The agri-food industry generates tons of waste rich in dietary fiber, a nutrient that can be recovered to be reused in the development of fiber-enriched foods and beverages. This could be a strategy to achieve resource-use efficiency and to promote adequate intakes of this nutrient, since a large part of the world population does not get the recommended daily amount. In this sense, this work was carried out to optimize the extraction of dietary fiber from quince (*Cydonia oblonga* Mill.) peel, using the response surface methodology. A 20-run experimental design was implemented, combining the factors time, temperature, and ethanol percentage at five levels. The yield of fibrous residue (FR) and its dietary fiber content and color parameters were used as dependent variables. The developed predictive models were statistically validated and used to determine optimal extraction conditions. The process was significantly affected by temperature and ethanol percentage, and the highest dietary fiber content (67% of FR) was obtained using 36% ethanol at 92 °C. Overall, these results showed that *C. oblonga* fruit peel could be upcycled into dietary fiber-rich food ingredients.

Keywords: food waste; extraction optimization; dietary fiber; response surface methodology

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1. Introduction

The agri-food sector is under pressure to move towards more sustainable production systems that are capable of providing high-quality food to a growing world population facing climate change and resource scarcity. This sector generates million tons of plant waste and by-products rich in dietary fiber, a non-starch polysaccharide that could be recovered and recycled inside the food chain as a food ingredient [1,2]. This could be a strategy to achieve circularity and resource efficiency and to promote adequate intakes of dietary fiber, since a considerable part of the world population does not get the recommended daily amount (25 g/day) [3]. Dietary fiber is a "nutrient of public health concern" that plays an important role in protecting against cardiovascular disease, type 2 diabetes, and certain cancers and in improving gastrointestinal health and body weight control [4,5]. Nowadays, new food products enriched in dietary fiber have been developed to meet the growing demands of health-conscious consumers [6,7]. Food products added with certain fibers can also have improved structural properties. Therefore, there is a need to identify sustainable raw materials rich in dietary fiber, mainly agri-food by-products.

Quince (*Cydonia oblonga* Mill.) is an astringent fruit used to produce sweet foods and drinks such as jam, marmalade, and liquor. Although its peel is usually discarded during the manufacture of these products, some studies have highlighted the potential of this by-product as a source of compounds with nutritional and technological value such as dietary fiber (including pectins) [8–10]. Thus, this work was conducted to optimize the recovery of dietary fiber from the *C. oblonga* fruit peel by dynamic maceration, using a three-factor experimental design coupled with response surface methodology (RSM).

2. Material and Methods

Cydonia oblonga fruit peel kindly provided by producers in Northeastern Portugal was freeze-dried, ground into a 20-mesh particle size powder sample, and then subjected to solid–liquid extractions by dynamic maceration. Five levels of time (*t*, 1–119 min), temperature (*T*, 26–94 °C), and ethanol percentage (*E*, 0–100%, *v*/*v*) were selected based on previous studies [11,12] and combined according to the 20-run central composite rotatable design shown in Table 1. The extractions were made in closed flasks, using a solid/liquid ratio of 60 g/L in all runs. Each extractive mixture was then centrifuged, and the fibrous residue (FR) was collected and oven-dried until constant weight for further analysis.

Table 1. Yield, dietary fiber content, and color of the FR samples obtained from *C. oblonga* fruit peel by dynamic maceration under the 20 runs of the experimental design.

Run	Experimental Domain			Experimental Results			
	***t* (min)**	***T* (°C)**	***E* (%)**	**FR (%, *w*/*w*)**	**DF (% FR)**	***L****	**RGB ***
1	−1 (25)	−1 (40)	−1 (20)	43.66	57.84	59.66	
2	1 (95)	−1 (40)	−1 (20)	43.08	56.78	56.75	
3	−1 (25)	1 (80)	−1 (20)	42.11	64.44	37.24	
4	1 (95)	1 (80)	−1 (20)	46.76	62.65	35.62	
5	−1 (25)	−1 (40)	1 (80)	51.90	57.27	55.88	
6	1 (95)	−1 (40)	1 (80)	51.59	58.66	56.02	
7	−1 (25)	1 (80)	1 (80)	49.41	57.73	59.51	
8	1 (95)	1 (80)	1 (80)	52.70	57.53	58.60	
9	1 (−1.68)	0 (60)	0 (50)	51.64	57.21	58.35	
10	119 (1.68)	0 (60)	0 (50)	50.25	58.64	56.80	
11	0 (60)	−1.68 (26)	0 (50)	39.82	59.83	51.76	
12	0 (60)	1.68 (94)	0 (50)	40.81	65.55	34.09	
13	0 (60)	0 (60)	−1.68 (0)	49.18	55.33	55.90	
14	0 (60)	0 (60)	1.68 (100)	65.53	52.46	70.62	
15	0 (60)	0 (60)	0 (50)	50.31	59.95	54.21	
16	0 (60)	0 (60)	0 (50)	48.18	58.11	56.62	
17	0 (60)	0 (60)	0 (50)	46.68	59.35	56.42	
18	0 (60)	0 (60)	0 (50)	48.27	57.86	57.30	
19	0 (60)	0 (60)	0 (50)	50.54	57.94	58.92	
20	0 (60)	0 (60)	0 (50)	47.97	60.48	58.33	

Process factors: *t*—time; *T*—temperature; *E*—ethanol percentage; FR—fibrous residue; DF—dietary fiber; *L**—lightness; RGB—red/green/blue color model. * Color representation of each FR sample.

The FR yield (%, *w*/*w*) was determined by a gravimetric method [13]. The dietary fiber (DF) content of the FR samples was determined by an enzymatic–gravimetric method (AOAC 985.29) involving gelatinization with heat-stable α-amylase (pH 6.0, 15 min at 95 °C) and digestion with protease (pH 7.5, 30 min at 60 °C) and amyloglucosidase (pH 4.5, 30 min at 60 °C) [14]. The dietary fiber content (% FR) was determined after discounting the measured protein (AOAC 920.152) and ash (AOAC 940.26) contents [14]. The CIELAB color parameters (*L**, lightness; *a**, redness; and *b**, yellowness) of the FR samples were measured with a portable CR-400 colorimeter (Konica Minolta Sensing Inc., Osaka, Japan) [13] and then converted to RGB (red, green, and blue) to create a color model for visual perception.

For optimization of the dynamic maceration process by RSM, the FR yield, dietary fiber content, and lightness were used as dependent variables; the three factors were set within the experimental domain, and the "maximize" option was selected for each dependent variable. Design-Expert software 11 (Stat-Ease, Inc., Minneapolis, MN, USA) was used to estimate the model coefficients and assess their significance at a 95% confidence level, as well as to generate the response surface plots.

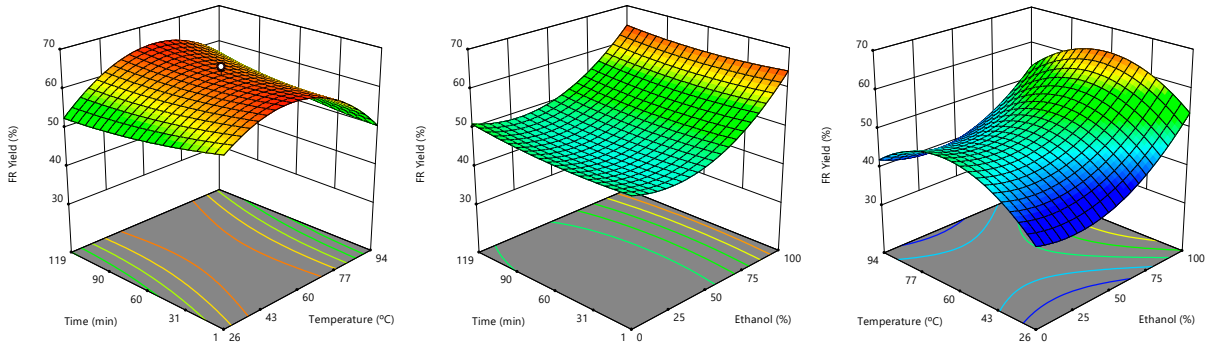
3. Results

The FR yields and their dietary fiber content and color parameters obtained with the 20 runs of the experimental design are shown in Table 1. The FR yield ranged from 39.82 to 65.53% (*w/w*) with the runs 11 and 14, respectively, and these FR samples were constituted by 52.46 to 65.55% of dietary fiber. Runs 12 (involving the highest temperature) and 14 (involving the highest ethanol percentage) resulted in the highest and lowest levels of dietary fiber and also the darkest and lightest FR samples, respectively. The redness (7.2 ± 0.7) and yellowness (20 ± 3) of the FR samples did not vary much with the 20 runs (data not shown).

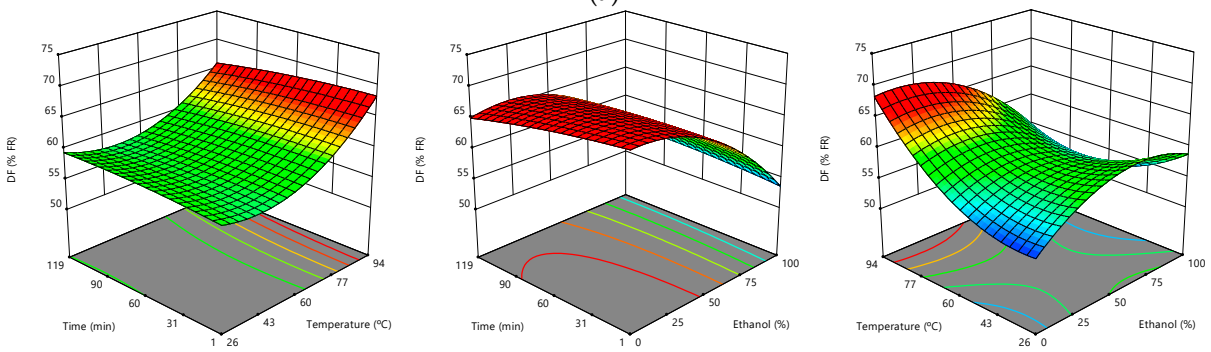
Predictive model equations were developed to translate the effects of the three factors on the dependent variables FR yield, dietary fiber content, and lightness, considering the significant parameters ($p < 0.05$) and those necessary for the hierarchy. These models were statistically significant ($p < 0.001$), had a non-significant lack of fit ($p > 0.345$), and R^2 and adjusted R^2 values greater than 0.909 and 0.877, respectively. Each parametric value of the equation models translated a certain change in the outcome as a function of its magnitude. The extraction trends for each dependent variable are represented on the response surface plots in Figure 1. The unplotted factor on each 3D plot was kept at its optimum.

Equation (1) shows that the FR yield was highly influenced by the ethanol percentage, as this factor induced marked linear and quadratic effects and interacted with time. The 3D graphs in Figure 1a show that the higher yield was promoted by higher ethanol percentages. In turn, temperature mainly induced a negative quadratic effect. Thus, the extraction of *C. oblonga* fruit peel powder for 69 min with 100% ethanol at 61 °C led to the highest FR yield of $64 \pm 2\%$ (*w/w*).

$$Y_{FR} = 49.05 + 0.35t + 0.18T + 4.21E - 3.40T^2 + 3.63E^2 + 1.10tE \quad (1)$$

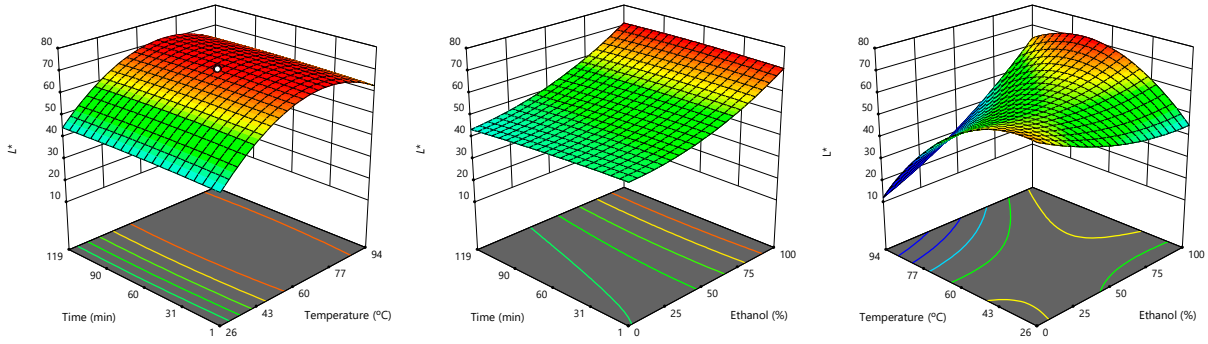


(**a**)



(**b**)

Figure 1. *Cont.*



(**c**)

Figure 1. Response surface plots illustrating the effects of the three factors on (**a**) yield, (**b**) dietary fiber (DF) content, and (**c**) lightness of the FR samples obtained from *C. oblonga* fruit peel by dynamic maceration. In each representation, the unplotted factor was kept constant at its optimum value.

Equation (2) translates extraction trends for dietary fiber. The recovery of these *C. oblonga* fruit peel constituents was affected by the positive effects of temperature and negative effects of ethanol percentage. Furthermore, these factors interacted negatively. Thus, as shown in Figure 1b, the FR samples with higher levels of dietary fiber were obtained using higher temperatures and lower ethanol percentages. The non-significant effects ($p > 0.05$) of processing time are also noted on the response surfaces. According to the predictive model, FR samples with 67 ± 1% of dietary fiber can be obtained by extraction at 92 °C with 35% ethanol. For time, there was no specific optimal value to maximize the process.

$$Y_{DF} = 58.79 + 1.57T - 1.12E + 1.55T^2 - 1.56E^2 - 1.64TE \quad (2)$$

When dietary fiber is added to foods for fortification purposes, the visual appearance of the developed food products should not be negatively affected by the fiber color of this nutrient. Therefore, the impact of the extractions on the color of the FR samples was measured, and lightness was the parameter that varied the most. As translated by Equation (3), the parametric values obtained for this dependent variable had an opposite sign to those of Equation (2) for dietary fiber, i.e., the parametric values of temperature were negative, and those of ethanol percentage were positive, as well as that of the interaction effect. Hence, while the higher-yielding FR samples tended to be lighter, the higher-fiber FC samples were darker.

$$Y_{L^*} = 56.86 - 4.91T + 4.80E - 5.37T^2 + 1.82E^2 + 6.22TE \quad (3)$$

The results of this study are consistent with those of Afifi et al. [15], who obtained an FR sample with 79.4% dietary fiber from date seeds and found a decrease in fiber contents with an increasing ethanol percentage. In turn, Al-Farsi and Lee [16] achieved 83.5% fiber in a date seed FR sample with an aqueous extraction. These authors highlighted the potential of date seeds as an inexpensive source of dietary fiber for application as a food ingredient.

4. Conclusions

Dietary fiber-rich FR samples were obtained from the fruit peel of *C. oblonga* through an optimized dynamic maceration process. Lower-yielding FR samples tended to contain higher dietary fiber percentages and be darker. The optimized maceration involved 92 °C and 36% ethanol as solvent and yielded 67% of dietary fiber, while the time factor was not significant. These results are the first step towards a large-scale implementation process, and the natural ingredient resulting from the valorization of this underutilized raw material can be used in the fortification of foods and drinks and in nutraceutical formulations.

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