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Effect of tiger-nut (*Cyperus esculentus*) milk by-product on the surface and diffusional properties of a wheat-based matrix

Samuel Verdú, Cecibel Alava, José M. Barat, Raúl Grau

Departamento de Tecnología de Alimentos. Universidad Politècnica de València, Spain.

*Author for correspondence: Samuel Verdú

Address: Edificio 8G - Acceso F – Planta 0

Ciudad Politécnica de la Innovación

Universidad Politécnica de Valencia

Camino de Vera, s/n

46022 VALENCIA – SPAIN

E-mail: saveram@upvnet.upv.es

Phone: +34 646264839

Abstract

The food processing industry generates huge volumes of waste and by-products which still contain valuable compounds. Tiger-nut milk production generates large amounts of a by-product with a high insoluble fibre content, which is interesting as a bioactive component from a nutritional viewpoint. This by-product is formed by two different tissues in composition, particle size and colour terms, so two different flours were obtained from them. Both flours were included in a wheat-based matrix at different substitution levels: 5%, 10% and 20% (d.b). The surface tension of matrices, and the wettability and diffusion of water and oil, were studied. The results showed the matrix's reduced capacity to interact with solvents, principally from the 10% substitution level, with diminished surface tension, and a longer time was needed for both water and oil to wet and diffuse.

Keywords: by-product, tiger-nut, surface tension, diffusional properties, wheat matrix

1. Introduction

Organisations such as the FDA and EFSA inform that many interesting and valuable compounds are contained in food processing waste and by-products. This group of compounds is formed from a mix of substances of different natures; e.g. organic acids, aromas, colorants, antimicrobial compounds, dietetic fibre, essential oils, etc. (Tuberoso, Rosa, Montoro, Fenu, & Pizza, 2016). Nowadays industry is considering improving its processes to recover these products, to turn them into an income source, and to also recirculate compounds of healthy and technological interest in other products. These by-products can be converted into profitable products as raw materials for secondary processes (intermediate compounds), operating supplies, or the ingredients of new products (Sánchez-Zapata et al., 2009).

Many are interesting because their proximal composition contains a large amount of dietetic fibre. Such interest is based on their benefits as a bioactive component from a nutritional point of view, where the relation of vegetable fibre and the mitigation and prevention of health diseases, e.g. type 2 diabetes mellitus, cardiovascular disease and colon cancer, have been clearly evidenced (J. Zhang, Cui, Yin, Sun, & Li, 2013). The overall composition of dietetic fibre is based on cellulose, non-cellulosic polysaccharides such as hemicellulose, pectins, gums, mucilages, and non-carbohydrate components like lignin (Dhingra, Michael, Rajput, & Patil, 2012), all of which are resistant to enzymatic digestion. Their incorporation into food products with small amounts of fibre natively is an extended practice in industry (bakery, drinks, beverages, dairy and meat products). However, changes in their physicochemical properties related to consistency, texture, rheological behaviour and sensory characteristics could appear (Bortnowska et al., 2016).

68 The by-product obtained from tiger-nut (*Cyperus esculentus*) milk production is an
69 excellent source of insoluble fibre, which is traditionally destined to organic mass for
70 combustion, compost and feed production. This false nut is the tuber of a Corsa sedge
71 genus perennial herb member of the grass family Cyperaceae (Ayeh-Kumi et al., 2014)
72 from which, after several cycles of grinding and pressing, most of its aqueous fraction
73 and soluble components are removed to give a fibrous gross flour classified as waste.
74 The interest in developing applications for this by-product also lies in the fact that vast
75 volumes of this waste are generated in several geographic zones of Spain, one of the
76 world's main tiger-nut milk producers. This agro-industry annually generates close to 5
77 million euros.

78 In this way, several studies have been reported about using tiger nut for enriching some
79 food products and for recovering other interesting compounds, such as oils or
80 flavonoids (Ezeh, Gordon, & Niranjan, 2016; Jing et al., 2016). However, the by-
81 product obtained from the tiger-nut milk process has been less studied as an ingredient
82 for developing products. Data about the impact of this by-product on some product
83 properties, such as pork sausages and burgers, and gluten-free breads, have been
84 reported in several studies (Sánchez-Zapata et al., 2010; Sánchez-Zapata, Zunino,
85 Pérez-Alvarez, & Fernández-López, 2013), but very little information about its effect on
86 some food matrices is available. Given its high fibre content, this by-product is quite
87 susceptible to being added to grain-based food, such as bread, pasta, breakfast cereals,
88 crisps, biscuits or crackers, whose wholemeal versions occupy an important volume of
89 total production in the cereal processing industry. Thus including new fibre sources in
90 cereal products and studying their repercussion on matrix physicochemical properties
91 represent a wide area of research on their impact on process chains, properties,
92 functionality, and then on end product quality. One basic aspect that affects product

quality is product properties as regards the interaction with liquids since most are destined to be accompanied by some liquid food (sauces, fat emulsions, milk, jam, etc.). These interactions could determine their optimum processing, storage conditions, stability of in contact-ingredients or adequate consumer use conditions. The characterisation of the impact caused by a new ingredient on these properties could be made by studying parameters such as the material's surface tension, permeability, wettability, diffusion assays, sorption isotherms, etc. The aim of this work was to study the effect of the tiger-nut milk by-product on the surface tension and solvent (water and oil) diffusional properties of a wheat-based matrix by an approach that simulates the basic processes for which a wheat-based food product could be destined.

2. Material and methods

2.1 Tiger-nut milk by-product conditioning and used flours

The tiger-nut milk by-product was obtained from a local tiger-nut milk manufacturing plant, presented as a wet fibrous flour. Two kinds of milled fibrous tissues can be differenced in this by-product from a macroscopic point of view. The first has a larger particle size (>800 μm) and is brown, like any grain bran. It is provided by the periderm (skin) and cortex of the tuber (Figure 1:A). It is a typical woody fraction characterised by high lignin content, among other insoluble polymers (Donaldson, 2001). The second has a smaller particle size (<800 μm) and is white (Figure 1:B), and is provided by internal tuber tissues, such as the perimedulla and medulla. It is a cottony material whose composition is based on insoluble carbohydrates, like cellulose, hemicellulose and non-digestible starches (Habibi, Mahrouz, & Vignon, 2009). As both are perfectly mixed once the tiger-nut milk process finishes, separation was done using a 180-μm sieve to isolate the white fraction. Thus two types of by-product flour were employed:

the whole by-product (F1), whose moisture was 56±0.4% and white fraction (F2), whose moisture was 47.2±0.4% of water. Both flours were dried to 14% of moisture (w.b) to be remilled in a stainless steel grinder (Retsch GmbH, ZM 200, Haan, Germany). Finally, the proximate composition of F1 was 1.9±0.7% of proteins, 13.3±0.1% of fat, 14.1±0.4% of water, 1.86±0.1 of ash and 68.2±0.4% of total dietary fibre (w.b). It was 1.5±0.7% of proteins, 11.8±0.2% of fat, 14.2±0.4% of water, 1.5±0.1 of ash and 71.1±0.3% of total dietary fibre (w.b) for F2.

The commercial wheat flour we used was obtained from a local producer (Molí del Picó-Harinas Segura S.L. Valencia, Spain). Its proximate composition was 14.7±0.6% of proteins, 1.1±0.03% of fat, 14.5±0.5% of water, and 0.32±0.1 of ash (w.b). The alveographic parameters were also facilitated by the company, which were P = 94±2 (maximum pressure (mm)), L = 128±5 (extensibility (mm)), W = 392±11 (strength (J-4)) and 0.73 of P/L.

The particle size of flours was also measured 6 times by laser scattering in a Mastersizer 2000 (Malvern, Instruments, UK), equipped with a Scirocco dry powder unit. The particle size of wheat flour was d (0.1) = 25.5±1.1, d (0.5) = 92.0±0.6, d (0.9) = 180.6±0.8 and D [4, 3] = 99.4±1.2. For F1, particle size was d (0.1) = 24.3±0.9, d (0.5) =182.7±7.1, d (0.9) = 663.6±20.6 and D [4, 3] =271.1±8.1 and was d (0.1) = 31.9±0.7, d (0.5) =197.4±7.7, d (0.9) = 516.5±32.4 and D [4, 3] =246.7±14.5 for F2. Particle sizes are expressed as the maximum size (μm) at 10% *(d (0.1))*, 50% (*d (0.5))* and 90% (*d (0.9))* as their averages (D [4, 3]) of the total volume of analysed particles.

2.2 Wheat-based matrices production

141 The selected matrix was close to cereal-derived foods destined to come into contact
142 with a liquid or spreadable food product (such as sauces or creams), which has a low
143 moisture and vitreous consistence. Several examples are crackers, toasted bread,
144 biscuits, tortillas, etc. The wheat-based matrices were produced by mixing wheat flour
145 with the tiger-nut milk by-product flours. As previously explained, two different by-
146 product flours were used for substituting wheat flour. Three substitution levels were
147 employed: 5%, 10% and 20% w/w in dry basis (d.b). The percentages of substituted
148 mass were calculated on the dry matter fraction of the used wheat flour. This was done
149 to maintain the g of water/ g of solutes ratio constant between formulas (0.58 g of
150 water/g of solutes). Finally, seven different formulas were prepared. The formula of the
151 control sample was 65% of wheat flour, 27.7% of water, 5.7% of oil (maximum acidity
152 0.2º Koipesol Semillas, S.L., Spain), 1.4% of salt (refined marine salt ≥ 97% NaCl
153 Salinera Española S.A., Spain). These percentages were kept constant for all the
154 substituted formulas, and the amount of by-product flour varied depending on each
155 case. The procedure was as follows:

156 1. Liquid components (water and oil) and salt were placed into a food mixer
157 (Thermomix® TM31, Vorwerk, Germany) and mixed to obtain a homogeneous
158 solution (1.5 minutes/ 50 rpm).
159 2. Pre-homogenised flour was added to the food mixer and mixed (4 minutes/550
160 rpm) with random turns of the mixer helix in both directions to obtain
161 homogeneous dough.
162 3. Dough was left to rest in a sealed bowl for 20 min at room temperature (20ºC)
163 4. Samples were shaped by separating balls of 10 g of dough, which were placed
164 inside a manual laminator (IMPERIA SM/220, FIMAR, Italy) to obtain sheets
165 of 2 mm thickness.

5. Baking was done in an oven (530x450x340, grill power 1200W, internal volume 32L, Rotisserie, DeLonghi, Italy) at 160ºC for 30 min
6. All the samples rested for 10 minutes at room temperature and were weighed to determine mass loss during the process based on Equation 1:

$$\Delta M_B = \frac{m_f - m_0}{m_0} \cdot 100 \quad (1)$$

where ΔM_b is the mass increment in % , m_f is mass post-baking and m_o is the initial mass before baking.

2.3 Surface tension of wheat-based matrices calculation

This parameter was determined according to the Zisman-plot method. It is based on the experimental finding that when a liquid spreads completely over an analysed surface, its surface tension is lower than or equal to that of the surface on which it spreads. It is interpreted theoretically as the surface tension of the liquid needed to completely wet the solid (contact angle between the solid and liquid is zero). The method was based on measuring contact angle θ, in degrees, between the wheat matrix surface and drops of probe liquids with different surface tensions γ_l. The cosines of each contact angle of the probe liquid were plotted against their respective surface tension γ_l values in mN/m. Then the model was extrapolated to cos=1 and the surface tension value for this point was taken as the surface tension of solid γ_s. This procedure was described by Zisman (Han et al., 2005), and is based on the relationship between the cosines of several liquids regarding a given solid material surface, and their surface tension, following function number 2:

189 $$cos\theta = a - b\gamma l = 1 + \beta(\gamma s - \gamma l) \quad (2)$$

190

191 where a, b, and β are constants. The three probe liquids were glycerol (Sigma Chemicals
192 Co., St. Louis, MO, USA), poliethyleneglycol-200 (Sigma Chemicals Co., St. Louis,
193 MO, USA) and dipropilen glycol (Sigma Chemicals Co., St. Louis, MO, USA). They
194 had a γ_l of 63.1, 45.5 and 33.9 mN/m, respectively. Drops of 3 µL were deposited onto
195 the wheat-based matrix with a microsyringe under room conditions (20ºC/50% R.H).
196 Contact solid-liquid angles were measured in thirty drops of each liquid for each matrix
197 formula. Drop images were captured with a camera (Webcam Logitech C920 HD Pro)
198 and a digital goniometer based on the ImageJ image analyser software, used to obtain
199 the dataset.

200

201 **2.4 Interaction between the matrix and model solvent**

202 **2.4.1 Wettability of model solvents**

203 The effect of the tiger-nut milk by-product on the surface wettability of the wheat-based
204 matrix was also studied throughout the contact angle θ generated between solvent drops
205 and the solid surface. In this case, the solvents that represented the base of most liquid
206 food products, emulsions and sauces were tested. Those model solvents were water and
207 a vegetable oil (sunflower), which could potentially come into contact with a multitude
208 of wheat-based matrix foods. Thirty drops of 3µL were deposited onto different zones
209 of the wheat-based matrix and their angle data were obtained in the same way as
210 described in Section 2.2. The evolution of drop angles over 25 seconds (divided into 5-
211 second intervals) was recorded to study the effect of time (Y. Zhang, Thompson, & Liu,
212 2011).

2.4.2 Solvent diffusion assay

The diffusion of the model solvents, water and oil was tested by mass uptake during solid immersion. This assay was carried out by immersing full sheets of the solid in water or oil, whose surface area (4±1.5 cm^2) and mass were known. This study was done to simulate a real situation of a wheat matrix food that comes into contact with any liquid food, such as water- or oil-based sauces and dressings. An area of pieces was obtained while they were scanned with Scanner Canon Lide 120 and the ImageJ image analyser software. Pieces were fixed by fine-tip tweezers to a universal support and were completely immersed in a glass with the solvent, which was placed onto a precision balance (FV120, Anapesing, Spain). Mass uptake was recorded in 10-second intervals for 40 seconds. Diffusion was calculated as flux *J* using Equation 3:

$$J = \frac{g}{(A \cdot 2) \cdot s} \quad (3)$$

where g is the mass updated at any time, A is the area of the piece in cm^2 duplicate because of the two contact sides of the piece, and s is the time in seconds. Thirty replicates were used for each case.

2.6 Statistical procedure

The results were studied by a one-way variance (ANOVA) study, and by multifactor when it was necessary to study the main effects and interactions on the evolution of the parameters. In the cases in which the effect was significant (P-value < 0.05), the average was compared by Fisher's least significant difference (LSD).

3. Results and Discussion

3.1 Mass loss during the baking process

Mass loss during the baking process was evaluated to know the final moisture Xw_f of the samples (see the results in Table 1). Presence of the by-product generated a slight and non-significant increase in mass loss during the baking process, independently of the substitution level and type of substitutive flour (*A* or *B*). The control formula presented minor mass loss compared to whatever the substituted formula was, and no differences were found among formulae. The results coincided with those provided by Sánchez-Zapata et al., (2009), who reported that the water retention capacity of this by-product was similar to that of wheat flour.

3.2 Surface tension of the wheat-based matrix

Figure 2 shows the Zisman-plot, where the models generated between cos θ *vs.* γ_l from the probe liquids are represented. Figure 2A includes the model for the control formula (black dots), whose linear fit is represented as a visual example to show the intersection point with the line of cos = 1. The rest of the formulae are not represented graphically, but the result for γ_s and correlation coefficients R^2 are included in Table 1. The R^2 of the linear regressions obtained values that came quite close to 1 in all cases, so the cos θ *vs.* γ_l relationship was successfully evidenced. However, the cos θ values of the probe liquids presented significant differences when the percentage of wheat substitution was taken into account. Both by-product flours led to reduced surface tension γ_s of the wheat matrix (Table 1), but F2 was that with the most accentuated effect. Increments between γ_s of the pure wheat formula and the substituted ones were calculated in % to make the observation of changes easier. Table 1 shows increments $\Delta\gamma_s$, where a progressive

reduction of γ_s to -1.8% for F1 and -8.4% for F2 took place. This phenomenon can be related to the compounds present in the by-product fibre fraction, which are formed almost completely by insoluble polymers, such as cellulose, lignin and some hemicelluloses. These molecules could modify the capacity of the surface to interact with other compounds because the heat process conditions improved their strong union with wheat components like starch chains (Slavutsky & Bertuzzi, 2014). This effect could explain the lower γ_s of F2, whose composition had a higher cellulose fraction. This compound is presented as a parenchymal structure with a larger contact surface (Figure 1: C-D), and therefore a better capacity to interact with wheat compounds during the process.

3.3 Wettability of model solvents

Figure 3 provides the results of wettability produced by the liquid food solvents, water and oil through the contact angle measure. After firstly analysing the results at time zero, oil in all cases generally presented high contact angles because of its apolar nature compared to the solid components, and with the strong presence of, e.g., carbohydrates and water. The effect of the by-product flours was observed in each solvent assay, where the control sample presented the smallest, and hence the most wettable, surface of them all for both water and oil. F1 and F2 obtained a significantly reduced surface wettability of the matrix for the 20% substitution level for them all, and also at the 10% level for oil. Differences in the wettability reduction tendency for F1 and F2 were observed because of the high slope presented by F2 for water (F1 =0.2043; F2 =0.278). However, few differences in the slopes for oil were found (F1=0.2754; F2 =0.2871).

Regarding the effect of time, the contact angle reduced due to the progressive absorption of the solvent in the matrix. The behaviour of the samples with the by-product flours followed the same pattern to time 0, and the contact angle increased (between substitution levels) at time 0. However from second 10, only F1 with water (Figure 3: A1) presented the same decrement (between substitution levels) to 40 seconds. All the other cases (Figure 3: A2, B1 and B2) followed the tendency given the difficult in absorption for both water and oil, and the cases that contained F2 were the most substantial ones. The results of the solvents' wettability agreed with the surface tension results. The increment in the by-product in the wheat matrix formula matched the reduced surface tension and then the difficulty to interact with solvents. As this effect did not appear to be related only to hydrophobicity as it was observed for both water and oil, perhaps the difficulties to wet the surface could also be attributed to the by-product's influence on the matrix structure. From this point of view, the effect of heat during baking could be important. This hypothesis could be leaned in previous studies, like that reported by Zia-ur-Rehman, Islam, and Shah (2003), where the partial degradation of insoluble polymers, such as cellulose and hemicelluloses, during the cooking process by non-enzymatic depolymerisation was observed in several food products rich in insoluble fibre. Such partial degradation could explain a better union with the interaction zones of starch and proteins (Slavutsky & Bertuzzi, 2014). Figure 1 provides an example of the fibre particles integrated into the wheat matrix after processing. Here we can see that the original parenchymal structure of the white fraction fibre was affected after processing, manifesting presence of starch across the resulting composite structure (Figure 1: C-D). Modification of the fibre from skin (Figure 1: E-F) was not visible, but its links were observed across the particle perimeter.

3.4 Solvents diffusion

To study the effects observed in previous assays from the practical wheat-based matrix food use perspective, the fluxes of the solvents inside the matrices were measured by immersion (see the results in Figure 4). The same tendency of the samples with a different by-product level was once again observed, but in this case they were in the inverse order and no clear linear behaviour was noted, but was inversely proportional to the contact angle in the wettability assay. The samples with a higher contact angle presented less flux. The differences between formulae were less marked under the immersion conditions. So once again the samples that contained F2 were those that presented less flux of solvents. Under the immersion conditions, the flux of oil was slightly higher than that of water.

In general, these observations were contrary to the results reported by Aravind et al., (2012), who suggested a disruption of protein-starch matrix continuity because of the addition of insoluble pollard fibre to wheat pasta, which generated spaces or cracks across the matrix that could promote faster water uptake during cooking. The differences probably lie in wheat matrix preparation, where the effect of heat was not present because pasta was dried, but not at such high temperatures reached during baking. As previously mentioned, the effect of heat produces degradation or changes in the structure of some polymers and cuts their length, whose impact is less on the protein-starch matrix structure when restructured during the dehydration process that takes place in the oven. This effect could also explain why the larger particle size of the by-product flours compared to wheat flour did not have the inverse effect, which could

be expected given its impact on gluten-network development (De La Hera, Rosell, & Gomez, 2014)

The differences between F1 and 2 followed the same lines as in the study of Rodr et al., (2014), where a higher water-holding capacity for a mix of lignin and cellulose extracted from olive stones was observed compared to pure cellulose. Almost equal values were reported for the oil-holding capacity. These results agreed with the behaviour observed of F1 and 2, respectively. Both by-product flours had a general solvent diffusion reduction effect, but the capacity of their components to interact with water and oil could also explain part of the variability noted in the differences found, apart from their influence on the matrix structure. This is coherent to the fact that F2, whose tissue compositions are basically cellulose, had more difficulties to absorb water than F1, which also contained considerable lignin.

To observe the differences in the relationship between the solvent mass updating in the matrix during the immersion time and the observed fluxes, the data of the fluxes and solvents content at each immersion time were plotted. Figure 5 shows the flux of the solvents against the water and oil fraction (X_w and X_o respectively) for each time, starting from the initial amount. The slowdown of flux was according to the increment in the substitution level. Several cases of samples that contained the by-product started at a slightly lower water fraction, but also had less flux, which was more pronounced in F2. Diffusion of oil followed the same pattern and, in this case, the initial oil amount presented by the control sample was smaller (Table 1). However at 40 s, flux was higher compared to the other samples. In this solvent, both types of by-product flours presented a maximum slowdown at the 20% substitution level. Therefore, it is important to note that deceleration of fluxes was not related to the concentration gradient differences between the concentrations of solvents inside and outside the matrices. Although fluxes

reduced following the by-product's substitution level, they approached the control values with time and mass updating.

4. Conclusions

The effect of the tiger-nut milk by-product incorporated into wheat-based matrices was evidenced. Substitution levels from 10% depleted the capacity of the matrix to interact with solvents. The surface tension of matrices diminished following an increase in the substitution level, and reached a maximum (8.4%) when F2 flour was employed at the 20% substitution level. The wettability of the matrix surfaces by water and oil also reduced when the by-product level increased, and significant increases in the degrees of the solvent-liquid contact angles were recorded. Diffusion of solvents was also influenced as the fluxes of both water and oil significantly reduced from the 10% substitution level. With the by-product-type flours, F1 had less impact on matrices than F2, which obtained more marked reductions for all the tested properties. The observed differences could respond to the type of mean insoluble compounds that their proximal composition contained. The heat effect on these insoluble fibre compounds coincided with results reported in previous studies, with changes noted in the capacity to interact with wheat flour components.

The tiger-nut milk by-product can be used to increase the fibre content in wheat-based products with no modifications when all the by-product (F1) is used until the 5% substitution level. However with the white fraction of the by-product (F2), all the substitution levels led to reduced water flux and from the 10% level with the oil flux.

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Figure 1

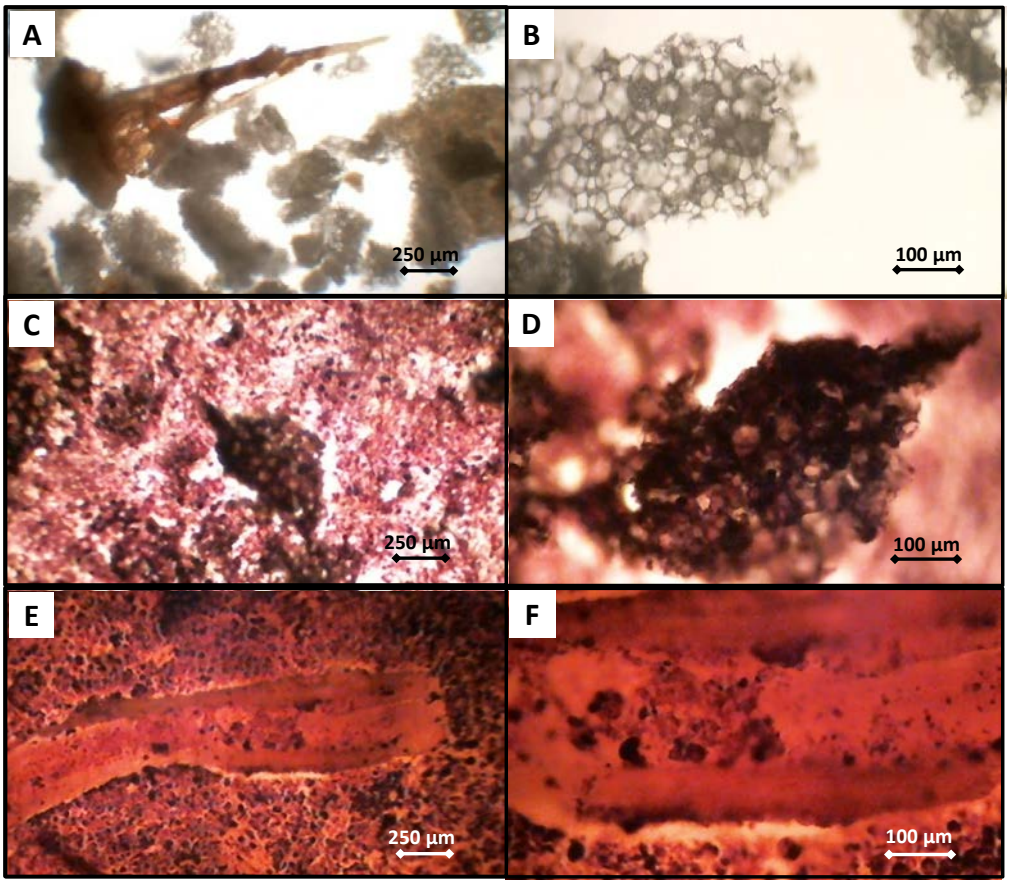

Figure 1. Optic microscopic view of the complete by-product (A), tuber internal tissue particle (B), particles of internal tissue included in the surface of the wheat matrix (C-D), particle of periderm included in the surface of the wheat matrix (E-F). Contrast in C, D, E,F carried out by lugol´s iodine staining.

Figure 2

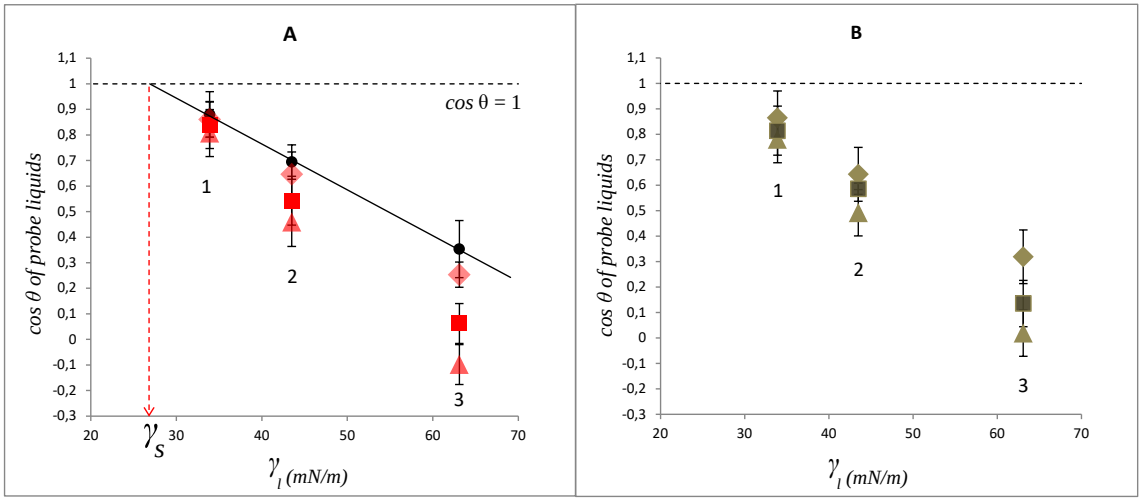

Figure 2. Zisman-plot representing cos θ *vs.* γ_l for the wheat-based matrix formulas. A: F1 substitutions and B: F2 substitutions. Numbers 1, 2 and 3 represent the cos θ of dipropylene glycol, polyethylene glycol 200 and glycerol, respectively. Control ●; F1 5% ♦, 10% ■ and 20% ▲; F2 5% ♦, 10% ■ and 20% ▲. Black dashed line represents cos θ = 1. Red dashed line depicts the γ_s for the control formula.

Figure 3

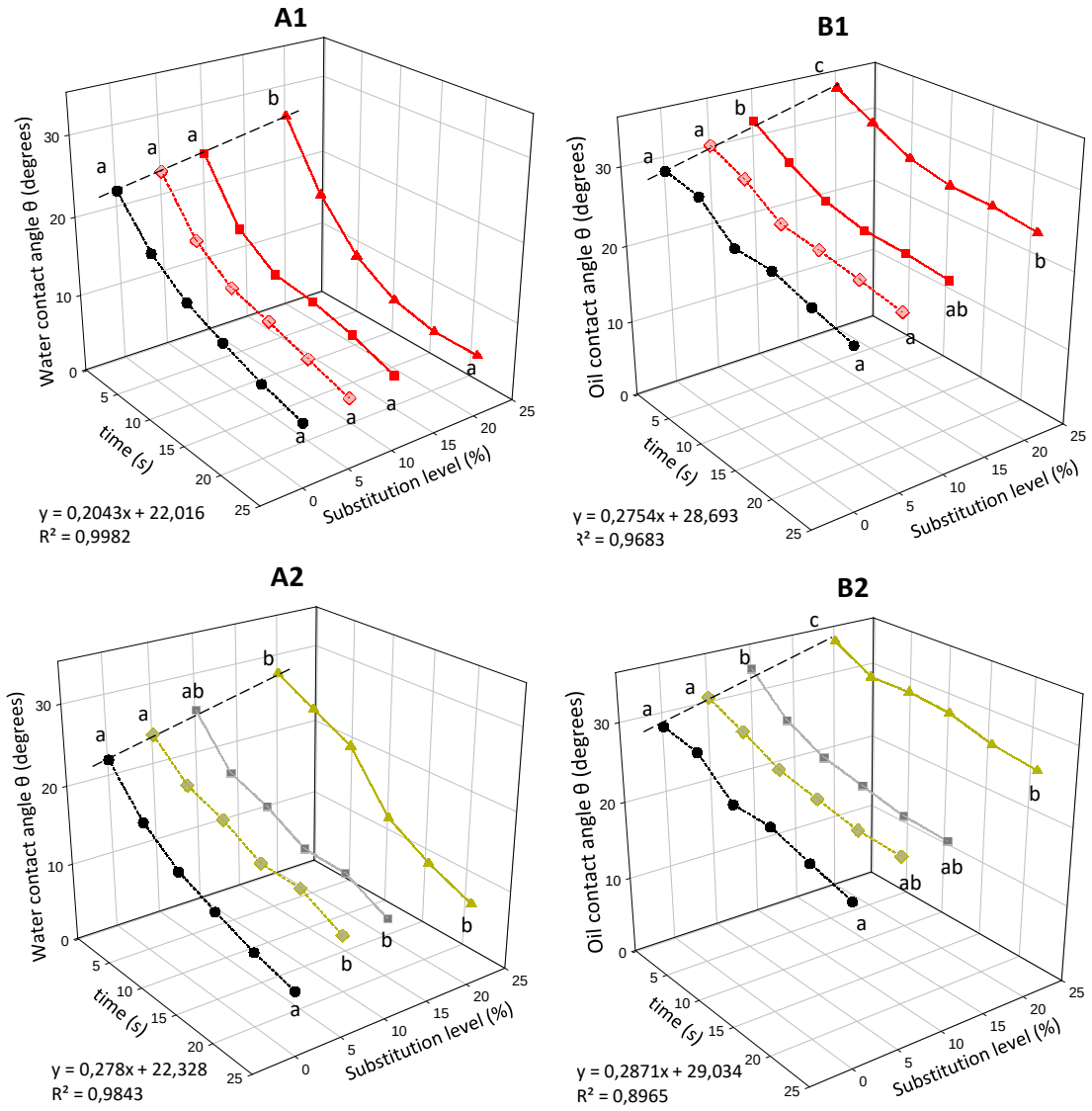

Figure 3. Drop contact angle degrees of water (A) and oil (B) *vs.* time (s) for the wheat-based matrix formulas. A1: A1: F1/water; B1: F1/oil; A2: F2/water; B2: F2/oil. Control ●; F1: 5% ♦, 10% ■ and 20%▲; F2: 5% ♦, 10% ■ and 20%▲. Linear fit at t0 is represented by dashed lines, and their regression equations and correlation coefficients are indicated. Letters mean significant differences of degrees and flours at t0 and t25 seconds in the same solvent at $p \leq 0.05$.

Figure 4

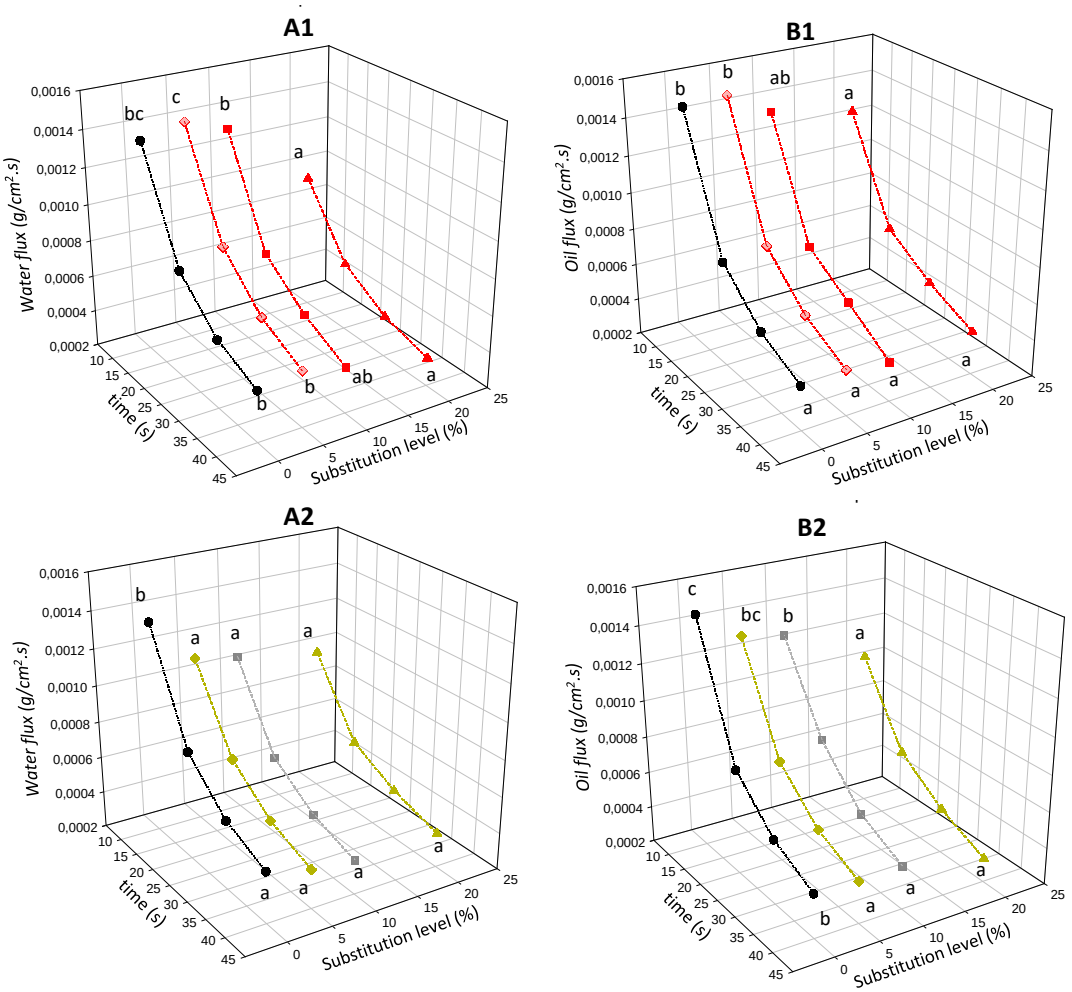

Figure 4. Flux of solvents with time (g/cm^2.s). A1: F1/water; B1 F1/oil and A2: F2/water; B2: F2/oil. Control ●; F1: 5% ♦, 10% ■ and 20%▲; F2: 5% ♦, 10% ■ and 20%▲. Letters mean significant differences of degrees and flours at 10 sec in the same solvent at $p \leq 0.05$.

Figure 5

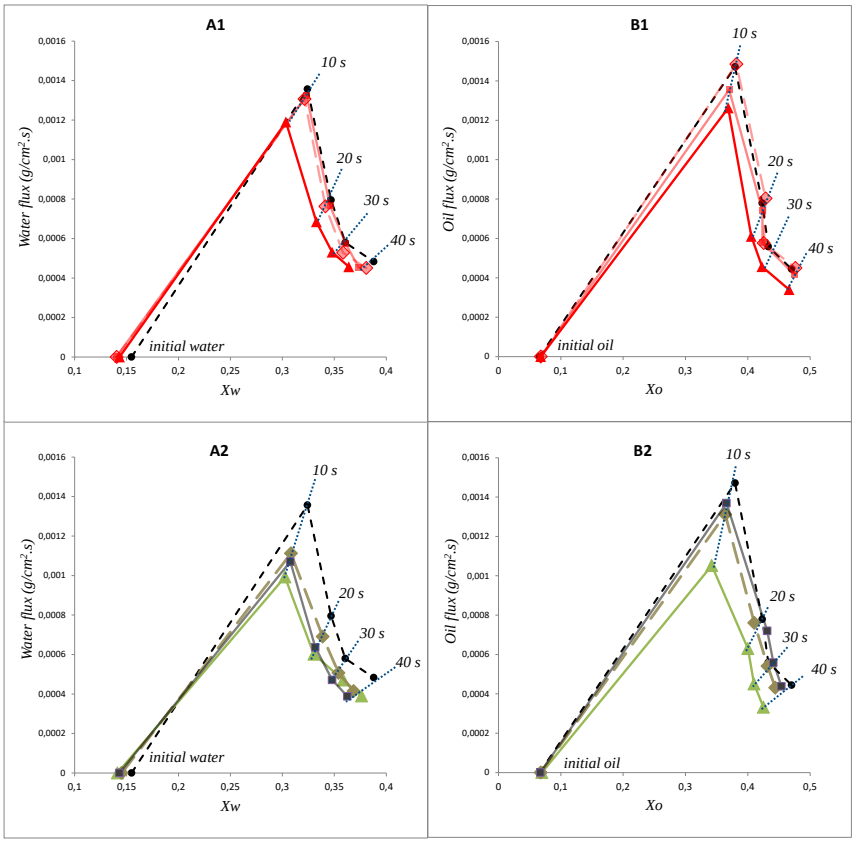

Figure 5. Flux of solvents (g/cm^2.s) *vs.* water content (X_w)/ oil content (X_o) during immersion. A1: F1/water; B1 F1/oil and A2: F2/water; B2: F2/oil. Control ●; F1: 5% ♦, 10% ■ and 20%▲; F2: 5% ♦, 10% ■ and 20%▲. Initial moisture and time sections in seconds are indicated.

Table1. Results of mass loss, water and oil amount, surface tension and solvent diffusion.

	Baking process and composition			Surface tension			Wettability		Solvents diffusion			
Formula	ΔM_b	X_w	X_o	R^2	γ_s	$\Delta\gamma_s$	$\theta_w\ t_{0s}$	$\theta_o\ t_{0s}$	$J_w\ t_{10s}\ (x10^{-3})$	$J_o\ t_{10s}\ (x10^{-3})$	X_{wf}	X_{of}
Control	-32.5 ± 0.1 a	0.154 ± 0.013 a	0.066 ± 0.001 a	0.999	26.9 ± 0.3 c	-	22,0 ± 0.6 a	28.3 ± 1.3 a	1.35 ± 0.09 c	1.47 ± 0.08 bcd	0.387 ± 0.01 b	0.47 ± 0.012 c
F1 5%	-33.6 ± 0.8 a	0.14 ± 0.021 a	0.068 ± 0.001 a	0.992	26.8 ± 0.5 c	-0.6	23.9 ± 0.8 a	30.1 ± 1.5 a	1.3 ± 0.1 bc	1.48 ± 0.16 d	0.38 ± 0.011 b	0.476 ± 0.011 c
F1 10%	-34 ± 0.5 a	0.14 ± 0.017 a	0.068 ± 0.002 a	0.989	26.5 ± 0.5 c	-1.6	23.9 ± 1,0 a	32 ± 1.7 b	1.32 ± 0.1 ab	1.35 ± 0.02 cd	0.373 ± 0.012 a	0.474 ± 0.009 c
F1 20%	-33.4 ± 0.5 a	0.143 ± 0.016 a	0.067 ± 0.002 a	0.963	26.5 ± 0.3 c	-1.8	26.1 ± 1,0 bc	33.8 ± 0.9 c	1.19 ± 0.1 a	1.26 ± 0.08 ab	0.363 ± 0.09 a	0.466 ± 0.011 bc
F2 5%	-34.2 ± 1.2 a	0.145 ± 0.016 a	0.067 ± 0.001 a	0.999	25.4 ± 0.3 b	-5.5	23.7 ± 3.7 a	30.6 ± 1.3 a	1.11 ± 0.11 bc	1.31 ± 0.03 abc	0.376 ± 0.01 a	0.444 ± 0.008 b
F2 10%	-35.1 ± 0.8 a	0.143 ± 0.01 a	0.067 ± 0.002 a	0.996	25.4 ± 0.4 ab	-5.8	25.5 ± 3.7 ab	33 ± 1.3 bc	1.07 ± 0.05 a	1.26 ± 0.03 bcd	0.368 ± 0.012 a	0.453 ± 0.01 b
F2 20%	-33.4 ± 1,0 a	0.141 ± 0.013 a	0.069 ± 0.001 a	0.977	24.7 ± 0.3 a	-8.4	27.6 ± 3.6 c	34.1 ± 1.1 c	0.99 ± 0.1 a	1.05 ± 0.1 a	0.362 ± 0.011 a	0.425 ± 0.012 a

F1: Flour 1; *F2*: Flour 2; ΔM_o: increment of mass during baking loss (%); X_w: water fraction (w.b); X_o: oil fraction (w.b); R^2: correlation coefficient between the surface tension of probe liquids and the cosine of the probe liquid-matrix surface contact angle; γ_s: surface tension of the matrix surface (*mN/m*); $\Delta\gamma_s$: increment of surface tension compared to the control sample (%);$\theta_w\ t0s$: contact angle of water/matrix in degrees; $\theta_o\ t0s$: contact angle of oil/matrix in degrees; $J_w\ t_{10s}$: water flux at time=10s ($g/cm^2.s$) ; $J_o\ t_{10s}$: oil flux at time=10s ($g/cm^2.s$);X_{wf} : water fraction at the end of the solvent assay; X_{of} : oil fraction at the end of the solvent assay. Letters in columns mean significant differences at $p \leq 0.05$.