

## Research Article

### Influence of the Composition of Coconut-Based Emulsions on the Stability of the Colloidal System

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**Abstract:** The object of this research was to determine the influence of the composition of coconut milk-based emulsions on its physical and chemical stability. Coconut represents a highly important product globally and its competitiveness is restricted to its commercialization in the form of cakes, flour and coconut oil mainly to the European Community. The search for new alternatives to diversification of products as raw materials for food, pharmaceutical and cosmetic sectors represents a potential challenge. Coconut Pulp (CP) was milled with its Coconut Water (CW), separating the Coconut Fiber (CF), which was then dried at 40°C and milled for its use in emulsions and homogenized at 10.000 rpm during 10 min. The surface response methodology was used with a composite central design (21 experiments), considering the independent variables: (CW+H<sub>2</sub>O)/CP (1.5-2.5); xanthan gum (G<sub>Xanthan</sub>): 0.25-0.75%, CF: 2.5-7.5%; tert-butyl hydroquinone (TBHQ): 100-200 mg/kg. In addition, dairy serum was used as surfactant and salt. The multiple regression method was used to predict the linear and quadratic terms and the interaction of the independent variables in the models. The optimal conditions were: (CW+H<sub>2</sub>O)/CP: 2.0; G<sub>Xanthan</sub>: 0.5%; CF: 5.0%; TBHQ: 200 mg/kg, reaching potential  $\zeta$ : -45.578±2.478 mV, index of stability through spectral absorption (R): 0.851±0.025; viscosity ( $\mu$ ): 741.7±25.5cP, color (L\*:67.5±0.7, a\*: 3.2±0.2, b\*:8.6±0.5), Peroxide index (PI): 0.142±0.038 meqH<sub>2</sub>O<sub>2</sub>/kg, particle size (D<sub>10</sub>:4.3±0.8 μm, D<sub>50</sub>:323.7±43.6 μm and D<sub>90</sub>:743.0±65.1 μm) and Total Solids (TS): 19.981±0.303%. The results confer good physicochemical stability in the colloidal system studied, which could guarantee its effective use in the subsequent process, like in spray drying to obtain coconut powder.

**Keywords:** *Cocos nucifera L.*, colloids, fiber, physicochemical stability, peroxide index, potential- $\zeta$

## INTRODUCTION

Coconut (*Cocos nucifera Limn*) is a palm tree from the Arecaceae family broadly distributed in tropical and subtropical climates, it is known as the tree of life due to the use of the totality of the CP, CW, husk, shell, wood, leaves, spikelet, etc. The fruit has an endosperm (CP), CW and a very hard husk or shell. The CP provides important food constituents, highlighting its water content (51.9%), fatty acids principally lauric and myristic acids (26.1%), carbohydrates (15.1%), vitamins C, thiamine, riboflavin, niacin; and minerals, like iron, calcium and phosphorus, which provide 293 kcal of energy (Ochoa-Velasco *et al.*, 2014).

The endosperm is economically the most important part of the fruit, providing products, like copra, coconut oil and coconut milk (Siriphanichet *et al.*, 2011). Coconut milk is a natural emulsion of oil in water, thermodynamically unstable, obtained from the aqueous extraction of the endosperm and used as a food

ingredient to provide creaminess and unique aroma in culinary, especially in regions of Asia and the Pacific. Additionally, the composition of coconut milk, including the protein, varies according to the coconut's variety, age, growth environment and the method and conditions used in the extraction process (Tangsuphoom and Coupland, 2008). Some researchers have indicated that coconut milk contains approximately 54% humidity, 35% fat and 11% without solid fat (Simuanget *et al.*, 2004; Tangsuphoom and Coupland, 2008).

Coconut is freely used as a refreshing beverage and as an ingredient in confectionery, ice cream, cookies, cakes and bread (Solangi and Iqbal, 2011; Appaiahet *et al.*, 2015). Oil extracted from coconut is used as cooking oil, hair oil, lamp oil and as an essential ingredient in soap manufacture (Solangi and Iqbal, 2011). Coconut water is an outstanding natural beverage that is gaining popularity in the field of sports science and the beverage industry, given its high

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nutritional value because it contains salts, sugars and vitamins, effectively replenishing electrolytes lost during exercise (Tan *et al.*, 2014; Prado *et al.*, 2015). In addition, it is attributed to potential therapeutic properties, including prevention and relief of many health problems: dehydration, constipation, digestive problems, fatigue, sunstroke, diarrhea, kidney stones and urinary tract infections (DebMandal and Mandal, 2011; Tan *et al.*, 2014) and an antiaging effect (Ge *et al.*, 2006; Prado *et al.*, 2015). A sub-product of the coconut agroindustry is the fiber obtained after extracting the coconut milk, which has an important content as a dietary fiber with health implications, including the prevention of chronic disease, such as cancer, cardiovascular disease and diabetes mellitus (Raghavendra *et al.*, 2006; Trinidad *et al.*, 2006; Yallegama *et al.*, 2013).

Emulsions and dispersions are colloidal systems thermodynamically unstable from a physicochemical point of view; rapidly or slowly, they can show phase separation (Piorkowski and McClements, 2014). System stabilization is achieved normally by adding to the colloidal system surfactant components of amphiphilic nature (polysorbates, phospholipids, among others) and/or proteins and/or thickening agents (gums, gelatin, among others). The stability of colloidal systems, according to the Derjaguin-Landau-Verwey-Overbeek (DLVO) theory, is generally governed by the combination of Van der Waals forces and electrostatic repulsion to obtain a global interaction potential between two particles, in function of their distance, when these are in an ionic medium (electrostatic colloid) (Piorkowski and McClements, 2014; Mirhosseini *et al.*, 2008). Further, other types of forces interact, like steric, hydration, hydrophobic and phase separation forces (Piorkowski and McClements, 2014).

The aim of the research was to evaluate the influence of the composition of coconut-based emulsions, native fiber, antioxidant and other additives on the stability of the colloidal system, for use in spray drying.

## MATERIALS AND METHODS

The study used coconuts (*Cocos nuciferaL.*) 'EnanoMalayo' (Manila) or 'Alto Pacífico' (typical) varieties from the Colombian Pacific region, with flowering age to the harvest of approximately 12 months and postharvest time between 15 and 36 days. The emulsion formulation used additives, like Tert-Butyl Hydroquinone (TBHQ) (Team) as antioxidant agent E-319, G<sub>Xanthan</sub> 200 mesh as a stabilizer, dairy serum instant WCP 80 as a surfactant and table salt (Refisal) to increase repulsive forces and the system's stability.

Characterization of the coconut and emulsions was carried out by using the following methodologies: humidity was determined according to the official AOAC method 930.15/90. pH was determined by using a potentiometer through immersion of the electrode in

the sample, with prior calibration with buffer solutions at pH 2, 4, 7 and 10 at 25°C (AOAC (Association of Official Analytical Chemists), 1990 method 981.12/90). Soluble solids were determined via refractometric reading (AOAC (Association of Official Analytical Chemists), 1990 method 932.12/90). Coconut water acidity was determined through the potentiometric titration method (AOAC method 942.05/90), expressed in malic acid and for CP the acidity index was determined in terms of the lauric acid. Water activity ( $a_w$ ) was determined with a spray point hygrometer at 25°C (Aqualab series 3TE, Decagon, Devices, Pullman, WA, USA) (Cortés and Chiralt, 2008). The peroxide index (PI) was determined through a spectrophotometric method based on the capacity of peroxides to oxidize ferrous ions into ferric ions that react with diverse reagents that produce colored complexes (Hornero-Méndez *et al.*, 2001). The PI serves as a useful indicator of the degree of oxidation of lipids, fats and oils and was expressed as meq of H<sub>2</sub>O<sub>2</sub>/kg sample Eq. (1), where A<sub>m</sub> and A<sub>b</sub> correspond to the absorbance of the sample and the target, respectively, at a 500-nm wavelength; m = slope of the calibration curve; W = sample weight (g); 2 = conversion factor to express as meqH<sub>2</sub>O<sub>2</sub>, 55.84 = molecular weight of iron and V<sub>t</sub> = final volume (mL) of the reaction mix was used to quantify the raw matter and the emulsion:

$$\frac{\text{meqH}_2\text{O}_2}{\text{kg sample}} = \frac{(A_m - A_b) * 1/m}{W * 2 * 55.84} * V_t \quad (1)$$

The extractable oil content was determined according to the method described by Bae and Lee modified (2008). One gram ( $\pm 0.0001$ ) of the sample was weighed in a 15-mL glass tube and 4 mL of water (40°C) was added and agitated with a *vortex* for 2 min. The initial mixture was mixed with 25 ml of hexane/isopropanol solution (3:1 v/v) agitating for 5 min; then, it was centrifuged at 3000 rpm/2 min, separating the organic phase, which was thereafter heated to 70°C/5 h. The extractable oil content was quantified as a difference of weights before and after heating. The Color was determined by using an X-Rite spectrophotometer with illuminant D<sub>65</sub> and a 10° observer as a reference. From the reflection spectra, color coordinates were obtained from CIE-La\*b\*, where L\* is an indicator of the luminosity, a\* (chromaticity green (-) a red (+)) and b\* (chromaticity blue (-) to yellow (+)) (Cortés and Chiralt, 2008). Viscosity ( $\mu$ ) of the emulsion and of the CW were determined by using a rheometer (Brookfield DV-III Ultra (Brookfield Engineering Laboratories, Inc., USA) coupled to a thermostat bath (Brookfield model TC-502), controlled temperature at 25°C, equipped with the RV4 spindle at a rate from 0.01 to 250 rpm, reporting viscosity in cP at a rate of 250 rpm (Mirhosseini *et al.*, 2008). For CP texture a texture analyzer was used (TA-TA-XT2i, Stable Microsystems Ltd., UK) with a 5-mm

stainless steel cylindrical probe, in a 25 kgf load cell. A puncture test was applied with a constant probe rate of 1.0 mm.s<sup>-1</sup> and 3 mm of penetration, determining the maximum force as the mean of the fluctuating peaks on the plateau before reaching 3 mm of penetration (Prieto *et al.*, 2011).

Zeta potential ( $\zeta$ ). The zeta potential of the emulsions was determined from the electrophoretic mobility and then by applying Henry's equation, using a Zetasizer Nano ZS90 (Malvern Instruments Ltd., Worcester, UK). To avoid multiple effects of particle dispersion and the presence of air bubbles, the emulsions were diluted with deionized water in an emulsion to water ratio (1:100). Experimental data were reported as the average of the three individual injections (Rezvaniet *et al.*, 2012). The stability index through spectral absorption (R) was determined from the ratio of the absorbance at two wavelengths (800 and 400 nm) ( $A_{800}/A_{400}$ ) (Mirhosseini et *al.*, 2008), using a UV-Visible spectrophotometer (Thermo Scientific Evolution 60). Emulsion samples were diluted in water (1:100) and performed in triplicate per emulsion.

Particle size was determined by using the Mastersizer 3000 (Malvern Instrument Ltd., Worcestershire, UK). The samples were dispersed in 500 ml of distilled water until obtaining a darkening value of 10±1%. The size distribution was calculated via Mie's theory using the refraction index of 1.52 (Millqvist-Fureby and Smith, 2007) and particle size was reported as percentiles D<sub>10</sub>, D<sub>50</sub> and D<sub>90</sub>.

Surface tension ( $\sigma$ ) was determined through Noüy's ring method (Makri and Doxastakis, 2007) at 15°C, using a tensiometer (KRUSS K20) with a platinum alloy ring with iridium and surface tension was determined in mN/m.

Bromatological characterization was carried out of the CP, determining the ash content according to the AOAC 942.05/90 method, fat according to the AOAC 920.39/90 method, proteins according to the AOAC 955.04/90 method and total dietary fiber through the official 985.29 method (AOAC (Association of Official Analytical Chemists), 1990).

The CP and CW yields were conducted by initially weighing the whole coconuts. These were washed and disinfected, then the CW was removed and the coconuts were scalded in boiling water ( $T \approx 96^\circ\text{C}$ ) during 20 min, removing the shell from the CP. The fruit yield was determined from the kg CP/kg coconut and kg CW/kg coconut ratios. The CP selected was again subjected to a wash process, disinfection, cut into pieces and milling (mill TM32 INOX BRAHER 3HP-16801002). Lots of emulsion weighing 3000 g were separated; initially, the CP and the CW were homogenized with potable water in a blender (Osterizer 600 Watts) in position III during 5 min. Then, the mixture was filtered by using a 500-μm mesh sieve to separate the fiber from the CM. The fiber obtained was subjected to a drying process at 40°C for 48 h and

milling (mill IKA MF 10.1, USA) to reduce particle size and add to the emulsion (Tangsuphoon and Coupland, 2008). Thereafter, the final emulsion was prepared in a homogenizer (Silverson series L5) using the emulsifying head at 10.000 rpm during 10 min, mixing CM, native milled fiber, dairy serum, table salt, G<sub>Xanthan</sub> and TBHQ. A cooling bath was used to keep the temperature from going over 35°C.

To develop the emulsion formulation, the surface response methodology was used with a central composite experimental design and, thus, establish the principal and combined effects of the independent variables: (A) (CW+H<sub>2</sub>O)/CP ratio (1.5-2.5), (B) G<sub>Xanthan</sub> (0.25-0.75%), (C) native fiber (2.5-7.5%) and (D) TBHQ (100-200 mg/kg) and dependent variables: a<sub>w</sub>, total solids, spectral stability index (R), Peroxide Index (PI), zeta potential ( $\zeta$ ), surface tension ( $\sigma$ ), color (L\*, a\*, b\*), viscosity ( $\mu$ ) and particle size (D<sub>10</sub>, D<sub>50</sub> and D<sub>90</sub>), obtaining 21 randomized experiments with five repetitions in the central point. Table 2 describes the experimental design used. The formulation was adjusted in its composition, using the dairy serum as a surfactant with prior determination of its Critical Micelle Concentration (CMC) (Abascal and Garcia-Fadrique, 2009) and table salt at a concentration of 9 mMol/L, to increase the repulsive forces and improve the stability of the colloidal system (Dickinson and Stainsby, 1982; Piorkowski and McClements, 2014).

To determine the CMC of the surfactant (dairy serum Instant WCP 80), solutions were prepared at 15°C with distilled water to reach the following mass fraction (p/p) concentrations in triplicate: 0% as control, 0.05%, 0.2%, 0.35%, 0.5% and 0.65%. The solutions were homogenized for 5 min at 300 revolutions per minute (rpm) with an agitator (IKA RW20). They were stored for 48 h until starting the determinations to observe possible precipitations and surface tension ( $\sigma$ ) was measured, as already described, by plotting the protein concentration vs. surface tension, determining the CMC (Abascal and Garcia-Fadrique, 2009; Makri and Doxastakis, 2007; González-Tello *et al.*, 2007).

To determine the effect of the independent variables, the multiple regression method was used to predict linear and quadratic coefficients and the interaction of the independent variables in the surface response models, to optimize the proportion of components of the emulsion in terms of the dependent variables. The polynomial model generalized to relate the response to the independent variables was the following:

$$Y = \beta_0 + \beta_A A + \beta_B B + \beta_C C + \beta_D D + \beta_A^2 A^2 + \beta_B^2 B^2 + \beta_C^2 C^2 + \beta_D^2 D^2 + \beta_{AB} AB + \beta_{CW} CW + \beta_{AD} AD + \beta_{BC} BC + \beta_{BD} BD + \beta_{CD} CD \quad (2)$$

where,  $\beta_0$  is a constant,  $\beta_A$ ,  $\beta_B$ ,  $\beta_C$  and  $\beta_D$  is the linear coefficient of each factor;  $\beta_A^2$ ,  $\beta_B^2$ ,  $\beta_C^2$  and  $\beta_D^2$  is the quadratic coefficient of each factor;  $\beta_{AB}$ ,  $\beta_{CW}$ ,  $\beta_{AD}$ ,  $\beta_{BC}$ ,  $\beta_{BD}$  and  $\beta_{CD}$  is the product coefficient of the interactions

of the factors. Adaptation of the models was determined by using the lack-of-fit test and the regression coefficient ( $R^2$ ). Additionally, the Analysis of Variance (ANOVA) was performed with 95% CI. The experimental design matrix, analysis of the results and the optimization procedure were conducted by using Statgraphics Centurion XVI.I software. The experimental data at optimal condition were compared to the adjusted values predicted by the models to verify the regression models.

## RESULTS AND DISCUSSION

**Physicochemical composition of coconut:** Table 1 presents the mean values plus the standard deviations of the distribution of the parts of the shelled coconut and the physical and physicochemical properties of the CP and CW with 15 days of harvest, fresh or pseudo-zero.

Note that the edible and usable part reaches approximately 77% of the whole coconut and where CW represents an important percentage, which is why its use independent or mixed with CP results interesting at industrial level. Haseena *et al.* (2010) found CW content of approximately 251 mL/coconut, while Appaiah *et al.* (2015) found a 1:3 water: pulp ratio, with a water content of  $117\pm42.5$  g/coconut and pulp weight of  $330\pm19.5$  g. Luengwilaiet *et al.* (2014) found a percentage distribution of shelled coconut similar to that in this research: CP (35-50%), CW (19-28%) and shell (25-34%). Other research found that in full maturity, the percentage distribution of coconut was: husk (31-54%), shell (12-16%), CP (28-33%) and CW between 6 and 25% (Guarte *et al.*, 1996; Siriphanich *et al.*, 2011; Assa *et al.*, 2010). This variability in the results from diverse investigations could be attributable to factors, type of soil, agronomic management, climatology, physiology of the coconut varieties used (non-climatic product), which is promoted by

respiration and transpiration and by absorption of CW by the solid endosperm (Siriphanich *et al.*, 2011).

The CP and CW, in general, show high values of  $a_w$  and humidity; while CW presents low acidity values ( $0.041\pm0.014$ ) corresponding to  $pH = 5.7\pm0.4$ , making them quite susceptible to microbial degradation, which is why after removing the husk these should be stored in refrigeration or freezer. In addition, total solids of the CP are higher than the CW, at a ratio of 10/1 and provided principally by their fat and fiber contents. Nutritionally, the coconut's fat content represents an important caloric contribution, with a lauric acid content of approximately 50% and other saturated fatty acids, like caprylic and myristic acids (Marina *et al.*, 2009; Raghavendra and Raghavarao, 2010; Assa *et al.*, 2010). An important fiber contribution is noted in CP, mainly comprised of dietary fiber (Raghavendra *et al.*, 2006; Trinidad *et al.*, 2006; Yalegama *et al.*, 2013). Tan *et al.* (2014) reported acidity values for mature CW of  $0.061\pm0.003$  (expressed as malic acid),  $pH 5.71\pm0.10$  and  $^\circ\text{Brix } 4.85\pm0.17$ ; while Prado *et al.* (2015) reported pH values: 5.01 and  $^\circ\text{Brix}$ : 5.00. Trinidad *et al.* (2006) reported higher contents of dietary fiber of the coconut fiber:  $60.0\pm1.0\%$  (56% as insoluble fiber and 4% as soluble), results similar to those reported by Raghavendra *et al.* (2006) and Yalegama *et al.* (2013).

The PI of the CP+CW mix was low, in spite of its high-fat content. However, this parameter can become very critical during its storage, given that it is associated to the degree of oxidation of lipids, fats and oils and could contribute to producing unpleasant flavors that affect negatively the quality of the fruit and its acceptance by consumers (Hornero-Méndez *et al.*, 2001). Raghavendra and Raghavarao (2010) found PI values of  $0.82\pm0.02$  and  $1.46\pm0.06 \text{ meqH}_2\text{O}_2/\text{kg oil}$  and acidity index of  $0.27\pm0.05$  and  $0.91\pm0.02$  in the coconut oil extracted and a commercial coconut oil, respectively.

Table 1: Physicochemical composition of the unshelled mature coconut fruit

Property	Unshelled coconut	CP	CW
Weight (kg)	622.3 $\pm$ 61.9	-----	-----
CP (%)	47.1 $\pm$ 3.1	-----	-----
CW (%)	30.1 $\pm$ 5.2	-----	-----
Shell (%)	26.0 $\pm$ 3.8	-----	-----
Humidity (%)	-----	50.4 $\pm$ 5.2	95.0 $\pm$ 1.6
Acidity index (%)*	-----	0.510 $\pm$ 0.240	-----
Acidity (%) **	-----	-----	0.041 $\pm$ 0.014
$a_w$	-----	0.978 $\pm$ 0.005	0.970 $\pm$ 0.030
$^\circ\text{Brix}$	-----	6.4 $\pm$ 3.0	3.7 $\pm$ 0.6
pH	-----	6.1 $\pm$ 0.2	5.7 $\pm$ 0.4
$\mu$ (Cp)	-----	-----	0.744 $\pm$ 0.06
Texture (N)	-----	81.1 $\pm$ 8.3	-----
Density (g/mL)	-----	-----	1.015 $\pm$ 0.001
L*	-----	71.9 $\pm$ 4.3	51.0 $\pm$ 1.5
a*	-----	-1.1 $\pm$ 0.2	0.3 $\pm$ 1.0
b*	-----	3.1 $\pm$ 0.8	-1.0 $\pm$ 0.3
ÍP (meqH <sub>2</sub> O <sub>2</sub> /kg oil)	-----	0.695 $\pm$ 0.340***	-----
Protein (%bh)	-----	3.27 $\pm$ 0.32	-----
Dietary fiber (%bh)	-----	12.93 $\pm$ 2.47	-----
Fat (%bh)	-----	19.89 $\pm$ 3.03	-----
Ashes (%bh)	-----	1.08 $\pm$ 0.275	-----

\*: Expressed as lauric acid; \*\*: Expressed as malic acid; \*\*\*: Mix of CP+CW

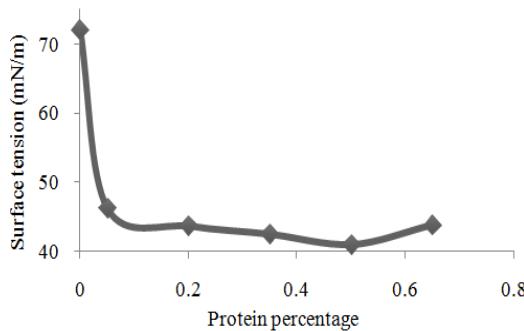


Fig. 1: Surface tension in function of the serum protein concentration

In CP, the acidity index was relatively low ( $0.51 \pm 0.24$ ); however, it constitutes a measurement of the degree of hydrolysis of the lauric, capric, caproic, caprylic, myristic, oleic, palmitic, stearic, vaccenic and linoleic acids present in the food matrix (Santoso *et al.*, 1996; Waisundara *et al.*, 2007; Assa *et al.*, 2010; Appaiah *et al.*, 2015). The cause of the existence of free fatty acids is the enzyme activity of the lipases. This indicates that free fatty acids have started to oxidize into oxygenated compounds, like-for example-hydroperoxides, because of the action of chemical agents (oxygen, temperature, light, metallic traces) or biochemical agents (microorganisms, lipoxidase enzymes) or the combination of both in function of storage conditions and of the composition of the oil stored; values  $\leq 0.8$  are recommended (Kishk and Elsheshetawy, 2013). With respect to color, the  $L^*$  of the CP ( $71.9 \pm 4.3$ ) was higher than that of the CW ( $51.0 \pm 1.5$ ), which makes it whiter or clearer and more brilliant on its surface, mainly because of the fat contribution and the fibrous content (Yalegama *et al.*, 2013; Raghavendra *et al.*, 2006); while chromaticities  $a^*$  and  $b^*$  behaved on the chromatic plane  $a^*b^*$  next to the point (0,0), indicating their a chromaticity (Luengwilai *et al.*, 2014). Some investigations have determined the color of the CP ( $L^* = 67$ ,  $a^* = -0.65$ ,  $b^* = 1.05$ ), results similar to those found in this study (Luengwilai *et al.*, 2014). In CW, according to that reported by Damar (2006), the data show slight changes in values of  $L^* = 59.74 \pm 0.17$ ,  $a^* = -1.71 \pm 0.02$ ,  $b^* = 1.84 \pm 0.02$ , in untreated coconut water, stored at  $25^\circ\text{C}$ , presenting a small decrease from week 0 to week 5 and increasing slightly at week 9, showing values and behavior similar to those found in this study (Purkayastha *et al.*, 2012).

The CP texture behaved like a turgid product with an average level of fracturability, mainly of its fibrous-porous components in the order of  $81.12 \pm 8.27$  N (Yalegama *et al.*, 2013; Raghavendra *et al.*, 2006). The CW rheology behaved like a more fluid product with viscosity lower than that of water, due to its composition, mainly constituted by soluble sugars, minerals, proteins and lipids, influencing significantly on it behaving like a Newtonian fluid. These results agree with that reported by Laux *et al.* (2014), who found equal behavior for cutting rates between 10 and  $400 \text{ seg}^{-1}$ , in all flow tests performed.

**Determination of the CMC:** It was found that dairy serum proteins have surface activity, upon reducing water surface tension to  $\sigma = 41 \text{ mN/m}$ , which is why it is interesting for its application in foods. The CMC was found, determining the minimum concentration at which we obtain the greatest surface activity by these proteins (González-Tello *et al.*, 2007) (Fig. 1).

This behavior is expected due to the amphiphilic nature of these proteins and their marked surface activity. Figure 1 also shows that at 0.05% mass fraction concentration the CMC was obtained, with a surface tension value of  $\sigma = 41 \text{ mN/m}$ . As of this concentration, values increase somewhat and, it is estimated, the asymptotic behavior will occur. This phenomenon is normal with these types of materials; it is known that surfactants have a maximum effective concentration, where the liquid's surface is saturated with the material and from there, its excess will generate aggregations known as micelles, which when associating affect characteristics of the solution, like freezing point and conductivity and even forming precipitates (Salager, 2005). These values are similar to those presented by González-Tello *et al.* (2007) with serum proteins at similar concentrations and to that reported by Abascal and Gracia-Fadrique (2009) with calcium caseinate and sodium solutions, even close values have been found with other proteins, like that extracted from *Phaseolus vulgaris* in the study by Makri and Doxastakis (2007).

**Design of coconut emulsions:** Table 2 presents the means values and standard deviations of the dependent variables evaluated according to the statistical design, while Table 3 presents the ANOVA results. Results of the dependent variables were fitted to a second-order

Table 2: Results of the formulation's experimental design and the homogenizing process of coconut-based emulsions

Exp.	A (CW+H <sub>2</sub> O)/CP	BG <sub>Xanthan</sub> (%)	C Fiber (%)	D TBHQ (mg/kg)	L*	a*	b*	R
1	2.00	0.50	5.00	100	66.3±0.5	3.4±0.1	6.4±0.2	0.86±0.01
2	1.50	0.75	7.50	200	65.1±3.6	4.1±0.2	7.0±0.3	0.86±0.01
3	2.00	0.50	5.00	150	65.7±0.1	4.1±0.2	5.7±0.2	0.83±0.01
4	2.00	0.50	2.50	150	67.3±0.5	4.1±0.1	5.1±0.1	0.84±0.01
5	2.50	0.50	5.00	150	67.9±0.3	2.6±0.1	6.8±0.2	0.83±0.01
6	1.50	0.50	5.00	150	67.6±0.5	2.4±0.2	7.0±0.2	0.87±0.00
7	1.50	0.25	2.50	100	70.8±0.5	1.9±0.2	6.5±0.3	0.90±0.01

Table 2: Continue

Exp.	$\mu$ (cP)	PI(meqH <sub>2</sub> O <sub>2</sub> /kg)	D <sub>10</sub> (μm)	D <sub>50</sub> (μm)	D <sub>90</sub> (μm)	$\zeta$ (μm)	aw (mV)	TS
1	705.2±22.6	0.14±0.07	3.1±0.1	50.3±10.9	537.2±48.6	-52.2±1.6	0.980±0.00	19.6±0.1
2	1530.7±40.3	0.10±0.03	3.2±0.3	127.0±38.0	648.4±49.1	-53.3±1.8	0.982±0.01	24.6±0.2
3	610.6±6.7	0.07±0.03	2.4±0.1	93.3±37.5	574.7±34.0	-55.6±1.8	0.987±0.00	19.1±0.1
4	369.4±2.9	0.06±0.01	1.9±0.5	11.9±3.0	481.8±36.7	-54.7±1.8	0.987±0.00	17.1±0.2
5	732.3±14.6	0.08±0.04	2.1±0.3	157.7±73.4	464.1±99.0	-45.4±6.5	0.992±0.00	16.1±0.2
6	746.5±11.2	0.04±0.03	4.7±0.4	139.5±48.5	546.8±54.7	-47.1±1.6	0.984±0.00	19.1±0.2
7	241.1±13.1	0.05±0.03	8.1±3.1	58.2±17.7	566.9±83.3	-47.9±1.5	0.987±0.00	17.0±0.1
8	2252.7±21.9	0.07±0.02	4.0±0.6	394.7±52.1	920.6±287.7	-47.3±1.0	0.987±0.00	21.0±0.2
9	840.3±10.8	0.04±0.01	3.2±0.2	222.2±36.4	601.4±47.5	-48.6±1.7	0.986±0.00	20.5±0.1
10	1503.3±96.0	0.06±0.02	26.1±14.0	302.1±17.1	666.8±43.4	-44.2±1.2	0.985±0.00	25.2±0.5
11	309.9±4.0	0.11±0.04	3.0±0.2	10.1±1.5	383.8±99.9	-44.7±3.3	0.986±0.00	16.1±0.2
12	1054.3±30.1	0.06±0.03	2.1±0.2	164.8±55.5	533.2±112.0	-45.4±1.3	0.984±0.00	20.2±0.2
13	587.6±21.5	0.03±0.03	2.4±0.2	234.8±46.3	631.6±55.4	-48.6±1.4	0.986±0.00	19.2±0.1
14	679.5±21.1	0.12±0.03	2.4±0.4	155.2±77.7	605.8±135.4	-44.1±1.4	0.985±0.00	21.6±0.1
15	268.8±5.8	0.09±0.04	2.5±0.2	81.9±47.0	621.1±51.3	-45.4±2.1	0.982±0.00	17.8±0.1
16	403.7±12.8	0.06±0.03	1.6±0.1	58.1±21.0	513.7±46.4	-42.8±1.0	0.985±0.00	18.7±0.2
17	406.4±20.9	0.04±0.03	1.7±0.1	126.1±65.4	624.0±99.6	-41.6±0.6	0.989±0.00	20.1±0.1
18	1360.0±85.4	0.09±0.02	3.0±0.2	249.2±38.8	600.9±57.5	-45.9±1.4	0.991±0.00	23.0±0.2
19	298.0±12.2	0.05±0.04	2.5±0.2	239.7±85.5	689.4±95.3	-39.1±2.8	0.988±0.00	19.7±0.2
20	857.3±52.0	0.09±0.02	1.9±0.2	136.5±78.1	647.9±147.1	-44.2±1.8	0.992±0.00	18.5±0.1
21	531.3±14.4	0.18±0.02	1.6±0.1	91.9±36.9	802.9±90.0	-45.2±2.1	0.972±0.01	13.6±0.1

Table 3: ANOVA (p-values) for surface response models

Variables	Principal effects				Quadratic effects			
	A	B	C	D	A <sup>2</sup>	B <sup>2</sup>	C <sup>2</sup>	D <sup>2</sup>
L*	0.0000*	0.9036	0.0220*	0.8739	0.0148*	0.5559	0.1724	0.0236*
a*	0.0000*	0.1945	0.0542	0.3675	0.0000*	0.0000*	0.0000*	0.0000*
b*	0.0834	0.0000*	0.0000*	0.3314	0.6763	0.0000*	0.0000*	0.0000*
R	0.7768	0.6701	0.1310	0.1161	0.3089	0.1615	0.5550	0.0726
$\mu$	0.0000*	0.0000*	0.0172*	0.5493	0.0863	0.9429	0.0000*	0.0000*
PI	0.1341	0.0103*	0.9708	0.0736	0.9951	0.5578	0.3366	0.0446*
D <sub>10</sub>	0.0000*	0.0000*	0.0000*	0.0000*	0.0193*	0.4071	0.3145	0.0430*
D <sub>50</sub>	0.5576	0.0000*	0.0000*	0.0000*	0.7829	0.0000*	0.0925	0.2158
D <sub>90</sub>	0.0505	0.0000*	0.6537	0.0000*	0.2154	0.0000*	0.7491	0.1253
Z	0.3405	0.0000*	0.0000*	0.0000*	0.1461	0.0000*	0.0000*	0.0000*
a <sub>w</sub>	0.3605	0.0126*	0.0772	0.0314*	0.4165	0.3126	0.1849	0.0000*
TS	0.0000*	0.0000*	0.0000*	0.2981	0.0000*	0.0409*	0.0210*	0.0345*
Effects of the interaction								
Variables	AB	AC	AD	BC	BD	CD		
L*	0.3904	0.0000*	0.1321	0.0345*	0.2622	0.0000*		
a*	0.6333	0.0000*	0.0009*	0.0000*	0.8573	0.0213*		
b*	0.0000*	0.6104	0.0000*	0.0000*	0.3222	0.0000*		
R	0.5116	0.1534	0.3463	0.4590	0.1666	0.6243		
$\mu$	0.0000*	0.1101	0.6171	0.0000*	0.0476*	0.0000*		
PI	0.0000*	0.0133*	0.5969	0.1250	0.9920	0.0901		
D <sub>10</sub>	0.0000*	0.0000*	0.0000*	0.0000*	0.0143*	0.0000*		
D <sub>50</sub>	0.0000*	0.0201*	0.0000*	0.6555	0.3469	0.0000*		
D <sub>90</sub>	0.0000*	0.0151*	0.0000*	0.0362*	0.0238*	0.8448		
Z	0.0306*	0.4558	0.0359*	0.0001*	0.9901	0.0000*		
a <sub>w</sub>	0.3407	0.0000*	0.6234	0.0365*	0.0000*	0.0000*		
TS	0.1336	0.0000*	0.8323	0.0000*	0.0293*	0.0000*		

\* Significant (p<0.05)

polynomial regression model and the ANOVA showed that the surface response models were significant with 95% CI for all the regression equations

**Water activity ( $a_w$ ) and total solids:** Figure 2 presents the surface response plots of the  $a_w$  and the TS in function of the independent variables. The  $a_w$  showed significant differences ( $p<0.05$ ) with respect to G<sub>Xanthan</sub> and to the TBHQ content; as well as with respect to the G<sub>Xanthan</sub>-fiber percentage interaction. The TS presented significant differences ( $p<0.05$ ) with respect to the G<sub>Xanthan</sub>, fiber percentage and TBHQ quadratic interactions and with the G<sub>Xanthan</sub>-TBHQ interaction.

The emulsion values of  $a_w$  showed little fluctuation ( $0.972\pm0.01$ - $0.992\pm0.00$ ), conserving high values that denote a favorable condition for microbiological growth and deterioration reactions, like lipid oxidation and

non-enzymatic browning (Appaiah *et al.*, 2015; Haseena *et al.*, 2010).

The  $a_w$  had an inversely proportional effect with respect to G<sub>Xanthan</sub>, while TBHQ had an opposite effect to G<sub>Xanthan</sub>. Note that TS varied between  $13.6\pm0.1$  and  $25.2\pm0.5\%$ , highlighting their increase due to the effect of increased fiber percentage, which is favorable in spray drying processes, given that it improves the productive and energetic yield (Phisut, 2012; Khuenpet *et al.*, 2016; Tontul and Topuz, 2017).

**Color:** Figure 3 plots the L\* surface response in function of the independent variables. The color of the emulsions (L\*, a\* and b\*) revealed the influence of the linear effects of the independent variables considered and their linear and quadratic interactions, varying the averages of the ranges (71.7-64.5), (4.1-1.8), (8.7-5.1), respectively. According to these results, changes on the

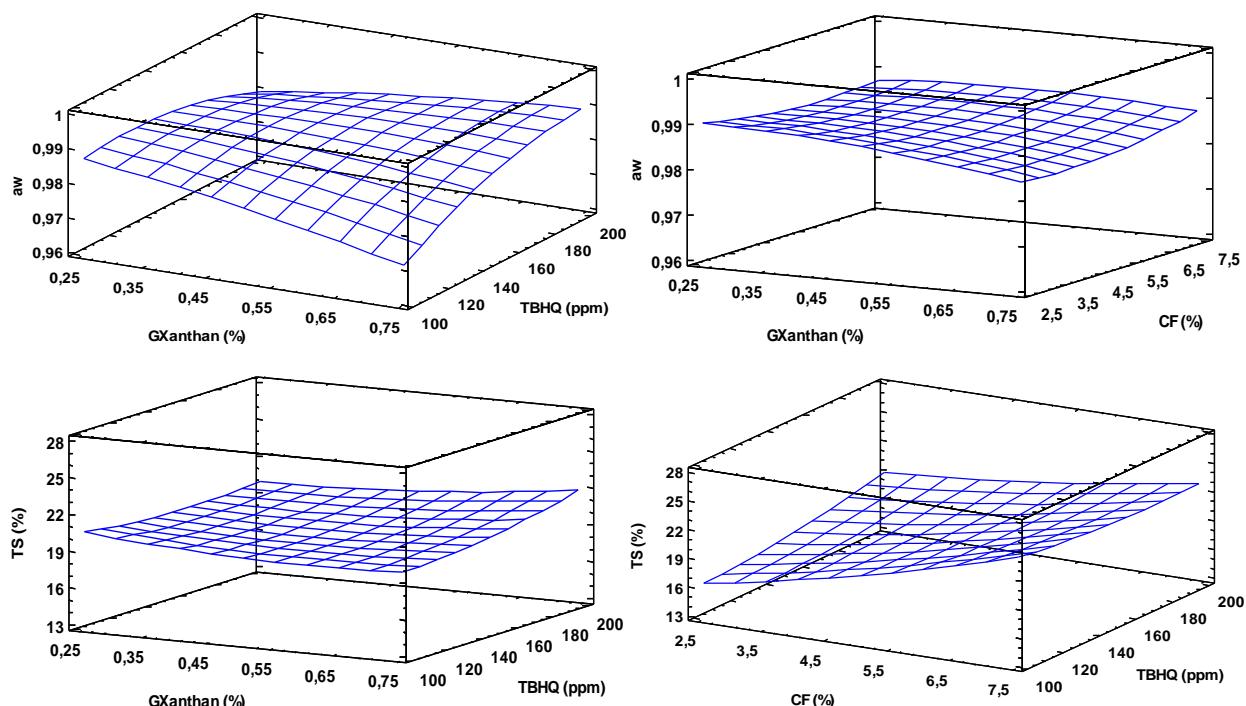


Fig. 2: Surface response plots of the  $a_w$  and the total solids in function of the independent variables

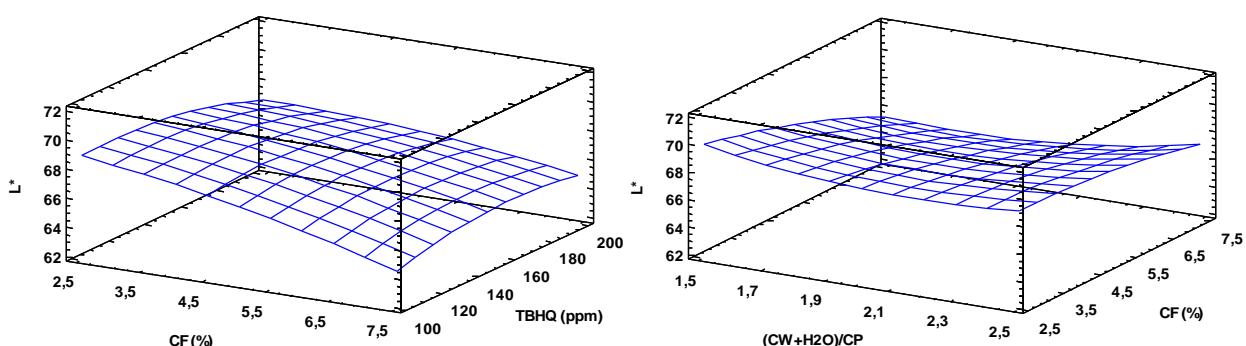


Fig. 3: Graphics of the L\* response surface

$a^*b^*$  chromatic planeare minimal or inappreciable to the human eye, given that they are in the grey zone (achromatic zone) (Gilabert, 1998; Alvarado and Aguilera, 2001), which is why  $L^*$  represents the most important color variable or control variable.

The ANOVA presented statistically significant differences ( $p<0.05$ ) in  $L^*$  with respect to the fiber percentage; additionally, due to the effect of the quadratic interactions of the (CW+H<sub>2</sub>O)/CP ration and of the antioxidant (TBHQ) and because of the G<sub>Xanthan</sub>-fiber percentage linear interaction. The  $L^*$  present a decreasing trend with the increased fiber percentage present, perceiving less clear emulsions.

This could be attributed to diverse factors, like the greater presence of the fiber generates a lower reflection of light (Prieto *et al.*, 2011) and loss of translucency of the emulsion, permitting the passage of light-although in a diffuse manner (Ng *et al.*, 2014). Furthermore, higher fiber content increases the content of brown pigment of the endocarp (zone adjacent to the husk).

A decrease of  $L^*$  occurs at low (CW+H<sub>2</sub>O)/CP ratios, which could be attributed to higher CP content in the formulation, corresponding to a higher contribution of fiber and oil contained in the formulation. Under these conditions and bearing in mind the addition of dry fiber and increased temperature (20→35°C) and air content during the homogenizing process, the emulsions are perceived darker. This phenomenon, as a whole, is associated to the higher content of solids, lipid oxidation with the production of hydro-peroxides and free radicals (Ochoa-Velasco *et al.*, 2014; Kuhn and Cunha, 2012), possible enzymatic browning due to the presence of peroxidase and polyphenol oxidase enzymes (Borda, 2011) and non-enzymatic (Prieto *et al.*, 2011). In addition, the positive fiber percentage-TBHQ interaction enhances diminished  $L^*$  at high fiber and TBHQ contents.

Some researchers, as described ahead, have evaluated the addition of dehydrated CP (60-80°C) in the final product, producing a decrease of  $L^*$  between 66 and 68, attributing the biggest cause of darkening to the Maillard reaction, given the flavor and odor of the final product (Prieto *et al.*, 2011). Other investigations report darkening of the pulp, emulsions and dry fiber due mainly to the process' increased temperature that favors the Maillard reaction, changing color from a pale target to brown and without registering an important loss of quality (Guarte *et al.*, 1996; Chiewchan *et al.*, 2006). Tipvarakarnkoon *et al.* (2010) report  $L^*$ : 81.72±1.50;  $a^*$ : -0.66±0.18,  $b^*$ : 2.1±0.73 values in "commercial coconut milk"; while in "coconut milk" elaborated with diverse types of acacia gum, with and without Tween 60, with and without G<sub>Xanthan</sub> and/or guar gum, they showed significant differences principally in  $L^*$ , but did not highlight notable changes in chromaticities  $a^*$  and  $b^*$ , results quite similar to those obtained in this research.

In general, changes in the optical properties of the coconut emulsions are mainly determined by their composition, temperature, relative refraction index, concentration and size distribution of the oil drops and dispersed particles, which have important implications in the development of transparent or opaque beverages (Piorkowski and McClements, 2014).

**Physicochemical stability of the colloidal system:** Figure 4 presents the surface response plot of parameters associated to the physicochemical stability of the emulsion:  $\zeta$ ,  $\mu$ , D<sub>10</sub>, D<sub>50</sub>, D<sub>90</sub> and  $\sigma$  in function of the independent variables.

**R index and particle size:** The stability index, R, did not show significant differences ( $p<0.05$ ) with respect to the independent variables, given that its variation range was low (0.83-0.90). The R index is based on light dispersion properties. It is related to the mean particle size (Mirhosseini *et al.*, 2008; Homayoonfal *et al.*, 2015), which is why it is considered that these values were high. This was evidenced in the D<sub>10</sub>, D<sub>50</sub> and D<sub>90</sub> percentiles that were big and fluctuating: D<sub>10</sub> (1.62±0.10 - 26.08±14.00  $\mu$ m), D<sub>50</sub> (11.90±2.96-302.11±17.01  $\mu$ m) and D<sub>90</sub> (383.78±99.86-920.56±287.75  $\mu$ m), which could favor phase separation and identify the need for other mechanisms for their stabilization.

The D<sub>10</sub> percentile presented significant differences ( $p<0.05$ ) with respect to las quadratic interactions of the (CW+H<sub>2</sub>O)/CP ratio and the TBHQ, in addition to the G<sub>Xanthan</sub>-TBHQ interaction, while the D<sub>50</sub> percentile was significant with the (CW+H<sub>2</sub>O)/CP-fiber percentage interaction. The D<sub>90</sub> percentile showed significant differences ( $p<0.05$ ) with respect to the linear interactions of the (CW+H<sub>2</sub>O)/CP-fiber percentage ratio, G<sub>Xanthan</sub>-fiber percentage and G<sub>Xanthan</sub>-TBHQ. Mainly highlighted is the increase in the D<sub>10</sub> percentile to high contents of fiber and low (CW+H<sub>2</sub>O)/CP ratio, besides-contrary to expected-with decreased G<sub>Xanthan</sub>. In addition, the increase in D<sub>50</sub> is produced with increased fiber percentage and the increase in D<sub>90</sub> is produced by increased G<sub>Xanthan</sub> and fiber percentage.

In general, the percentile differences can be attributed mainly to the complexity of the raw matter regarding fruit maturity and composition (oil fiber, protein, others), to the G<sub>Xanthan</sub> composition that correlates directly with viscosity and its shear effect and particle disintegration, acting to reduce the size of the oily drops and of the high-hardness coconut fiber (Raghavendra *et al.*, 2006). It is considered that among the particle size distribution, the smallest are associated to drops of oil, while the biggest are associated to the suspended fibers in the emulsion (Wallecan *et al.*, 2015). This behavior is very similar to that reported by Homayoonfal *et al.* (2015) and Tipvarakarnkoon *et al.* (2010), which report that the intensity of the shear

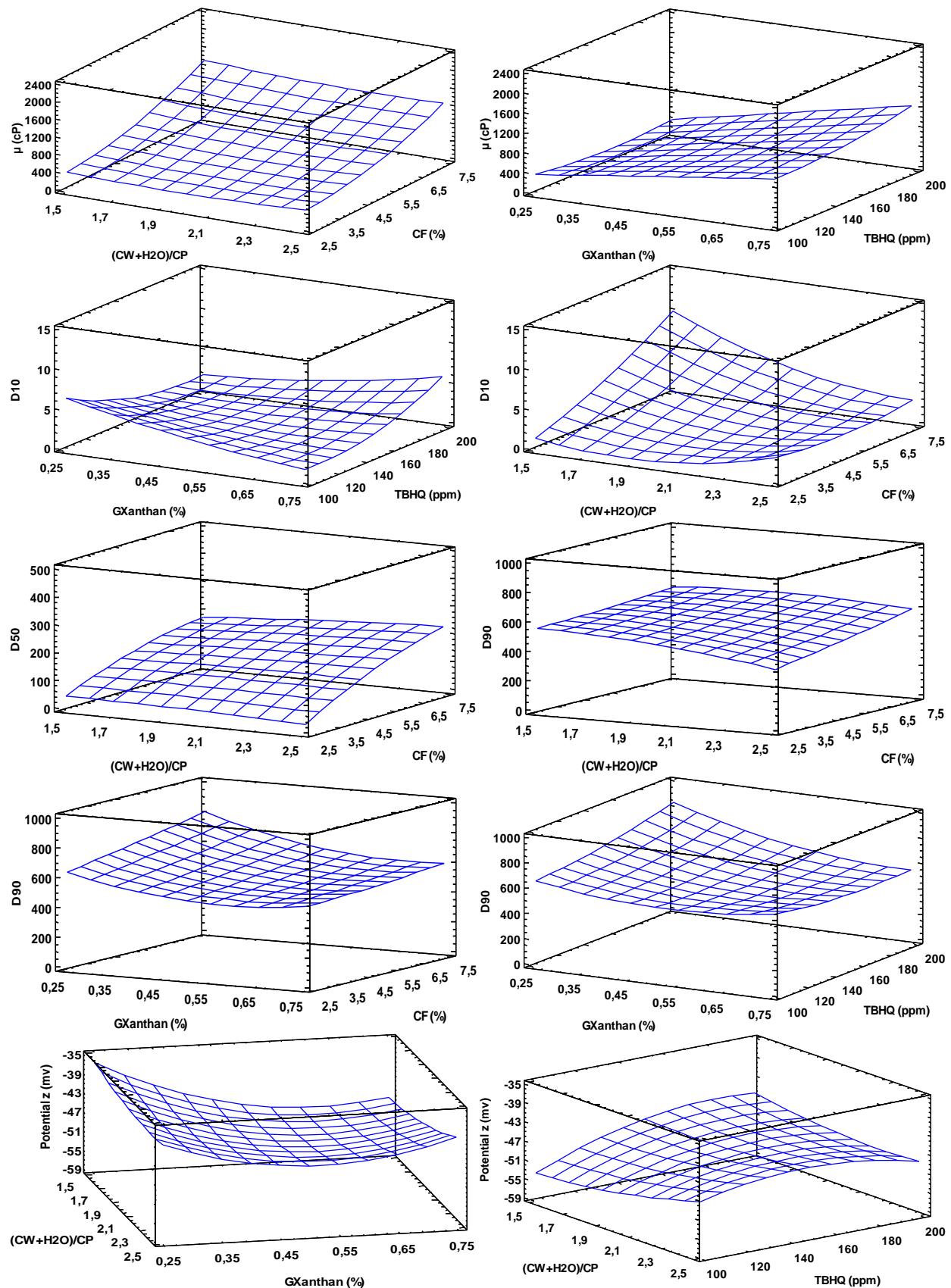


Fig. 4: Surface response plots of parameters associated to the stability of the emulsion in function of the independent variables

force, turbulence and cavitation created by the homogenizer determine the mean particle size and increased homogenization time leads to a higher temperature, provoking decreased interfacial tension and viscosity (Anarjan *et al.*, 2010).

**Viscosity:** The  $\mu$  presented significant differences ( $p<0.05$ ) with respect to fiber percentage and to the G<sub>Xanthan</sub>-TBHQ linear interaction, highlighting a trend to increase  $\mu$  with increased G<sub>Xanthan</sub> content, which can bind water subjecting itself to a double helix conformational transition of an aggregate complex through hydrogen bonds and polymer entanglement (Jayme *et al.*, 1999; Niu *et al.*, 2017), which finally modifies the rheological properties of the continuous phase and favoring system stability by reducing the mobility of the colloidal system's particles and delaying phase separation (Jayme *et al.*, 1999; Piorkowski and McClements, 2014; Niu *et al.*, 2017). The increased content of suspended solids provided by the added fiber, also favored increased viscosity; however, given the variability and large particle sizes found in the D<sub>10</sub>, D<sub>50</sub> and D<sub>90</sub> percentiles does not favor system stability.

Some authors have reported that "coconut milk" exhibits pseudo-plastic behavior (Vitali *et al.*, 1985; Simuang *et al.*, 2004; Chiewchan *et al.*, 2006), which is affected by the presence of stabilizers and fat content (Simuang *et al.*, 2004; Tangsuphoom and Coupland, 2005; Chiewchan *et al.*, 2006; Peamprasart and Chiewchan, 2006; Tipvarakarnkoon *et al.*, 2010; Ng *et al.*, 2014; Li and Nie, 2016; Piorkowski and McClements, 2014). Other research on "coconut milk" have used Montanox 60 (emulsifier) and carboxymethylcellulose, reporting increased  $\mu$  of the continuous phase (Jirapeangtong *et al.*, 2008; Tipvarakarnkoon *et al.*, 2010) and favoring its stability upon delaying phase separation or reducing the particle aggregation kinetics (McClements, 2004a, 2004b; Klinkesorn *et al.*, 2004; Phungamngoen *et al.*, 2004; Mantzouridou *et al.*, 2012). The aforementioned coincides with that reported by Mirhosseini *et al.* (2008), with orange beverage emulsions containing high concentrations of gum arabic and G<sub>Xanthan</sub>, exhibiting high apparent viscosity and relatively medium drop size.

The  $\mu$  showed the tendency to increase with increased fiber content, which has been reported by diverse authors (Pelegrine *et al.*, 2002; Valencia *et al.*, 2004; Elleuch *et al.*, 2011; Castro *et al.*, 2013). Additionally, it is considered that homogenization produces changes of particle properties, influencing on the rheology of the colloidal system (Elleuch *et al.*, 2011; Castro *et al.*, 2013). These changes of  $\mu$  can be higher in some fibers than in others, given the difference of the type of pectin present in the cell structure and its hardness, which makes the homogenization response different. Furthermore, the way groups of cells interrupt through the cell wall or

through the middle lamella seems to have an effect on the rheological properties of the suspensions (Day *et al.*, 2010; López-Sánchez *et al.*, 2011; Elleuch *et al.*, 2011; Castro *et al.*, 2013).

**Potential- $\zeta$ :** The potential- $\zeta$  showed significant differences ( $p<0.05$ ) with respect to the (CW+H<sub>2</sub>O)/CP-G<sub>Xanthan</sub> and (CW+H<sub>2</sub>O)/CP-TBHQ interactions, which derives into a curvature behavior on the response surface. The influence of the G<sub>Xanthan</sub> by being an anionic polysaccharide, due to the carboxyl groups present from the lateral chains (Piorkowski and McClements, 2014), contributed to increasing the repulsive forces in the proximities of the co-ion layer, making their potential- $\zeta$  more negative with the increase of the hydrocolloid in the formulation. The magnitude of the polysaccharide's electric charge depends on pH in relation to the pKa value (Jayme *et al.*, 1999; Mirhosseini *et al.*, 2008; Piorkowski and McClements, 2014). Due to this, this polysaccharide will have a higher contribution of negative electric charge, given that the pH values of the emulsions ( $5.935\pm0.212$ ) are above the pKa value (2.00-2.87) (Souza and Garcia-Rojas, 2017; Mirhosseini *et al.*, 2008; Niu *et al.*, 2017). Proteins are negative above the isoelectric point ( $pI = \sim 4$ ) (Thaiphanit *et al.*, 2016), which favored the values of potential- $\zeta$  being more negative.

The potential- $\zeta$  presented a variation between  $-39.1\pm2.8$  and  $-55.6\pm1.8$  mV, which favors the emulsion's thermodynamic stability, given that it denotes a good repulsive interaction or repulsive forces among colloid particles (Estevinho *et al.*, 2014; Mirhosseini *et al.*, 2008; Rezvani *et al.*, 2012). Coconut is rich in mineral salts, like potassium, sodium, phosphorus, iron, copper and chlorine (Appaiah *et al.*, 2015; Siriphanchit *et al.*, 2011; Tan *et al.*, 2014; Jayalekshmy *et al.*, 1986; Piorkowski and McClements, 2014), which when dissociating produce an important ionic force in the continuous phase, whose ions are strongly adsorbed in the particle interface, which-likewise-have ionizable groups of opposite charge, producing or resulting in a strongly adsorbed co-ion layer of negative charge on the particle interface. This situation also produces an electrical potential outside this adsorbed layer (Stern layer) associated to the zeta potential found (Malvern Instruments, 2004); besides contributing to the formation of a second co-ion diffuse layer distributed in the dissolution next to the interface. Both layers form the known ion double layer or Debye length ( $\kappa^{-1}$ ) (Jayme *et al.*, 1999; Piorkowski and McClements, 2014; Niu *et al.*, 2017).

The bibliographic review shows an important contribution of the electrostatic forces in stabilizing food colloidal systems:  $\approx 16$  mV in "homogenized coconut milk" without fiber and with active surface stabilizers added (Tangsuphoom and Coupland, 2009); between -17.1 and -13.8 mV in dressings with orange

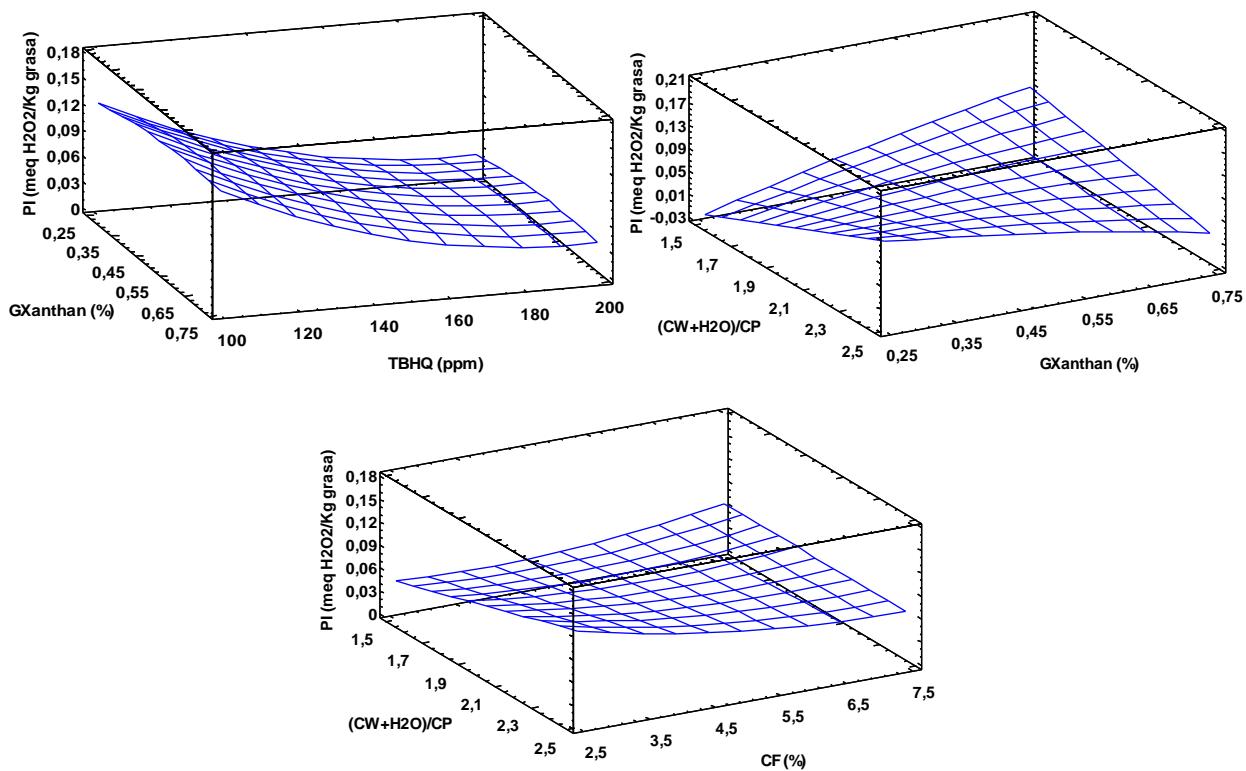


Fig. 5: Plots of PI surface response in function of the independent variables

pulp fiber (Chatsisvili *et al.*, 2012); between -54.0 and -35.0 mV in emulsions with chia oil (Julio *et al.*, 2015); and between -43.6 and -11.5 mV in emulsions of sunflower oil and sodium caseinate/sodium alginate mixes (Sosa-Herrera *et al.*, 2012). Mirhosseini *et al.* (2008) reported values between -27.0 and -30.0 for emulsified orange beverages, where the effect of the G<sub>Xanthan</sub> concentration favored the repulsive forces, as in this research.

**Emulsion oxidation:** Figure 5 presents the PI Surface response plots in function of the independent variables. The ANOVA showed that the PI had statistically significant differences ( $p<0.05$ ) with respect to G<sub>Xanthan</sub>, the (CW+H<sub>2</sub>O)/CP-fiber percentage interaction, as well as the quadratic effect of TBHQ. However, the changes presented were low, fluctuating between  $0.03\pm 0.03$  and  $0.18\pm 0.02$  meq H<sub>2</sub>O<sub>2</sub>/kg, indicating minimum oxidation on the fatty acids and surely as consequence of the imposed operation conditions: raw matter in excellent quality conditions (<30 after harvest), short homogenization times, low processing temperatures. It is worth highlighting the effect of TBHQ on PI, acting on its role as antioxidant upon contributing to its diminishing when its concentration increases. Within this context, the hydrophobic antioxidant action in the fat-water interface at high concentrations is more effective by reducing the oxidation of the oily phase (Kishk and Elsheshetawy, 2013).

The negative (CW+H<sub>2</sub>O)/CP-fiber percentage interaction increases PI when the emulsion has a high

content of aqueous phase and low fiber percentage added, a situation that favors its fluidization during homogenization, occluding greater air and generating smaller particle sizes in the oil drops that increases its surface area and favors oxidation and greater formation of hydro-peroxides. Further, PI presented a tendency to increase with increased G<sub>Xanthan</sub>, in the whole range of the (CW+H<sub>2</sub>O)/CP ratio, being likewise greater when the emulsion is more fluid. This effect could be attributed to the G<sub>Xanthan</sub> capacity to bind to Fe<sup>2+</sup> ions in the anionic sites of pyruvate along the polysaccharide chain. Consequently, the level of Fe<sup>2+</sup> free ions available to promote lipid oxidation would be reduced, but could be affected by other metals (Qiu *et al.*, 2015). This translates into the fiber's chelating capacity, which has *in vitro* cationic exchange capacity as mineral binding means; one of the possible consequences is that these ions can be kept from operating in the activation of the lipid oxidative reactions. It has been shown that some fibers have ionic exchange capacity with copper. Additionally, pectin can combine *in vitro* with bivalent ions, like iron, calcium, copper and zinc (Borderías *et al.*, 2005; Yalegama *et al.*, 2013; Qiu *et al.*, 2015).

**Mathematical modeling of the response surfaces:** Table 4 and 5 present the estimated regression coefficients of the polynomial surface response models with the corresponding R<sup>2</sup> values, lack-of-fit test and the experimental and theoretical or predicted values, besides the Relative Mean Error (RME).

Table 4: Regression coefficients,  $R^2$ , and probability values of the lack of fit of the models for the dependent variables

Regression coefficients	$a_w$	TS	$L^*$	$a^*$	$b^*$	R
$\beta_0$	0.998	-5.874	90.756	-7.749	9.797	1.084
$\beta_A$	-0.022	28.130	-17.583	11.579	-3.288	-0.028
$\beta_B$	-0.100	-24.517	1.256	5.054	-25.411	-0.069
$\beta_C$	-0.005	2.219	-1.527	-0.480	1.660	-0.016
$\beta_{Dx} \times 10^{-2}$	0.047	-3.913	-0.900	-1.917	1.680	-0.141
$\beta_{A^2}$	0.004	-7.618	3.229	-1.912	0.167	-0.022
$\beta_{B^2}$	-0.021	7.449	3.096	-7.646	6.846	0.119
$\beta_{C^2}x \times 10^{-2}$	0.028	8.462	-7.189	10.692	-7.980	0.050
$\beta_{D^2}x \times 10^{-4}$	-0.016	1.929	-3.111	1.761	1.104	0.038
$\beta_{AB}$	0.012	3.417	-2.988	-0.622	9.204	0.035
$\beta_{CWx} \times 10^{-2}$	0.237	-54.033	55.633	-30.800	-2.289	0.343
$\beta_{ADx} \times 10^{-2}$	-0.003	0.238	2.489	-1.279	-1.431	0.025
$\beta_{BC}$	0.002	0.737	-0.627	0.672	-0.479	0.004
$\beta_{BDx} \times 10^{-3}$	0.517	50.3	37.011	-2.222	9.956	-0.743
$\beta_{CDx} \times 10^{-3}$	-0.026	-7.883	5.474	1.282	-2.16	0.012
$R^2$	74.854	94.992	71.106	64.856	66.367	59.657
Lack of fit (p-value)	0.600	0.8641	0.5785	0.349	0.4751	0.207
Regression coefficients	$\mu$	$D_{10}$	$D_{50}$	$D_{90}$	$\zeta$	PI
$\beta_0$	2785.410	91.725	-109.118	-562.712	-70.217	-0.145
$\beta_A$	-2042.280	-55.417	-139.927	844.839	-14.274	0.374
$\beta_B$	-4894.760	-81.340	-3473.790	-2363.13	-115.877	1.073
$\beta_C$	173.046	7.565	117.114	-20.842	7.235	0.001
$\beta_{Dx} \times 10^{-2}$	-351.331	-43.114	1138.260	1300.440	62.597	-0.403
$\beta_{A^2}$	243.066	6.585	-14.060	-114.147	4.668	0.0003
$\beta_{B^2}$	-40.003	9.266	802.784	1237.630	82.405	-0.124
$\beta_{C^2}x \times 10^{-2}$	2976.260	11.248	-344.692	117.633	-46.218	0.204
$\beta_{D^2}x \times 10^{-4}$	376.399	5.683	-63.282	141.408	-12.088	0.1087
$\beta_{AB}$	2102.200	29.412	1182.400	912.778	17.611	-0.415
$\beta_{CWx} \times 10^{-2}$	-2552.670	-174.21	1360.070	2545.560	27.000	-1.527
$\beta_{ADx} \times 10^{-2}$	88.233	13.604	-296.559	-703.056	-8.539	-0.035
$\beta_{BC}$	234.707	-2.754	-5.183	-43.800	-2.869	-0.019
$\beta_{BDx} \times 10^{-3}$	7130.000	174.322	1215.570	-0.00529	1.000	0.013
$\beta_{CDx} \times 10^{-3}$	2351.400	-19.619	-488.393	20.3333	-11.344	0.103
$R^2$	96.400	69.367	67.625	67.0092	63.344	66.208
Lack of fit (p-value)	0.875	0.570	0.4963	0.485	0.000	0.486

Table 5: Experimental and theoretical or predicted values through the mathematical models of the dependent variables of the coconut emulsion

Variable	Theoretical or predicted			
	Experimental value	value	Difference	RME
$L^*$	67.5±0.7	66.4	-0.906	0.404
$a^*$	3.2±0.2	3.2	0.007	1485
$b^*$	8.6±0.5	7.9	0.771	2.451
R	0.851±0.025	0.858	-0.007	0.177
$\mu$	741.7±25.5	776.196	-34.464	0.454
$\bar{P}$	0.142±0.038	0.141	0.001	2.304
$D_{10}$	4.3±0.8	3.4	0.958	5.748
$D_{50}$	323.7±43.6	331.5	-7.791	2.962
$D_{90}$	743.0±65.1	626.9	116.164	4.084
Potential- $\zeta$	-45.6±2.5	-48.2	2.653	1.878
$a_w$	0.983±0.05	0.986	-0.003	0.044
TS	19.981±0.303	20.573	-0.592	0.269

RME: Relative mean error

Although the  $R^2$  values for the response variables were not sufficiently high, the lack-of-fit test permits determining the exactness of the model to predict the variation, where the high reliability and precision of the response data were confirmed by the RME values by being significantly lower than 10%. In addition, the results did not reveal statistically significant differences ( $p>0.05$ ) between the responses observed and those predicted, where the regression models agree with the values observed, demonstrating the precision and reliability of the empirical models that resulted significant ( $p>0.05$ ) in terms of the response variables

studied, which indicates that the models were adequate to describe data behavior.

The viscosity and TS variables are critical for optimization, presenting values of 0.964 and 0.95 that indicate that the response surface model explains 95 to 96% its variation in function of the emulsion's composition and homogenizing process.

**Emulsion optimization:** Numerical experimental optimization is carried out to obtain optimal values for independent variables, thus, determining the desired response parameters that permit achieving a final product with adequate quality attributes. For this case

and according to the statistical results found, the criteria were the following: Approximate viscosity of 1000 cP (condition of maximum viscosity permitted by the drier), higher TS percentage, higher L\*, lower enzymatic browning and lipid oxidation (< PI and < potential- $\zeta$ ). These response variables were significantly influenced by the G<sub>Xanthan</sub> and fiber percentage, being the variables that seek better emulsion behavior and where the antioxidant becomes a support factor that tends toward the good performance of the G<sub>Xanthan</sub> interaction and percentage of fiber.

The optimal conditions reached by the independent variables were the following: (CW+H<sub>2</sub>O)/CP = 2.0; G<sub>Xanthan</sub>= 0.5%; Fiber percentage= 5.0 and TBHQ = 200 mg/kg, with 10-min homogenizing time and controlling emulsification temperature to 30°C. Table 5 compares the dependent variables of the emulsion obtained from three replicates at the optimal condition and the values predicted by the mathematical model. Note that the values predicted by the models have approximately 95% acceptance level and can establish the required optimal preparation conditions for coconut emulsions and/or suspensions by adding its own fiber.

## CONCLUSION

The generation of value in the coconut agricultural chain represents a challenge from the research point of view, given that the matrix is quite complex due to its composition and mechanical properties. Development of a coconut based colloid formulation with its original fiber is possible by using statistical tools for its optimization, with the final emulsion obtained a potential and adequate base from the technical point of view, for its use in a future development of a product in coconut powder. The potential use of the fruit and the fiber present could represent a health benefit and a substantial economic boost for its agroindustry.

In general, the intermediate levels of the hydrocolloid and fiber added are those that seek better behavior of the emulsion. The (CW+H<sub>2</sub>O)/CP ratio, although influencing in some specific cases, is not very determining, while the antioxidant becomes a support factor that tends toward the good performance of the hydrocolloid and fiber interaction.

The surface response methodology permitted identifying the principal effects of the dependent variables and their interactions on the attributes of emulsion quality. In general, the components selected and the ranges evaluated of the dependent variables in the formulations were quite correct. A stable emulsion was obtained with important solid content due to the dairy serum action in its surfactant role (assuming a high contribution in reduced free energy in the interphase), the excellent negative electric potential favoring the repulsive forces of the particles and the effect of the hydrocolloid on the rheology of the continuous phase that restricts the mobility of the oily drops and suspended fibers. In addition, the excellent oxidative control was a result of the synergy between

the TBHQ action and the controlled preparation temperature.

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