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Optimization of Tiger Nut Milk Microencapsulation Process: Evaluation of Solubility and Oxidative Stability

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***Abstract*— *The appropriate concentrations of inulin and modified tiger nut starch were evauated in a sequential experimental design to improve the microencapsulation process of tiger nut milk. The stability emulsification index was the response variable studied to evaluate the effectiveness of the microencapsulation process. The concentrations observed through the central composite design (CCD) that most improved the stability emulsification index corresponded to 9.40% inulin and 0.40% modified tiger nut starch. The preservation capacity of the microencapsulated tiger nut milk was evaluated by determining the solubility in water (76%) and oxidative stability (46 h) of the microspheres. The chemical composition indicated important concentrations of protein (5.40%), calcium (191.65 mg / 100 g), carbohydrate (65.10%) and vitamin C (3.17%).*
Keywords*— *Microencapsulation; Optimization; Tiger nut starch; Tiger nut milk; oxidative stability, solubility.

I. INTRODUCTION

The "tiger nut milk" or "horchata" is a non-alcoholic beverage widely consumed in Spain and some African countries. In recent years,it has been expanding around the world. It has low acidity (6.3-6.8) and high nutritional quality, which increases its marketing potential. This product is rich in starch, oleic acid, linoleic acid, proteins and amino acids, mainly arginine [1-4]. It can be used as flavoring agents for ice creams, bakery products, biscuits, instant soups, and as an alternative source of extract for fermented products such as yoghurt, jams, jelly, beer and liquor [2,5]. Despite being an attractive product for commercializationthe fresh natural tiger nut milk may not be heated above 72 °Cdue to starch jellification, which limits its shelf-life[6]. One way to overcome this problem would be microencapsulation.

Microencapsulation is a technology that allows sensitive ingredients to be physically enveloped in a coating to protect them from adverse reactions, volatile loss or nutritional deterioration[7]In addition, microencapsulation can simplify food manufacturing processes by converting liquids to solid powders and decreaseproduction costs[8].Microencapsulation presents numerous applications in several segments of industry, such as the incorporation of hydrophobic substances into aqueous systems [9]; the masking of unpleasant taste and odor; increase the stability and shelf life.[10,11] Various materials may be used as "wall material" or encapsulating material to produce microspheres. Among these materials are those of natural or semisynthetic origin, such as carbohydrates, proteins,natural gums, esters of cellulose and some lipids[7]

In this work, the optimization of tiger nut milk microencapsulationwith inulin and modified tiger nut starch as wall material was carried out taking into account the oxidative stability and solubility of the microspheresproduced.

II. MATERIALS AND METHODS

2.1 Materials

All reagents and biopolymers used in this work were purchased by Sigma-Aldrich CO., MO, USA.

2.2 Tiger nut milk and tiger nut starch

Tiger nuts (*Cyperus esculentus*) with 0.5 to 1.0 cm diameter were obtained in the city of Morros, east of the State capital of Maranhão. The tuber was sanitized with 10% sodium hypochloritesolution (v/v) and then stored in a freezer (- 4° C). Tiger nut milk was obtained after a procedure optimized by COSTA NETO et al. [12]

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with 8 hours of soaking time, 22 °C soaking temperature, 0.20% sodium metabisulphite concentration and 1:1 water proportion. The extraction process was performed according to the procedure described by GURAYA et al.[13].

2.3 Chemical modification of the tiger nut starch.

The tiger nut starch was modified by the esterification of the hydroxyls of the starch with the octenyl succinic acid anhydrous (Sigma-Aldrich CO., MO, USA) in alkaline medium according to the procedure described by SONG et al. [14].

2.4- Microencapsulation of tiger nut milk

2.4.1Microencapsulation process

The microencapsulation process was carried out through lyophilization of tiger nut milkright after the preparation of this beverage and addition of the encapsulating materials in order to avoid any oxidation of its components and / or loss of its microbiological stability. The encapsulants were suspended in tiger nut milk (10 ml) and stirred for 10 min using a magnetic stirrer bar; then frozen in ultra-freezer at -40 °C for 24 h and lyophilized in L101 benchtop freeze-dryer for approximately 48 h. The sample outlet temperature was 25 °C at approximately 77 µmHg.

2.4.2Evaluation of encapsulating material

Thecombination of biopolymers tested for tiger nut milk microencapsulation was based in the study of SILVA et al. [15], as follows: 10% arabic gum / 8.5% maltodextrin (AG/MTD);0.6% xanthan gum / 1.0% maltodextrin (XG/MTD);10% inulin (IN) and 10% inulin / 0.5% modified tiger nut starch (IN/TNS). Emulsion index(E.I.) and pH of the reconstituted tiger nut milk(microencapsulated tiger nut milk solubilized in water) were used as parameters to chose the best combination. These parameters were also evaluated for the control, which was the tiger nutmilk lyophilized without any encapsulant.

2.4.3Optimization of microencapsulation

Central composite design (CCD) was used to study the individual and synergistic effect ofinulin concentration (Z1) and modified tiger nut starchconcentration (Z2) on microencapsulation process. The CCD was designed with two levels,fourfactorial points, fouraxial points and fivereplicates at the center (13 experiments), reported in Table 1. The response variable was the emusification index (E.I).

Table 1- Factors and levels studied through a rotational central composite design to evaluate the emulsification index of the microencapsulated tiger nut milk reconstituted in water.

Factors *	Levels			Axial point (α = 1,41)	
	-1	0	+1	- α	+ α
Z_1 (%)	5.00	7.50	10.0	3.96	11.03
Z_2 (%)	0.50	0.75	1.00	0.39	1.100

* Inulin concentration of inulin (Z1); modified tiger nut starch concentration (Z2)

2.5Analysis of the microencapsulated tiger nut milk

2.5.1 Analytical Methods

The physicochemical analyses of microencapsulated tiger nut milk composition were performed using the methods described in Official Methods of Analysis of AOAC International [16]. The parameters analyzed included: protein, total carbohydrate, lipid, moisture, ash, calcium, iron content, ascorbic acid, total sugar, reducing sugar and starch.

2.5.2Solubility

Solubility of the microcapsules was determined according to the modified methodology described by CANO-CHAUCA et al. [17]. For thisanalysis, 25 ml of distilled water was added to 0.25 g of each sample.The resulting suspension was centrifuged at 900*g* for 5 min. Twenty milliliters of supernatant was placed in a dry, empty Petri dish and taken to an air circulation oven at 105 ºC for 5h, after which was weighed. Empty and dry Petri dishes were weighed before the experiment. The solubility was calculated by the mass difference between the Petri dish with the dryed supernatant and the empty Petri dish and the results are expressed as a percentage of solubility.

2.5.3Emulsification index (E.I) and pH

For these analyses, 2.5 g of microencapsulated tiger nutmilk were reconstituted with 10 ml of distilled water in 15 ml test tubes under agitation (magnetic stirrer) for 10 min.

To evaluate the emulsion stabilityof the reconstituted microcapsules in water, emulsification index was determinedwith amodified methodology of FONTES

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et al. [18]. For this analysis, the reconstituted microencapsulated tiger nut milk was left to stand still for 24 h. After this period, the emulsionsizewas measured vertically. The E.I is given as a percentage of emulsified layer height (cm) divided by total height of the liquid column (cm).

The pH of the reconstituted microcapsules in water was determined with a pH meter (Digmed DM-22).

2.5.4Oxidative stabilitymilk

Oxidative stability of the microencapsulated tiger nut milkwas analysed according to Rancimat method [19]using Metrohm 743 model Rancimat (Herisau, Switzerland) instrument. Samples of 3 g were analysed under heating block temperature of 110 °C and constant air flow rate of 10 l/h for 70 h. Curves (condutivity *versus* time) obtained by the Rancimat method were evaluated by a graphical tangential method to calculate the induction period (IP), expressed in hours. All determinations were performed in duplicate and the mean value is reported.

III. RESULTS AND DISCUSSIONS

3.1 Evaluation of encapsulating material for microencapsulation

Preliminary tests were conductedto determine thebest encapsulating materials for tiger nut milk. The concentrations and encapsulants initially defined were based on the work of SILVA et al. [15] for oily samples. The encapsulants used were: 10% Arabic gum / 8.5% maltodextrin (AG/MTD); 0.6% xanthan gum / 1.0% maltodextrin (XG/MTD); 10% inulin (IN); and 10% inulin / 0.5% modified tiger nutstarch (IN/TNS).

After lyophilization the control sample (tiger nut milk without encapsulant) featured an oily aspect and a yellow coloration, probably due to oxidation of the lipidic fraction of tiger nut milk. Samples with encapsulant material were less oily and showed a reduced yellow intensity, evidencing that these materials are, in a way, protecting the core material. AG/MTD was the closer to the control sample in visual aspect, followed by XG/MTD and IN.

On the other hand, when inulin was used withmodifiedtiger nut milk (IN/TNS) a white color and absence of oily characteristicswere noted. Therefore, it seems that the encapsulant IN/TNSpresented better results when compared to the other encapsulants studied. However, other paremeters were measured to ensure this result.

The results obtained after the reconstitution of the microencapsulated tiger nut milkin water are displayed in Table 2.It is important to emphasize that the pH value in a range of 6.70 to 7.03 (95%) confirms the freshness of the tiger nutmilk[6]. It was possible to observe that arabic gum with maltodextrine fails in preservingthefreshness (pH < 6.70), oremulsion stability. Three well-defined phaseswere formed as well as a precipitate.

Table 2 – Parameters obtained for the reconstituted microencapsulated tiger nut milk in water.

Encapsulating	pH	Precipitate **	E.I ***
Control*	6.23	(+)	0.131
AG/MTD	6.43	(+)	0.136
XG/MTD	7.03	(+)	0.991
IN	6.80	(+)	0.850
IN/TNS	6.80	(-)	0.995

*No encapsulant material; AG/MTD = 10% Maltodextrin and 8.5% gum arabic; XG/MTD = 0.6% xanthan gum and 1.0% matodextrin; IN = 10% Inulin; IN/TNS = 10% Inulin and 0.5% modified tiger nut starch. ** (+) = present; (-) = absent. *** E.I = emulsification index.

Despite preserving the freshness (pH = 7.03) and having a high stability emulsification index (0.991), the reconstituted tiger nut milk with xanthan gum and maltodextrinpresented high viscosity, possibly due to the presence of xanthan gum. Inulin and inulin with modified tiger nut starch were good encapsulants to preserv freshness (6.70 ≤ pH ≤ 7.03) [6] maintain emulsion stability in comparison to the control. However, a slight precipitation was observed for IN, whichled to choosing IN/TNS for the next step as no precipitate was noted with this sample. It is possible that the hydrophobic fractionof the modified starch favored the micelle formation, whichinteractedwith the oily portion of the milk and allowed a higher emulsification index.

With these result acentral composite design (CCD) was designedto evaluate the optimum concentrations of inulin and modified tiger nut starch regarding the emulsification index (E.I).

3.2 Central Composite Design (CCD) to optimize inulin and modified tigernut starch concentrations

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The concentration of inulin (Z_1) and modified tiger nut starch (Z_2) weredefined as independent variables in a rotational central composite design (CCD) to obtain the highestemulsion stability (dependent variable) for the reconstituted encapsulated tiger nut milk in water.

According to Table 3, the emsulsification index ranged from 0.121 ± 0.03% to 0.998 ± 0.03% with a variance of 8.11%. It is important to emphasizethat the closer to the absolute value 1.00, the greater the emulsion stability. The variance for the response variable was 0.168% at the central point, indicating a significant repeatability of the experiments since it hada value lower than 10%, as described by BOX et al. [20].

Table 3– Response variables studied by central composite design to evaluate the emulsification index of the microencapsulated reconstituted tiger nut milk.

Run	Z_1*	Z_2 *	E.I (%) **
1	-1(5.000)	-1(0.500)	0.322
2	+1(10.00)	-1(0.500)	0.720
3	-1(5.000)	+1(1.000)	0.121
4	+1(10.00)	+1(1.000)	0.875
5	-1.41(3.964)	0(0.7500)	0.122
6	+1.41(11.03)	0(0.7500)	0.891
7	0(7.500)	-1.41(3.964)	0.998
8	0(7.500)	+1.41(11.03)	0.861
9	0(7.500)	0(0.7500)	0.676
10	0(7.500)	0(0.7500)	0.750
11	0(7.500)	0(0.7500)	0.710
12	0(7.500)	0(0.7500)	0.750
13	0(7.500)	0(0.7500)	0.710

* concentration of inulin (Z_1); concentration of modified tiger nut starch (Z_2); emsulsification index (E.I)

The pareto chart analysis(Figure 1) showed that the independent variables (Z_1 and Z_2) and itslinear interaction were statistically significant at a significance level of 5% ($p < 0.05$). Only the linear term of the variable Z_2 was not statistically significant ($p > 0.05$).

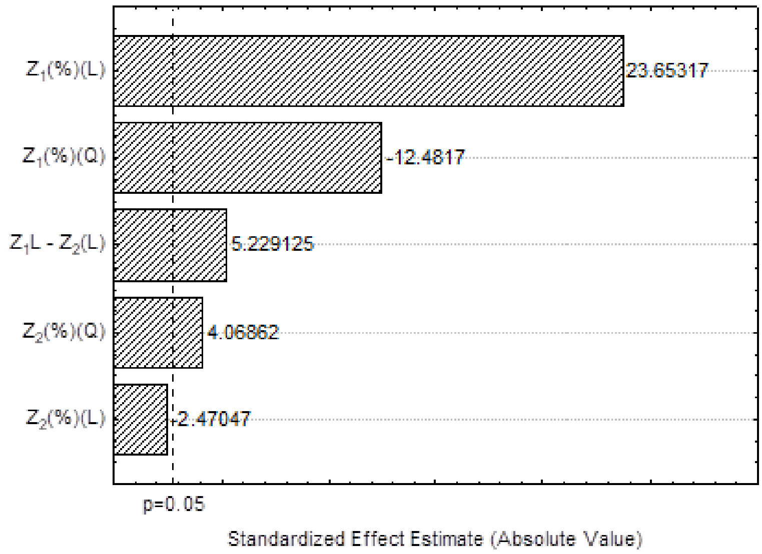

Fig.1 - Central composite design Pareto chart for the emulsification index of the reconstituted microencapsulated tiger nut milk. Concentration of inulin (Z_1) and concentration of modified tiger nut starch (Z_2).

The independent variable that most influenced the response was the linear term of the concentration of inulin (Z_1) (L) with a positive effect. The increase in inulin concentration increases the emulsion stability of the reconstituted beverage. The same behavior was observed for the linear interaction between both independent variables (Z_1 and Z_2) (L-L), emphasizing the synergistic effect of this interaction on the stability of the

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reconstituted milk. However, it is important to emphasizethat concentrations greater than 10% of inulin (Z_1) and 1.0% of modified tiger nut starch (Z_2) led tothe formation of a biphasic emulsion due to the appearance of a precipitate, which is undesirable for the commercialization of the product. Therefore, it was not possible to evaluate the emulsification index behavior regardinginulin and modified tiger nut starch concentrations at the values indicated in this CCD.

Table 4 summarizes the analysis of variance (ANOVA) performed in order to optimize the best concentrations of inulin and modified tigernut starch in relation to the emulsification index. With those results, an adjusted predictive model ($p < 0.05$) was determined.

Table 4- Analysis of variance (ANOVA) from the CCD of the model adjusted for the emulsification index of the reconstituted microencapsulated tiger nut milk ($R^2 = 0.900$).

*

Factor*	SS[a]	DF[b]	M.S[c]	F_{-value}[d]	P_{-value}
$1 - Z_1$ (L)	0.626609	1	0.626609	559.4723	**0.000019**
Z_1 (Q)	0.174488	1	0.174488	155.7928	**0.000237**
$2 - Z_2$ (L)	0.006836	1	0.006836	6.1032	0.068912
Z_2(Q)	0.018540	1	0.018540	16.5537	**0.015240**
$Z_1L - Z_2L$	0.030625	1	0.030625	27.3438	**0.006388**
Lack of fit	0.088818	3	0.029606	26.4339	**0.004253**
Pure error	0.004480	4	0.001120		
Total SS	0.968831	12			

Concentration of inulin (Z_1), Concentration of modified tiger nut starch (Z_2); [a]SS: quadratic sum; [b]DF: degrees of freedom; [c]M.S: mean square; [d]Test to compare the model variance with the residual variance (error).

Table 4 results demonstrated a goodcoefficient of determination for the emulsification index (R^2 = 90.0%). This value demonstrates a possible significance of the model since itwas unable to explain only 10.0% of the total variations (100%).However, it can be observed that the lack of fit was significant ($p < 0.05$). According to Rodrigues and Iemma [21], the lack of fit is not important in the development of a predictive model when the central point of the associated pure error has alow variability. In order toprove this, Rodrigues and Iemma [21] calculated the error of fit and relative error presented in Equations 1 and 2 regarding the interactionbetween the experimental response and the predicted response ofthe model. These terms are numerical representations of the plotpredicted values versus experimental values.

$$Error of fit = Y - \hat{Y} \quad (1)$$

$$Relative error = \left(\frac{Y - \hat{Y}}{Y}\right) \times 100 \quad (2)$$

Where: Y = experimental response;
$\hat{Y}$ = model response.

Analysingthe results obtained in Table 4 for the emulsification index (E.I, response variable), it can beobserved that the pure error of the model (0.001) was very low, tending to zero. It satisfies the prediction and optimization condition of a significant lack of fit, described by Rodrigues and Iemma [21]. Therefore, it was possible to obtain the model (Equation 3) from real variables based on the ANOVA and the Regression coefficient (0.90).

$$E.I = -0.214 + 0.391Z_1 - 0.026Z_1^2 - 2.41Z_2 + 0.142Z_1.Z_2 \quad (3)$$

After the development of the mathematical model for the emulsification index of the reconstituted tiger nut milk encapsulated by the inulin and modified tiger nut starch, the possibility of prediction and optimization of this model was confirmed bythe results obtained from the error of fit (Equation 1) and relative error (Equation 2). The results are summarized in Table 5.

Table 5- Emulsification index values of the reconstituted microencapsulated tiger nut milk predicted by the model and deviations of the CCD.

Run	Z_1*	Z_2*	E.I (%)	E.I pred.	Error of fit	Relative error (%)
1	-1(5.000)	-1(0.500)	0.322	0.452	-0.131	**-40.543**
2	+1(10.00)	-1(0.500)	0.720	0.835	-0.114	-15.860
3	-1(5.000)	+1(1.000)	0.121	0.215	-0.094	**-77.300**
4	+1(10.00)	+1(1.000)	0.875	0.872	0.126	12.577
5	-1.41(3.964)	0(0.750)	0.122	0.005	0.117	**95.989**
6	+1.41(11.03)	0(0.750)	0.891	0.799	0.092	10.351
7	0(7.500)	-1.41(3.964)	0.998	0.872	0.126	12.577
8	0(7.500)	+1.41(11.03)	0.861	0.782	0.080	9.255
9	0(7.500)	0(0.750)	0.676	0.782	0.080	9.255
10	0(7.500)	0(0.750)	0.711	0.723	0.034	4.539
11	0(7.500)	0(0.750)	0.758	0.723	-0.012	-1.755
12	0(7.500)	0(0.750)	0.758	0.723	0.034	4.539
13	0(7.500)	0(0.750)	0.715	0.723	-0.012	-1.755

* Concentration of inulin (Z_1); Concentration of modified tiger nut starch (Z_2); emulsification index (E.I); Experimental emulsification index (E.I); Predicted emulsification index (E.I pred.)

Table 5 demonstrates that the relative errors were low in the desired region where the emulsification index is maximized.Relative errors inferior to 20% are considered satisfactory, according to Rodrigues and Iemma [21]. This result corroborates with the very low value of the pure error (0.001), confirming the possibility of developing a predictive model to optimize the emulsification index even if it presented a significant lack of fit ($p < 0.05$). However, runs 1, 3 and 5 had higher relative errors, respectively -40.543%, -77.300% and 95.989%, which presented lower emulsification index values: 0.322, 0.121 and 0.122 respectively. This proves that the model did not adjust to concentrations equal to or lower than 5.0% of inulin but the prediction and optimization capacity of the model was not affected[21]. In addition, the experimental conditionof 3.964% (α = -1.41) of inulin (run 5) was the most critical, confirming that the microencapsulated tiger nut milk did not show an emulsion stability in low concentrations of inulin. Moreover, it is possible to confirm that the response variable inulin concentration was the main factor in the CCD due to its greatest effect already observed in the Figure 1.

Therefore, the mathematical model was used to optimize the concentrations of inulin and modified starch and to obtain the response surface,represented in Figure 2.

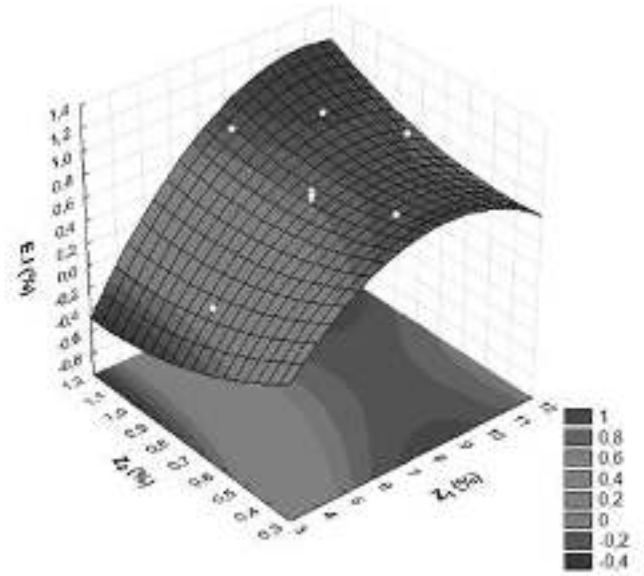

Fig.2 - Response surface for the emulsification index (E.I) of the reconstituted microencapsulated tiger nut milk with inulin (Z_1) and modified tiger nut starch (Z_2).

It can be observed that the stability of tiger nut milk increase with low concentrations of modified tiger nut starch (Z_2) and higher concentrations of inulin (Z_1). The optimum concentrations of inulin (Z_1) and modified tiger nut starch (Z_2) were 9.40% and 0.40%, respectively.

3.3 Chemical composition of microencapsulated tiger nut milk

After the microencapsulation process with the blend of inulin (9.40%) and modified tiger nut starch

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(0.40%), the microencapsulated tiger nut milk showed a tendency towards a white color with a sandy aspectand high solubility in water. The reconstituted microencapsulated milk also presenteda tendency for white coloration, as the fresh tiger nut milk.The chemical composition of the microencapsulated tiger nut milkis displayed in Table 6.

Table 6 - Chemical composition of the microencapsulated tiger nut milk with 9.4% of inulin and 0.4% of modified tiger nut starch.

Compounds	Microencapsulated tiger nut milk (%)
Starch	30.20±0.06
Reducing sugar	16.80±0.04
Total sugar	33.60±0.03
Moisture	4.70±0.03
Ash	1.10±0.05
Lipid	23.70±0.02
Protein	5.40±0.01
Total carbohydrate	65.10±0.04
Iron *	1.84±0.02
calcium *	191.65±0.03
Vitamin C *	3.17±0.05

* Values expressed in mg/100g

The microencapsulated tiger nut milkwas composed mainly of starch, total sugar, lipid, calcium and carbohydrate. This is associated with the biological origin of the tiger nut (tuber) [5,22,23]and the possible influence of the encapsulants used in the microencapsulation process of this milk.The moisture content is in agreement with products of this nature.

The presence of inulin as microencapsulationmaterial favoredthe beverage food functionality, increasing its potential in the food industry mainly for sports and therapeutic purposes[5,6]

3.4 Solubility of the microencapsulated tiger nut milk

Figure 3 shows no significant variation ($p = 0.073$) (t test) in the solubility of thetiger nut milk microspheresover 60 days. The solubility during this period ranged between73.0±0.15% and76.3±0.10%. This demonstrated an evident stability of tiger nutmilk microsphere solubility over the studied period. In addition, it is important to note that the freeze-drying process (-50 °C) (lyophilization) did not affect the solubility of these microspheres. This result suggests that the microencapsulation process using the blend of inulin andmodified tigernut starch occurred satisfactorily.

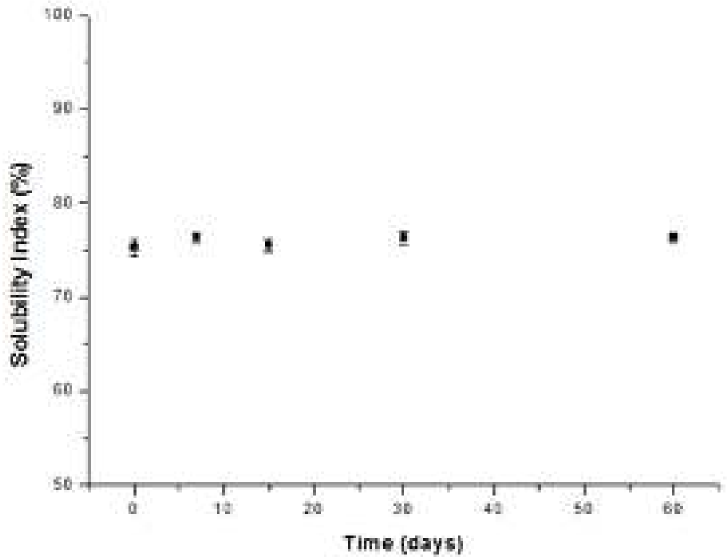

Fig.3 - Solubility of the tiger nut milk microencapsulated with 9.4% of inulin and 0.4% of modified tiger nut starch over 60 days.

Moreover, it is important to note that the solubility value obtainedfor these microspheresobtainedwithinulin (9.40%) and modified tiger nutstarch (0.40%) was similar to the results obtained by LACERDAet al.[24]. The researchers found solubilitiesof 76.8 to 85.0% for jussara pulp microsphere using inulin and matodextrin as encapsulants via spray drying.

Other similar results were obtained by FAZAELI et al. [25] using arabic gum and matodextrin (77-85% of solubility) as encapsulants; bySANTOS et al. [26] using arabic gum and porous granules of rice starch and gelatin (77.01-78.33% of solubility); byCASTRO et al. [27] using cashew gum and arabic gum (76.22%). Furthermore, microencapsulation processes using arabic gum presented higher values of solubilities due to their branched structure, which causes their structural folding and allow rapid reconstitution of the microencapsulated in water [27].

3.5 Oxidative stability (volatile acids) of the microencapsulated tiger nut milk

For the determination of the oxidative stability of the tiger nut milkmicrospheres obtained with 9.4% of inulin and 0.4% of modified tiger nut starch, the conductometry technique was used, which is based on the record of the conductivity variations of the distilled water from the collection of low molecular weight acids obtained after forced oxidation of the sample [28].

The oxidative stability presentedin Figures 4(a) and 4(b), shows that the microsphere undergoes oxidation from the induction time of 46 hours at 15 µS.cm^{-1}.

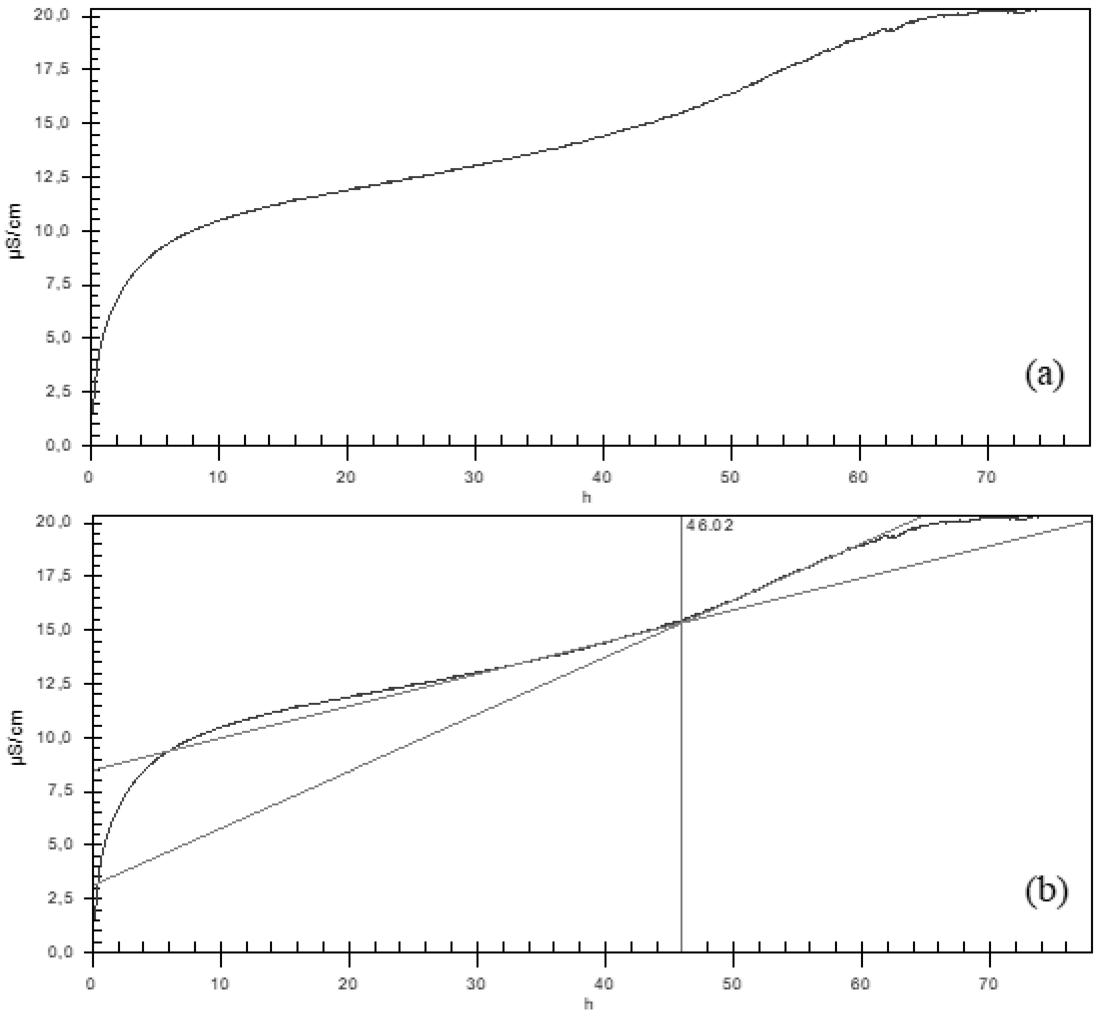

Fig.4 - Oxidative stability of microencapsulated tiger nut milk with 9.4% of inulin and 0.4% of modified tiger nut starch: (a) evolution of the oxidation process; (b) determination of the time in which the microspheres underwent the oxidation process.
* The Curve refers to the oxidation process; ** Cross-sectional diagonal lines refer to the point of intersection which comprises oxidation of the microspheres; *** Vertical line refers to the induction time for lipid oxidation of the microspheres

The induction time of 46 hours for lipid oxidation of the microsphere indicated that the encapsulation using inulin (9.40%) and modified tiger nut starch(0.40%) was able to protect the active material from oxidation for 46 hours. After this time, the microspheres were completely oxidized.

It should be emphasized that oleic acid is the most abundant oil in tiger nut[29].Soybean milk, which is also rich in oleic acid, has an induction time of 5.2 hours to undergo the oxidation process [30].Therefore, it shows that the encapsulation process was of greatimportance to maintainthe nutritional quality of the tiger nut milk.

IV. CONCLUSION

The blend of inulin and modified tiger nut starch was more suitable for the microencapsulation process of tiger nut milk. The best concentrations of inulin and modified tiger nut starch to microencapsulate tiger nut milk via lyophilization were: 9.40 and 0.40 % respectively.

The microencapsulated tiger nut milkpresented prebiotic characteristics as a function of the insertion of inulin as an encapsulant. This was important sinceit amplified the intrinsic nutritional potential of this milk. Microencapsulation was paramount for the oxidative processes to occur slowly and to maintain an excellentsolubility during a 60–daystorage.

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