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Palm Oil Carotenoids Extraction: Preparation Process Optimization

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Carotenoids from palm oil were recovered through a process involving neutralization and transesterification of palm oil followed by molecular distillation (Maciel et.al., 2006).

Most of the time, the raw material preparation process is necessary before the molecular distillation process and in fact, may play an important role in the process performance. Bearing this in mind, in this work is proposed a pre-treatment procedure and its optimization is persuaded. Crude palm oil can contain solids of the extraction oil process. The soap is also other pollutant that can be present in the raw material and must be treated. Neutralization reaction with sodium hydroxide (NaOH) was used to decrease the acidity and to produce soap. Centrifugation was used to separate the soap from the oil.

Neutralization was optimized by a factorial design to find the optimal quantities of sodium hydroxide (NaOH), temperature and reaction time to the maximum to lower acidity without damaging the amount of β-carotene in the sample. The amounts of sodium hydroxide ranges between 1,2078 g to 4,28 g used by 200 g of crude oil, the temperature between 43,2 to 76,8 °C and the reaction time between 6,6 to 23,4 minutes. Next step is the transesterification of the neutral palm oil. The objective of this reaction is transforming the glycerides in ethyl esters.

Neutralization was optimized by a factorial design and superficial analysis response. The experimental results are presented in this work.

Introduction

Crude palm oil has a rich-orange color due to its high content of carotenes (approx. 7 500-1000 ppm), making it one of the richest natural sources of carotenoids. Carotenoids are the precursors of Vitamin A, with β carotene having the highest provitamin A activity. Palm oil has 15 times more "vitamin A" activity than carrots and 300 times more than tomatoes.

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Palm oil is extracted from the mesocarp of the oil palm fruit. The palm tree *Elaeis guineensis is* originated in West Africa and probably is the most common raw material for crude palm oil production (Choo et. al., 1992).
Crude palm oil contains approximately 1% of: carotenoids, vitamin E (tocopherols and tocotrienols), sterols, phospholipids, glycolipids, terpenic and aliphatic hydrocarbons, and other trace impurities. Because of the important physiological properties, carotenoids and vitamin E are most important component.

1.1 Neutralization

Carotene extraction includes a preparation of crude oil. The preparation of the sample is essential step to guarantee an efficient extraction of β carotene. The crude palm oil contain a lot of contaminant. First of all, the oil was submitted a neutralization reaction to reduce its acidity and separate the soap by centrifugation. Figure 1 presents the schematic process of neutralization.

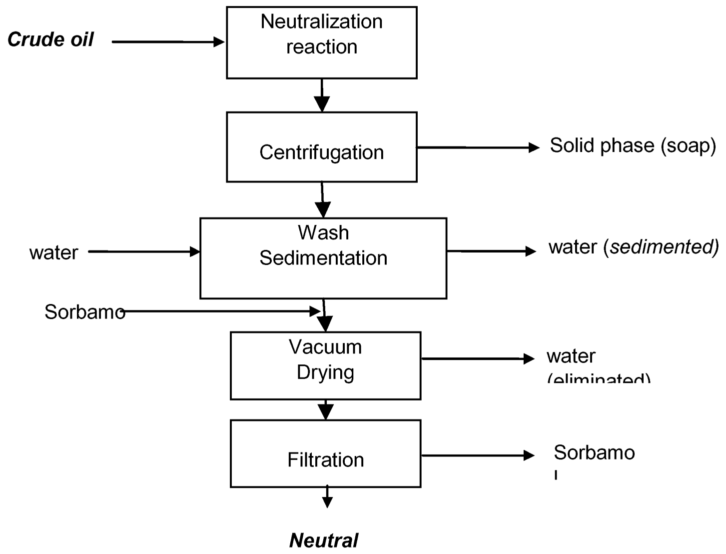


Figure1: Palm oil neutralization.

Due to the importance of the preparation process it was submitted an experimental design for optimize this process by a surface analysis response.

1.2 Experimental Design

The experimental design is a punctual analysis of limited set of variables (Boss et. al, 2004). This method is advantageous in relation to the conventional method of manipulation of a single parameter per trial, as such an approach frequently fails to locate optimal conditions for the process due to its failure to consider the effect of possible interactions between factors; moreover, the factorial design makes it possible to take advantage of practical knowledge about process during the final response surface analysis (Kalil et. al, 2000). The present work proposes the preparation process optimization using experimental design methodology through the surface analysis response.

Initially was used a full factorial design $2^3 = 8$ trials adding three central points but the generated model does not fit well with the experimental data, then was used a star experimental design with 17 trials involving the axial points of the independent variables, which fits the experimental data and to analyze more accurately the value of the acidity and loss of beta-carotene (response variables)

2. Material and Method

Brazilian crude palm oil was used as raw material in all experiments.
The variables selected for each response were the following: temperature (T), reaction time ($t_{reaction}$) and quantity of sodium hydroxide (NaOH) . The responses analyzed were the acidity and the loss of β carotene. Table 1 shows the variable level of the experimental design.

Table 1: Variable level for the acidity and loss of β carotene.

Variables	Level -1,68	Level -1	Level 0	Level +1	Level +1,68
T (°C)	43,2	50	60	70	76,8
$t_{reaction}$ (minute)	6,6	10	15	20	23,4
NaOH (g)*	1,2078	1,83	2,745	3,66	4,28

* Amount of NaOH used by 200 g of crude oil

The central point was chosen based on preliminary experiments that showed satisfactory results when you work with these values.

3. RESULTADOS

Table 2 outlines the experimental design and the results obtained for the acidity and the loss of β carotene.

Table 2: Experimental design and the results.

Trial	**T**	**NaOH**	$t_{reaction}$	**acidity (%)**	**β carotene loss(ppm)**
1	-1	-1	-1	0,76	89,20
2	1	-1	-1	0,61	114,70
3	-1	1	-1	0,22	94,51
4	1	1	-1	0,23	88,80
5	-1	-1	1	0,32	116,40
6	1	-1	1	0,38	132,89
7	-1	1	1	0,19	107,95
8	1	1	1	0,22	187,88
9	0	0	0	0,16	161,43
10	0	0	0	0,16	174,88
11	0	0	0	0,16	167,77
12	-1,68	0	0	0,16	77,27
13	1,68	0	0	0,19	105,03
14	0	-1,68	0	0,16	105,10
15	0	1,68	0	0,16	130,59
16	0	0	-1,68	0,19	148,55
17	0	0	1,68	0,22	193,78

Interactions can be observed to variables levels that produce low acid and low loss of beta carotene, which is wanted in this work.

The effects of the variables on each response are presented in Table 3 and 4 for a 90% of confidence level for codified values.

Table 3: Effects estimates on acidity.(%).

	Effect	Pure Error	t(2)	p
Mean/Interaction	0,1476	0,0002	610,1960	0,0000
(1)T (L)	0,0012	0,0002	5,0792	0,0366
T (Q)	0,0939	0,0003	374,7415	0,0000
(2)NaOH (L)	-0,1754	0,0002	-771,4438	0,0000
NaOH (Q)	0,0823	0,0003	328,6436	0,0000
(3)$t_{Reaction}$ (L)	-0,0958	0,0002	-421,2600	0,0000
$t_{Reaction}$ (Q)	0,1162	0,0003	464,0611	0,0000
1L by 2L	0,0340	0,0003	114,5614	0,0001
1L by 3L	0,0584	0,0003	196,5683	0,0000
2L by 3L	0,1562	0,0003	526,0010	0,0000

Table 4: Effects estimates on loss of β carotene (ppm)

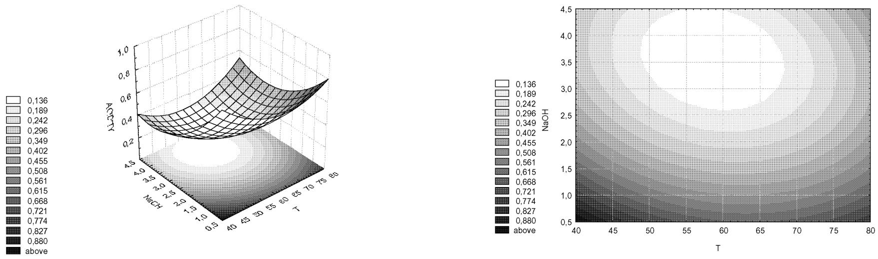
	Effect	Pure Error	t(2)	p
Mean/Interaction	168,5322*	3,8759*	43,4817*	0,0005*
(1)T (L)	23,8693*	3,6425*	6,5531*	0,0225*
T (Q)	-57,8579*	4,0128*	-14,4182*	0,0048*
(2)NaOH (L)	10,0821	3,6425	2,7679	0,1095
NaOH (Q)	-38,9439*	4,0128*	-9,7048*	0,0105*
(3)$t_{Reaction}$ (L)	34,2815*	3,6425*	9,4116*	0,0111*
$t_{Reaction}$ (Q)	-1,1580	4,0128	-0,2886	0,8001
1L by 2L	8,0551	4,7570	1,6933	0,2325
1L by 3L	19,1544*	4,7570*	4,0265*	0,0565*
2L by 3L	16,7842*	4,7570*	3,5283*	0,0718*

* Significant for a 90% confidence level.

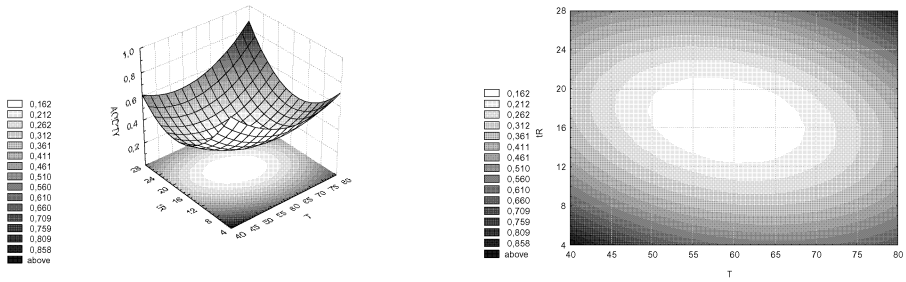
As can be seen in the column of effects from Table 3, an increase of the quantity of NaOH as well as in the reaction time decrease the acidity of the oil. The temperature and the reaction time almost not produce effect on the acidity of the oil. The table 4 shows how the loss of β carotene is directly related with the temperature reaction. As larger the temperature of the reaction larger the loss of β carotene and show too that as larger the reaction time larger the loss of β carotene.

Figures 2 presents the response surface and the contour diagrams of acidity as a function of NaOH and T (a), $t_{Reaction}$ and T (b).

Figures 3 present the response surface and the contour diagrams of loss of β carotene as a function of NaOH and T (a), $t_{Reaction}$ and T (b).

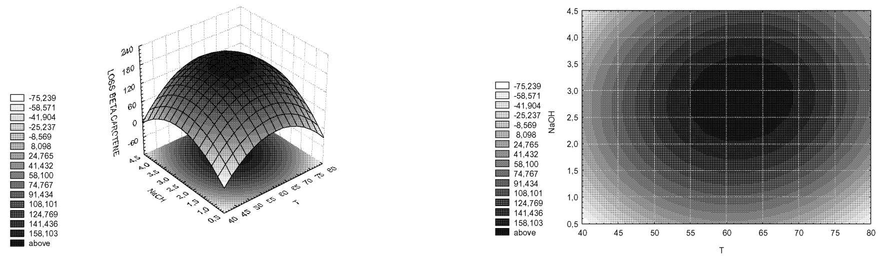


(a)

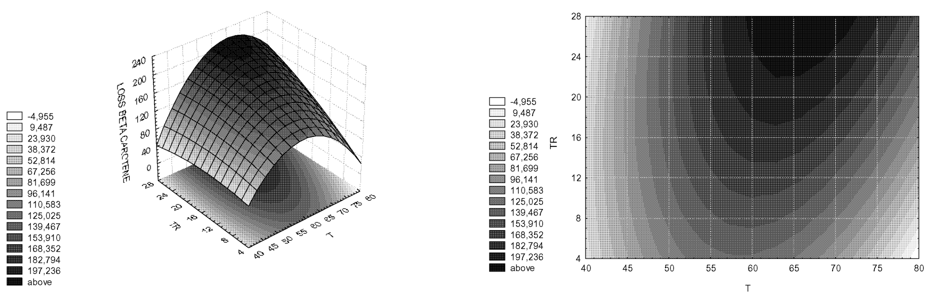


(b)

Figure 2: Response surface and the contour diagrams of acidity as a function of: NaOH and T (a), $t_{Reaction}$ and T (b). BETA=β



(a)



(b)

Figure 3: Response surface and the contour diagrams of loss of β carotene as a function of: NaOH and T (a), $t_{Reaction}$ and T (b). BETA=β

As can be seen in Figure 2, to obtain a product with acidity below 0,3%, is necessary a reaction time above 15 minutes and more than 2,745 g of NaOH (Amount of NaOH used by 200 g of crude oil). Temperature values should be in the range: 45- 72 ^{o}C. Figure 3 depicts that the temperature of reaction needs to be below 45 ^{o}C to reduce the loss of β carotene on the neutralization. Above 50 ^{o}C as larger the reaction time larger the loss of β carotene.

4. Conclusion

For an efficient neutralization of crude palm oil so that is may lead to reach an acidity below 0,3% is indispensable to use a suitable amount of NaOH: 2,745g **(**Amount of NaOH used by 200 g of crude oil). The minimum reaction time needs to be above 15 minutes.

The loss of β carotene in the neutralization reaction is directly related to the reaction temperature. As smaller the temperature of reaction smaller the loss of this component.

Taking into account economy versus quality, the optimization of the crude palm oil neutralization has as values of the operational variables: NaOH above central point, temperature in the inferior axial point (43,2 ^{o} C) and reaction time of 15 minutes. In such conditions it is possible to obtain an oil with less acidity than 0,3% without significant losses of β carotene.

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