

## A new hypothesis concerning continuous distillation with stripping gas and its application in the physical refining of edible oils

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### RESUMEN

**Una nueva hipótesis sobre la destilación en continuo con arrastre de vapor. Aplicación a la refinación física de aceites comestibles.**

Se estudia la influencia de la concentración en ácidos grasos libres en el gas de arrastre en la cabeza del desodorizador durante la refinación física en continuo, y se establece una hipótesis para explicar los resultados: En un proceso continuo de desacidificación por destilación de ácidos grasos a baja presión (2 - 3 mbar), alta temperatura (180 - 265°C), con arrastre de vapor, con igual temperatura para los gases en la cabeza del desodorizador y para el aceite, aplicado a aceites con una acidez inicial  $\leq 7,4$  % peso/peso, en el equilibrio se cumple que la relación entre la concentración en ácidos grasos libres en el aceite desodorizado y la concentración en ácidos grasos libres en el gas situado en la cabeza del desodorizador es constante. Esta hipótesis se somete a discusión.

**PALABRAS CLAVE:** Aceites comestibles – Proceso continuo de destilación con arrastre de vapor – Refinación física.

### SUMMARY

**A new hypothesis about continuous distillation with stripping gas and its application the physical refining of edible oils.**

The influence of the free fatty acid concentration in the gas inside the continuous deodorizer in continuous physical refining was studied and a hypothesis was formed to explain the results: In a continuous process of deacidification by distillation of free fatty acids at low pressure (2 - 3 mbar), high temperature (180 - 260 °C), with stripping gas, initial free fatty acid content of oil  $\leq 7.4\%$  w/w and similar temperature for both the gas distillate inside the continuous deodorizer and the oil, in the equilibrium the ratio between the free fatty acid content of the deacidified oil and the concentration of free fatty acids in the gas inside the continuous deodorizer is constant. This hypothesis is submitted to discussion.

**KEY-WORDS:** Continuous distillation with stripping gas – Edible oils – Physical refining.

### 1. INTRODUCTION

The oil temperature is an important operational parameter in industrial deacidification (physical refining), affecting the final free fatty acid content in

the deacidified (refined) oil, the flow of stripping gas required for deodorization, the time required to reach one's goal (Graciani *et al.* 1994, Ruiz-Méndez 1996) and the final quality of the oil (the content in trans fatty acids, etc) (León *et al.* 2001, 2003, 2004, Graciani *et al.* 2006, 2006a, Tubaileh *et al.* 2002a, 2002b). The vapor temperature inside the industrial deodorizer decreases substantially from the oil temperature and the condensation of fatty acids (known as reflux) occurs. This is a common phenomenon in industrial refining.

The effect of equal temperature of the gas distillates inside the deodorizer and the oil, was initially demonstrated by Deffense E (Deffense 1994) in the discontinuous deacidification in the laboratory. The effects and benefits of controlling the gas distillate temperature inside the discontinuous and/or continuous deodorizer during the oil deacidification (physical refining) has been discussed by Prieto *et al.* (Prieto *et al.* 1999, 2007a, 2007b, 2008a) and Bada *et al.* (Bada *et al.* 2000). Prieto *et al.*, in their original paper entitled "Optimization of deacidification of mixtures of sunflower oil and oleic acid in a continuous process" have demonstrated that "The results showed that the oil temperature and maintaining the same temperature in the oil and the gas distillates produced the most relevant effects, having a positive effect on most of the responses" (Prieto *et al.* 2007b). These effects are on the final free fatty acid (on the refining oil), on the rate of free fatty acid loss (Prieto *et al.* 2007b) and the final quality of the oil (León *et al.* 2001, 2003, 2004, Graciani *et al.* 2006, 2006a, Tubaileh *et al.* 2002a, 2002b) and not on additional losses of sterols in the refining oil (2007a). However, a great increasing of the temperature from the gas distillates inside the deodorizer above that of the oil sometimes produces a small negative effect on deacidification (2007a). Heating the stripping gas above the oil inside the deodorizer does not incur a high energy cost, because the specific head of the gas is very low and the flow rate is relatively small.

The authors of this paper, in physical refining, always recommend operating with equal temperatures for the gas distillates inside the

deodorizer and the oil. This working criterion has lead to our research group to apply for a patent for the removal the washes during deacidification (physical refining) of edible oils (Bada *et al.* 2008).

We therefore propose that the free fatty acid concentration in the gas inside the deodorizer in continuous physical refining is a dependent variable of the deacidification process.

The free fatty acid concentration in the gas of the deodorized process depends of the vapor pressure of the fatty acids (Bada *et al.* 1994, Defense 1994, Prieto *et al.* 2007a, Vian *et al.* 1964). So the variable depends on the operating temperature and absolute pressure. The new variable also depends on the deodorizer used.

In the present paper, the relationship between the free fatty acid concentration in the gas inside the continuous deodorizer and the free fatty acid concentration in the oil inside the deodorizer is studied and a hypothesis is established to explain the results:

In a process of deacidification by distillation of free fatty acids at low pressure, high temperature, with stripping gas and similar temperature for both, the gas distillate inside the continuous deodorizer and the oil, in the equilibrium, the ratio between the free fatty acid content of the deacidified oil and the concentration of free fatty acid in the gas inside the continuous deodorizer is constant at a constant temperature.

The free fatty acid content of the deacidified oil also depends on the operating temperature, the absolute pressure, the stripping gas flow and the deodorizer used (Bada *et al.* 1994, Defense 1994, Prieto *et al.* 2007a, Vian *et al.* 1964).

An application of this hypothesis consists of considering the deacidification by distillation of free fatty acids in continuous as a hypothetical extraction of a component (the free fatty acids) between two fluids (oil and stripping gas) and governed by the extraction coefficient. The extraction coefficient is dependent on the solubility component of the two fluids. Therefore it depends on the variables of operation: temperature, absolute pressure and the deodorizer used.

This work was carried out in a continuous deodorizer as described by Prieto *et al.* (2007a, 2007b, 2008), and an adjustment to these constants is made with experimental data (constant, temperature and absolute pressure). So their application to other apparatus for physical refining involves a certain degree of extrapolation.

## 2. MATERIALS AND METHODS

The study is based on the results of 48 assays previously developed and their results reported (Prieto *et al.*, 2007a, 2007b, 2008). The deodorizer and the operating conditions for the deacidification (physical refining) trials are shown in Table 1.

Gas free fatty acid concentration inside the continuous deodorizer ( $GFFA_m$ , mol/mol<sup>-1</sup>) was calculated as follows:

The free fatty acids distilled in g h<sup>-1</sup> for one hour are:

$$10 * (I_oFFA - D_oFFA) * F_o * 0.9$$

where  $I_oFFA$  and  $D_oFFA$  are the free fatty acid contents (% w/w) of the initial and of the deacidified oil, respectively;  $F_o$  is the flow of oil (l h<sup>-1</sup>) and 0.9 is, approximately, the oil density (g cm<sup>-3</sup>).

The equation in mol h<sup>-1</sup> would be:

$$10 * (I_oFFA - D_oFFA) * F_o * 0.9 / 282 \text{ mol h}^{-1}.$$

(282 g as average molecular weight of the free fatty acids)

The concentration of free fatty acids into the gas inside the continuous deodorizer ( $GFFA_m$ ) was calculated by the ratio between the number of mol h<sup>-1</sup> of free fatty acids distilled and the number of mol h<sup>-1</sup> of stripping gas

$$GFFA_m = 10 * (I_oFFA - D_oFFA) * F_o * 0.9 / 282 * F_g.$$

where  $F_g$  is the gas flow rate in mol h<sup>-1</sup>.

The concentration of free fatty acids of the deacidified oil (mol/mol) would be:

$$D_oFFA_m = 10 * D_oFFA * 884 / (1000 * 282).$$

(884 g as the average molecular weight of oil).

## 3. RESULTS AND DISCUSSION

Table 1 summarizes the results for different trials. Columns 1 and 10 identify the trials. Columns 2-8 show the process variables: Oil flow ( $F_o$ ); oil temperature ( $T_o$ ); gas distillate temperature inside the continuous deodorizer ( $T_g$ ); total pressure; gas flow rate ( $F_g$ ); and the  $I_oFFA$  and  $D_oFFA$ . Column 9 shows  $GFFA_m$ .

As can be observed, the values for  $GFFA_m$  were highly variable reaching values as high as 1.105 mol of the free fatty acids *per* mol of stripping gas (essay 23). On the other hand, the heating of the gases in the head up to the operation temperature ( $T_g = T_o$ ) increases  $GFFA_m$  as can be confirmed by comparing the assays 1-13; 4-19; 5-6; 5-20; 6-20; 7-8; 7-21; 8-21; 9-28; 10-25; 11-26 and 12-44. However, the increase of  $T_g$  above  $T_o$  in 20°C has no influence in practice (assays 21-46, 42-47 and 44-48).

Figure 1 shows the relationship between  $D_oFFA_m$  and  $GFFA_m$  for those samples in Table 1 having  $T_o = T_g = 240$  °C and  $F_o = 3.125$  molh<sup>-1</sup>. A relationship between the content of free fatty acids in the deacidified oil ( $D_oFFA_m$ ) and the concentration of free fatty acids in the gas ( $GFFA_m$ ) can be observed.

An interpretation of this tendency would be to consider, ideally at least, that in the continuous process of the deacidification of free fatty acids by distillation with vacuum, high temperature, stripping gas and in the indicated conditions ( $T_g = T_o$ ), when equilibrium is reached in the deodorizer, the ratio  $GFFA_m / D_oFFA_m$  is constant. This constant depends on the solubility of free fatty acids into the oil and on their vapor pressure (both variables depending on the temperature) and on the type of oil. In the equilibrium,  $D_oFFA_m$  and  $GFFA_m$  are governed by this constant coefficient.

The function that relates the constant described with the independent variables of the physical

Table 1  
Results obtained in different deacidification assays

Assay	F <sub>o</sub> l h <sup>-1</sup>	T °C		p mbar	F <sub>g</sub> molh <sup>-1</sup>	I <sub>o</sub> FFA % w/w	D <sub>o</sub> FFA % w/w	GFFA <sub>m</sub> mol/mol	References Prieto et al.
		T <sub>o</sub>	T <sub>g</sub>						
<b>T<sub>o</sub> &gt; T<sub>g</sub></b>									
1	4,8	226	173	2,2	3,125	3,93	2,70	0,060	[2008]
2	3,0	240	191	3,9	1,786	7,40	1,00	0,343	[2008]
3	5,0	240	195	10,1	1,785	7,20	5,20	0,179	[2008]
4	3,0	240	166	6,8	3,125	2,50	1,70	0,025	[2008]
5	5	240	194	3,2	3,125	2,50	0,70	0,092	[2007a]
6	5	240	210	1,7	3,125	2,50	0,34	0,110	[2007a]
7	5	240	199	1,9	3,125	7,30	1,90	0,276	[2007a]
8	5	240	210	1,7	3,125	7,30	1,12	0,316	[2007a]
9	4,8	245	166	2,6	6,250	3,93	1,64	0,056	[2008]
10	5	260	192	3,3	1,786	2,40	0,60	0,161	[2008]
11	5	260	209	2,5	3,125	2,50	0,38	0,108	[2007a]
12	5	260	207	1,9	3,125	7,30	0,46	0,349	[2007a]
<b>T<sub>o</sub> = T<sub>g</sub></b>									
13	4,8	226	226	2,2	3,125	3,93	0,73	0,157	[2008]
14	4,5	235	235	3,9	3,125	1,92	0,33	0,073	[2008]
15	5,0	240	240	10,1	1,786	2,50	0,14	0,211	[2007b]
16	5,0	240	240	6,8	1,786	2,50	1,9	0,054	[2008]
17	5,0	240	240	3,2	1,786	2,50	0,6	0,170	[2008]
18	5,0	240	240	1,7	1,786	7,00	0,39	0,590	[2007b]
19	3,0	240	240	1,9	3,125	2,50	0,04	0,075	[2007b]
20	5,0	240	240	1,7	3,125	2,50	0,11	0,122	[2007a]
21	5,0	240	240	2,6	3,125	7,30	0,64	0,340	[2007a]
22	5,0	240	240	3,3	3,125	7,00	0,21	0,347	[2007b]
23	5,0	240	240	2,5	3,125	26,33	4,69	1,105	[2007a]
24	5,0	240	240	1,9	8,036	26,33	1,96	0,483	[2007a]
25	4,5	245	245	2,2	3,125	1,92	0,49	0,066	[2008]
26	4,5	245	245	3,9	1,786	1,92	0,52	0,113	[2008]
27	4,8	245	245	10,1	3,125	3,93	0,31	0,177	[2008]
28	4,8	245	245	6,8	6,250	3,93	0,32	0,088	[2008]
29	4,5	255	255	3,2	3,125	1,92	0,11	0,083	[2008]
30	5,0	255	255	1,7	3,125	2,90	0,19	0,138	[2007a]
31	5,0	255	255	1,9	3,125	7,30	0,35	0,356	[2007a]
32	5,0	255	255	1,7	8,036	15,55	0,12	0,306	[2007a]
33	5,0	255	255	2,6	8,036	20,36	0,15	0,401	[2007a]
34	5,0	260	260	3,3	1,786	2,50	0,16	0,209	[2007b]
35	5,0	260	260	2,5	1,786	2,40	0,40	0,179	[2008]
36	3,0	260	260	1,9	3,125	2,50	0,05	0,075	[2007b]
37	5,0	260	260	2,2	1,786	2,50	0,20	0,205	[2008]
38	5,0	260	260	3,9	1,786	7,00	0,20	0,608	[2007b]
39	3,0	260	260	10,1	1,786	7,20	1,50	0,306	[2008]
40	5,0	260	260	6,8	1,786	7,20	2,20	0,447	[2008]
41	3,0	260	260	3,2	1,786	7,40	0,5	0,370	[2008]
42	5,0	260	260	1,9	3,125	2,50	0,09	0,123	[2007b]
43	3,0	260	260	1,7	3,125	7,00	0,09	0,212	[2007b]
44	5,0	260	260	2,6	3,125	7,30	0,33	0,356	[2007a]
<b>T<sub>o</sub> &lt; T<sub>g</sub></b>									
45	5,0	240	260	2,0	3,125	2,50	0,27	0,114	[2007a]
46	5,0	240	260	1,3	3,125	7,30	0,67	0,339	[2007a]
47	5,0	260	280	1,7	3,125	2,50	0,13	0,121	[2007a]
48	5,0	260	280	1,5	3,125	7,30	0,32	0,356	[2007a]

Abbreviations: F<sub>o</sub>, oil flow; T<sub>o</sub>, oil temperature; T<sub>g</sub>, gas distillates temperature inside the continuous deodorizer; I<sub>o</sub>FFA and D<sub>o</sub>FFA, free fatty acid content of initial and deacidified oil, respectively. GFFA<sub>m</sub>, mols of distilled fatty acids per mol of stripping gas.

refining process (temperature and absolute pressure), is unknown. For this reason, for a small range of temperatures (between 220 and 265°C) and absolute pressures (between 2 and 4 mbar),

that unknown function can be equated to a second degree equation in both variables:

$$GFFA_m / D_oFFA_m = A + B * T_o + C * \text{total pressure} + D * (T_o)^2 + E * (\text{total pressure})^2.$$

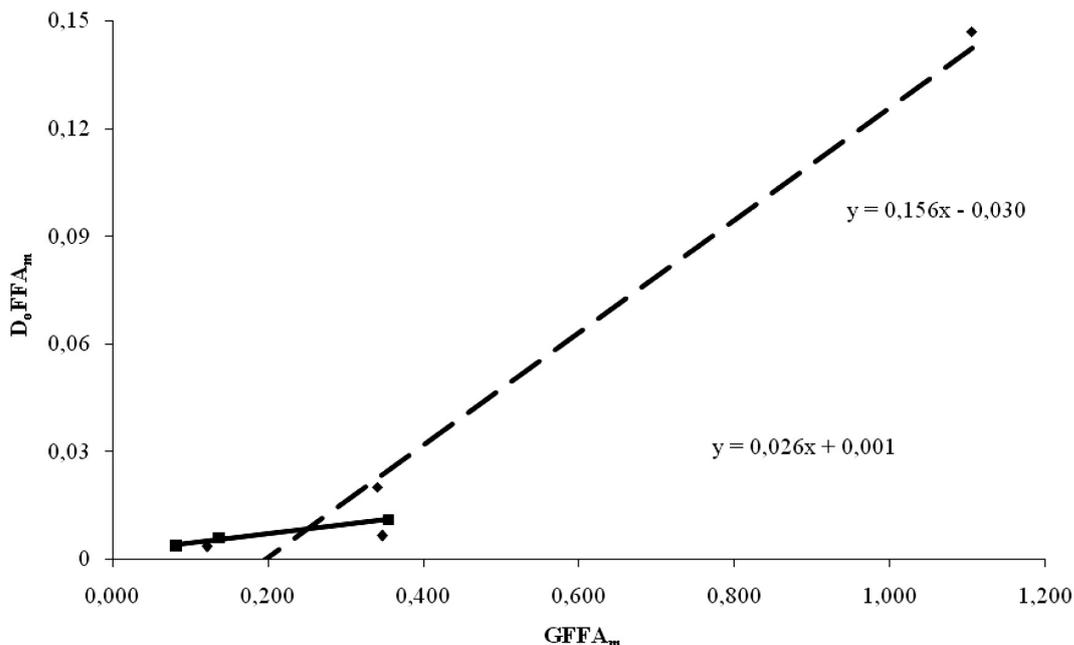


Figure 1  
 Relationship between the content of free fatty acids in the deacidified oil ( $D_{o,FFA_m}$ ) and the concentration of free fatty acids in the gas ( $GFFA_m$ ). Assay n<sup>o</sup> 20,21,22 and 23 ( $T_o = 240$  °C,  $T_g = T_o$  and  $F = 3,125$  mol  $h^{-1}$ ); assay n<sup>o</sup> 29, 30 and 31 ( $T_o = 255$  °C,  $T_g = T_o$  and  $F = 3,125$  mol  $h^{-1}$ ).

According to this, an adjustment by minimum quadrates to the following equation (Davies 1965) have been used with all data (Table 1,  $T_g = T_o$ ):  $A = -222,4$ ;  $B = 1,26$ ;  $C = -4,48$ ;  $D = -0,00079$ ;  $E = -0,0062$ .

This interpretation shows that in equilibrium conditions ( $T_g = T_o$ ), for a determinate temperature and total pressure, if a determinate  $D_o,FFA$  is

required, the GFFA is determined by the constant mentioned above. Therefore,  $F_g$  depends on the free fatty acid distillates *per* hour ( $10 * (I_o,FFA - D_o,FFA) * F_o * \text{density of the oils}$ ) and on the constant  $D_o,FFA$  ( $\approx 0.03$  %).

Figure 2 corresponds to the expression of  $F_g$ . This chart is only adequate to the conditions shown in table 1. For others oils, stripping gases and

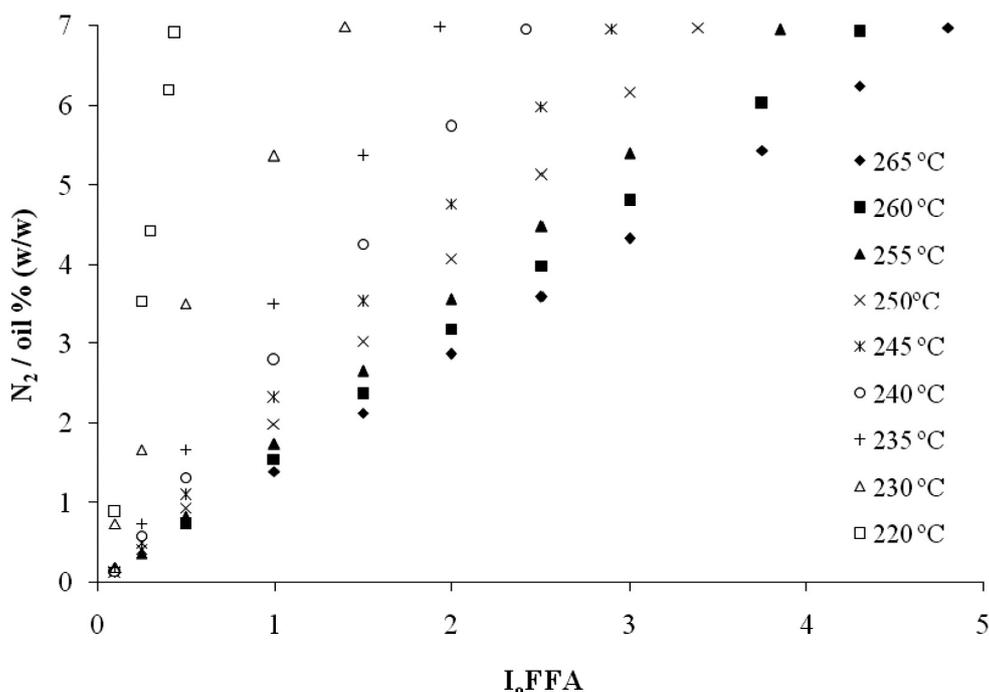


Figure 2  
 Continuous oil deacidification at 3 mm Hg, Oil flow of 5 l  $h^{-1}$  and free fatty acid content of the deacidified oil, 0.05 % w / w. Relationship between the ratio of stripping gas to oil and the initial free fatty acid content of the oil ( $I_o,FFA$ ).

deodorizers, there will be differences due to the different solubilities of free fatty acids in oils, to the interaction of the stripping gas (steam or nitrogen) with the oil and to the deodorizer design.

The maximum rate of the stripping gas that can be used depends on the oil particles which are being stripped and is a constant of the deodorizer design for each oil height. The maximum oil flow is conditioned by the necessary residence time in the deodorizer in order to its thermal decoloration. Knowing both limits, the optimum stripping gas flow must be used to minimise costs.

#### 4. CONCLUSIONS

The following hypothesis is submitted to discussion:

“In a continuous process of deacidification by the distillation of free fatty acids with stripping gas at low pressure (2-4 mbar), high temperature (180 – 265°C) and similar temperature for both the gas distillate inside the continuous deodorizer and the oil, the ratio between the free fatty acid content of the deacidified oil and the concentration of free fatty acids in the gas inside the continuous deodorizer is constant in the equilibrium”.

$$D_{\text{oFFA}_m} / G_{\text{FFA}_m} = \text{constant}''$$

The application of this hypothesis consists of considering the deacidification by distillation of free fatty acids in continuous, when  $T_g = T_o$ , as a hypothetical extraction of a component (the free fatty acids) between the two fluids (oil and stripping gas) and governed by the partition coefficient.

According to that, the physical refining in continuous ( $T_g = T_o$ ) should be developed into steps. For a better optimization, the deodorizer should have several stages in which the distillation is carried out at different temperatures ( $T_g = T_o$ ), and with different flow rates for both phases (oil and stripping gas) depending on the free fatty acids to be eliminated in each stage.

According to the exposed hypothesis, to deacidify an oil with  $I_{\text{oFFA}} = 7\%$  w/w, the first stage would be carried out with  $T_o = T_g = 260^\circ\text{C}$ ,  $F_o = 10\text{ l h}^{-1}$  and  $F_g (\text{N}_2 / \text{oil}) = 1.6\%$  (w/w), being  $D_{\text{oFFA}} = 0.35\%$  (w/w); and the second stage with  $T_o = T_g = 240^\circ\text{C}$ ,  $F_o = 10\text{ l h}^{-1}$  and  $F_g (\text{N}_2 / \text{oil}) = 1.6\%$  (w/w), being  $D_{\text{oFFA}} = 0,03\%$  (w/w).

The first stage should be carried out at the appropriate temperature and stripping gas in order to distillate the residues of the sanitary treatments during seed cultivation or storage. The increase in energy cost that the industrial application of this physical refining form may involve is compensated by the improved quality of the oil obtained and the possibility of applying it to oils with acidity  $> 2\%$  (w/w free fatty acids content) and in some cases with higher acidity.

In any extraction, equilibrium is achieved sooner if the contact surface between the phases increases and also if the liquid phases distance that the substance to extract has to cover until the

interface between both phases decreases. The latter two criteria have been taken into account by some manufacturers in the development of new deodorizers; the first part of the deodorizer contains an adequate packing support to decrease the thickness of the oil film as much as possible in order to favor a closer contact between phases.

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