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1 **Modeling of sodium nitrite and water transport in pork meat**

2

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13

14 **Abstract**

15 Four models were used to simulate nitrite uptake and water loss during pork meat  
16 curing with sodium nitrite: three empirical ones (the Azuara, the Peleg and the  
17 Zugarramurdi and Lupin) and one theoretical (the diffusional).

18 By means of the Azuara and the Peleg models, the equilibrium moisture content  
19 and the equilibrium nitrite content were properly identified.

20 Zugarramurdi and Lupin's model did not provide information about process  
21 parameters.

22 The effective diffusivities of water ( $D_{we}$ ) and nitrite ( $D_{Ne}$ ) were calculated. The  
23 activation energy ( $E_{Na}$  and  $E_{wa}$ ) was evaluated from the parameters of both the  
24 Peleg and the diffusional models. The results were similar; the Peleg model  
25 having the advantage of simplicity of calculation.

26 The effect of meat anisotropy was confirmed from the diffusional model; the  
27 perpendicular transport of nitrite is easier than the parallel.

28 This study highlighted the importance of choosing the most appropriate model  
29 depending on the objective to be achieved.

### 30 **Keywords**

31 Modelling, nitrite, water, diffusion, pork meat

### 32 **1. Introduction**

33 Nitrate and nitrite are present in the human diet in two ways: as nutrients in many  
34 vegetables and as food preservation substances (Sindelar and Milkowski 2012).

35 Nitrites are added to meat products for different reasons, such as for the purposes  
36 of inhibiting potentially pathogenic microorganisms, stabilizing the product's color  
37 during curing, acting as an antioxidant or developing the typical aroma and flavor  
38 of these products (Honikel, 2008; Hospital et al., 2012). In the last few years,  
39 however, there has been growing controversy surrounding nitrate and nitrite  
40 safety in the human diet (Sindelar and Milkowski 2012). On the one hand,  
41 different studies highlight the contribution of nitrites to human nutrition and their  
42 therapeutic potential to prevent cerebrovascular accidents, myocardial infarction,  
43 hypertension or gastric ulceration (Lundberg and Weitzberg, 2009; Lundberg et  
44 al., 2008; Rocha et al., 2011). Bedale et al. (2016) point out that dietary nitrate  
45 and nitrite have positive health attributes associated with nitric oxide metabolism  
46 that are only now being understood. On the other hand, some epidemiological  
47 studies associate the ingestion of red and processed meats with colorectal cancer  
48 (Abid et al., 2014). The association with processed meats is partially attributed to  
49 nitrosamines, which are formed by the action of nitrites through a reaction with  
50 secondary amines in an acidic environment, such as that present in the stomach

51 (Butler, 2015). However, according to Butler (2015), the presence of nitrites in  
52 food does not represent a health hazard. This author could find no substantial  
53 epidemiological evidence of a correlation between nitrosamine formation and the  
54 incidence of gastric cancers.

55 In the EU, potassium and sodium nitrite are currently restricted by Regulation no.  
56 1129/2011 (Commission Regulation (EC) No 1129/2011), which is urging the  
57 meat industry to modify the technologies used in cured meat production in order  
58 to reduce the nitrites added to meat products. Nevertheless, this reduction could  
59 affect the quality and safety of cured products (Dineen et al., 2000). It is, thus,  
60 essential to monitor the curing process, which implies a better understanding of  
61 nitrite uptake kinetics and the factors governing the process (e.g. temperature).

62 To this end, mathematical models are very useful due to the cost and time  
63 involved in experimental salting and curing studies (Chabbouh et al., 2012).

64 Models in general, and those for salting and curing processes in particular, can  
65 be classified as theoretical or empirical. Theoretical models are developed from  
66 mass and energy balances, considering the principles of chemistry, physics and  
67 biology (Gómez et al., 2015a). Of these models, the diffusional ones are widely  
68 used for meat salting and curing. Usually, water diffusion and salt diffusion are  
69 considered separately and an effective diffusivity is calculated for both  
70 substances (Uribe et al., 2011; Chabbouh et al., 2012; Gómez et al., 2015b;  
71 Gómez et al., 2017).

72 Empirical models are not based on general or specific laws. As a general rule,  
73 the simpler the model, the easier its mathematical solution (Gómez et al., 2015a).

74 In fact, the main advantage of empirical models is that no complex mathematical  
75 algorithms are needed, shortening the calculation time with a reasonably good

76 description of the process. Of the empirical models used to describe meat salting  
77 and curing, Azuara's model (Schmidt et al., 2009; Corzo et al., 2012), Peleg's  
78 model (Corzo et al., 2012; Chabbouh et al., 2012) and Zugarramurdi and Lupin's  
79 model (Chabbouh et al., 2012; Corzo et al., 2013) are worth highlighting.

80 As Gómez et al. (2015a) points out, the level of complexity needed in a model  
81 depends on the objective to be reached. A compromise between the simplicity of  
82 the model and a good description of the experimental results should be  
83 guaranteed; thus, it is advisable to analyze the model to be used in each case  
84 according to the objective of the study to be carried out.

85 Based on what has been mentioned above, the objective of this study is to test  
86 different models with which to simulate nitrite gain and water loss kinetics during  
87 the curing of pork meat in a saturated brine of sodium nitrite at different  
88 temperatures prior to the optimization of the operating conditions.

89

## 90 **2. Materials and methods**

### 91 **2.1 Raw material**

92 Eight pork legs from different animals were selected from a local slaughterhouse  
93 (average weight,  $9.6 \pm 1.2$  kg; pH 45 hours *post mortem* > 6.0 and pH 24 hours  
94 *post mortem* =  $5.9 \pm 0.1$ , measured in *Semimembranosus*, SM, muscle). The legs  
95 were wrapped in a polyvinyl chloride film and stored at  $2 \pm 1^\circ\text{C}$  for 13-14 h before  
96 separating the SM muscle from each leg. Twelve cylinders, 8.4 cm in height and  
97 2.4 cm in diameter, were obtained from each muscle, keeping the orientation of  
98 the meat fibers parallel to the cylinder axis, as explained in Gómez et al. (2017).

99

100

## 101 **2.2 Curing of the meat pork**

102 The curing of meat cylinders was carried out in duplicate at four temperatures (0,  
103 4, 8 and 12 °C), as in experiment II by Gómez et al. (2017), although NaNO<sub>2</sub> was  
104 used as a curing agent instead of NaNO<sub>3</sub>.

105 For each temperature and replication, ten of the twelve cylinders obtained from a  
106 muscle were used for curing with a saturated brine of sodium nitrite (NaNO<sub>2</sub>).  
107 Another cylinder was used to determine the equilibrium concentration of nitrite  
108 and water (7 days of immersion) and the remaining one was used to characterize  
109 the initial conditions of the meat. A total of 96 cylinders were analyzed: 8 for initial  
110 conditions, 8 for equilibrium concentration and 80 for the experimental kinetics.

111 The brine was prepared with an excess of NaNO<sub>2</sub> in order to compensate for the  
112 amount of salt absorbed by the meat.

113 The curing process lasted 5 days; one cylinder was removed from the brine every  
114 12 hours and, by using a bore, two sections were obtained: an internal (1.2 cm  
115 diameter) and an external one. The evolution of the nitrite and water content of  
116 both sections over time was determined.

117

## 118 **2.3 Analytical techniques**

### 119 **2.3.1. pH determination**

120 The pH (45 hours *post mortem* and 24 hours *post mortem*) was measured using  
121 a lab pH-meter for solids (Mattäus pH-STAR CPU, Pötmes, Germany).

### 122 **2.3.2. Water content.**

123 Both the initial water content and the evolution of the water content of each  
124 cylinder section over time were determined by the AOAC methodology (AOAC,  
125 1997). The determinations were carried out in duplicate.

126 **2.3.3. Nitrite determination**

127 The nitrite concentration was determined following the procedure described in  
128 Gómez et al. (2015b).

129

130 **2.4. Modelling**

131 Four models were used to model the experimental curing kinetics. The goodness  
132 of fit was evaluated for all of them by means of the percentage of explained  
133 variance (%var) and the mean relative error (%EMR).

134 **2.4.1. Azuara's model**

135 Azuara et al. (1992) proposed a model for both water loss (equation 1) and salt  
136 uptake (equation 2).

$$\frac{t}{w} = \frac{1}{k_{Aw}w_e} + \frac{t}{w_e} \quad (1)$$

$$\frac{t}{s} = \frac{1}{k_{As}s_e} + \frac{t}{s_e} \quad (2)$$

137 **2.4.2. Peleg's model**

138 Peleg's model (Peleg, 1988) is widely used in food processing. Equations 3 and  
139 4 show the water loss and the salt uptake during curing, respectively.

$$\frac{t}{X - X_0} = k_1 - k_2t \quad (3)$$

$$\frac{t}{X_s - X_{s0}} = k_3 + k_4t \quad (4)$$

140 The equilibrium moisture content can be calculated from Peleg's constant,  $k_2$   
141 (Equation 5). In the same way, the equilibrium salt content can be calculated from  
142  $k_4$  (Equation 6).

$$X_e = X_0 - \frac{1}{k_2} \quad (5)$$

$$X_{se} = X_{s0} + \frac{1}{k_4} \quad (6)$$

143 **2.4.3. Zugarramurdi and Lupin's model**

144 Zugarramurdi and Lupin (1980) proposed a model for the curing process.

145 Equation 7 describes water loss and salt uptake is described by Equation 8.

$$X = X_0 \exp(-k_{zw}t) + X_e(1 - \exp(-k_{zw}t)) \quad (7)$$

$$X_s = X_{s0} \exp(-k_{zs}t) + X_{se}(1 - \exp(-k_{zs}t)) \quad (8)$$

146 **2.4.4. Diffusional model**

147 A simplified diffusional model based on Fick's second law was used to describe

148 the experimental curing kinetics. The following assumptions were made:

- 149 - at the beginning of the curing process, the concentrations of water and nitrite
- 150 are constant and homogeneous in the meat samples
- 151 - one-dimensional transport perpendicular to the meat fibers takes place,
- 152 implying an infinite cylinder geometry.
- 153 - the external resistance to mass transfer is negligible
- 154 - the solid is homogeneous and isotropic
- 155 - the effective diffusivity is constant
- 156 - the dimensions of the samples are constant throughout the experiment

157 The solution of the governing equation that considers both the initial and boundary

158 conditions described above gives Equations 9 and 10.

$$\frac{C(r, t) - C_e}{C_0 - C_e} = 2 \sum_{n=1}^{\infty} \frac{e^{-D_e \lambda_n^2 t}}{\lambda_n R J_1(\lambda_n R)} J_0(\lambda_n r) \quad (9)$$

$$\lambda_n/J_0(\lambda_n R) = 0 \quad (10)$$

159 where  $\lambda_n$  represents the characteristic values ( $m^{-1}$ ).

160 The average nitrite and water content for both the internal cylinder (I) and the  
161 external section (E) at a given time was calculated by integrating Equation 9  
162 between 0 and  $R/2$  for section I, and between  $R/2$  and  $R$  for section E. A detailed  
163 description of the calculation can be found in Gómez et al. (2017).

164 To estimate the effective diffusivity, an optimization problem was formulated. The  
165 SOLVER tool of EXCEL™ (Microsoft Excel) was applied to solve this optimization  
166 problem, which uses a non-linear optimization method, namely the generalized  
167 reduced gradient. The nitrite diffusivity ( $D_{Ne}$ ) and water diffusivity of ( $D_{we}$ ) were  
168 calculated by minimizing the mean of the squared differences between the  
169 experimental and calculated concentrations, using the model.

#### 170 **2.4.5. Influence of temperature on model parameters**

171 The influence of temperature on the water and nitrite transport was determined by  
172 applying the Arrhenius equation.

173

### 174 **3. Results and discussion**

#### 175 **3.1 Water content**

176 The experimental average moisture content of the two cylinder sections during  
177 the curing process at different temperatures is shown in Fig. 1. It can be observed  
178 that the moisture content in both cylinder sections dropped when the curing time  
179 lengthened and the temperature rose. The moisture content fell more quickly  
180 during the first 2 days, thereafter remaining nearly constant. As expected, during  
181 this initial period, the external section, in contact with the brine, presented a faster  
182 dehydration than the internal one; thus, the first part of the curve shows a more

183 marked slope. In this same period, the temperature was observed to exert an  
184 influence in both cylindrical sections, so that the higher the curing temperature,  
185 the greater the initial moisture loss. The same behavior has been observed in  
186 previous research studies on curing (Gómez et al., 2015b; Gómez et al., 2017).  
187 The equilibrium moisture content of the meat samples after 3 days of curing was  
188 0.84 kg water/kg dry matter for 0°C and 4°C in both sections, while for 8°C and  
189 12°C, it was 0.75 kg water/kg dry matter. Similar values were obtained by Gómez  
190 et al. (2015b) when curing pork meat with sodium nitrite ( $\text{NaNO}_2$ ) perpendicularly  
191 to meat fiber.

192

### 193 **3.2 Nitrite content**

194 The experimental results for the nitrite content of the two cylinder sections are  
195 shown in Fig. 2. A faster increase in the nitrite content of the external cylinder  
196 was observed at every experimental temperature during the first day of curing,  
197 whereas this increase was slower in the internal cylinder. There are two factors  
198 behind this rapid movement of the nitrite on the meat cylinder surface in the initial  
199 period: first, the large concentration gradient between the meat surface and the  
200 brine at the beginning of the curing process and, second, the high moisture  
201 content of the samples (Fig. 1), which easily facilitates nitrite diffusion in meat  
202 (Gómez et al., 2015b). Other authors reported that salt intake and water loss  
203 occurred simultaneously during curing and these two events mutually affected  
204 each other (Akköse and Aktas, 2014). Temperature was observed to have an  
205 effect on nitrite transport, increasing the nitrite content of the samples as the  
206 temperature rose. At the end of the studied period, the nitrite concentrations in  
207 the internal and external sections were similar, with values close to equilibrium:

208 160.5 g nitrite/L (0.13 kg nitrite/kg dry matter) at 0°C, 173.3 g nitrite/L (0.15 kg  
209 nitrite/kg dry matter) at 4°C, 181.6 g nitrite/L (0.14 kg nitrite/kg dry matter) at 8°C  
210 and 197.55 g nitrite/L (0.15 kg nitrite/kg dry matter) at 12°C, indicating that a  
211 homogeneous distribution of the sodium nitrite was attained.

212

### 213 **3.3. Mathematical modelling**

214 The experimental results were modelled from the average experimental kinetics  
215 data. Tables 1, 2 and 3 show the results for the empirical models. A good fit was  
216 obtained between the experimental and calculated data, as confirmed by the  
217 percentage of explained variance, which was higher than 94% for every  
218 experiment, and the mean relative error, which was lower than 10%. In Figure 3,  
219 the fit between experimental and calculated values for the three empirical models  
220 is presented. As can be observed, all the values are close to the diagonal ( $R^2 =$   
221  $0.86$  for water content and  $R^2 = 0.93$  for nitrite content) which confirms the good  
222 agreement between the experimental kinetics and the values calculated by  
223 means of the empirical models.

224 The equilibrium moisture content ( $X_e$ ) and the equilibrium nitrite content ( $X_{se}$ )  
225 obtained from Azuara's model coincide with the experimental values. The  
226 equilibrium values obtained by means of Peleg's model ranged between 0.68 and  
227 0.69 kg water/kg dry matter and 0.15 and 0.16 kg nitrite/kg dry matter,  
228 respectively, which also agree with the experimental ones. It can be thus stated  
229 that both models are useful for determining the equilibrium values under the  
230 experimental conditions of this study.

231 The values obtained for the models' parameters are of the same order as the  
232 ones found in the literature concerning meat products (Chabbou et al., 2012;  
233 Corzo et al., 2012; Corzo et al., 2013).

234 A key aspect when modeling is to determine the influence of the process  
235 parameters on the results. In this study, the experimental kinetics were  
236 determined at four temperatures; thus, the influence of temperature on the  
237 parameters of the model has to be achieved. For both the Azuara and the  
238 Zugarramurdi and Lupin models, no relationship was found between either  
239 models' parameters and the temperature (Tables 1 and 3). However, in the case  
240 of the  $k_1$  and  $k_3$  parameters from Peleg's model, the higher the temperature, the  
241 lower they were. Specifically, the influence of temperature was assessed by  
242 means of an Arrhenius equation. Furthermore, the activation energy for water  
243 ( $E_{wa}$ ) and nitrite ( $E_{Na}$ ) were 51.11 kJ/mol ( $R^2= 0.93$ , EMR = 0.99%) and 20.17  
244 kJ/mol ( $R^2 = 0.99$ , EMR = 2.77%), respectively. These results agree with others  
245 found in the literature (Gómez et al 2017; Gómez et al., 2015b; Gou et al., 2003).  
246 The results from the diffusional model are shown in Table 4, while Figure 4 shows  
247 the fit between the experimental values and the ones calculated using this model.  
248 As can be observed in Figure 4, a good fit is obtained between the experimental  
249 and calculated values ( $R^2 = 0.95$  for water content and  $R^2 = 0.95$  for nitrite  
250 content); moreover, the percentage of explained variance is high and the  
251 percentage of mean relative errors is low (Table 4), all of which allows us to state  
252 that the proposed diffusional model is good for describing meat curing kinetics.  
253 Both water and nitrite diffusion coefficients in Table 4 increased when the  
254 temperature rose. This effect has been observed by other authors during salting  
255 and curing experiments for the diffusion of salts (Gómez et al 2017; Gómez et al.,

256 2015b; Telis et al., 2003; Pinotti et al., 2002) and water (Gómez et al 2017;  
257 Gómez et al., 2015b; Gou et al., 2003). The activation energy results obtained by  
258 means of the Arrhenius equation were 54.17 kJ/mol for water ( $E_{wa}$ ,  $R^2$  0.96, EMR  
259 =  $2.4 \cdot 10^{-9}$  %) and 17.57 kJ/mol for nitrite ( $E_{Na}$ ,  $R^2$  =0.98, EMR =  $2.14 \cdot 10^{-10}$  %).  
260 These results are similar to the ones obtained by using Peleg's model and are  
261 also in agreement with others found in the literature on pork meat (Gómez et al  
262 2017; Gómez et al., 2015b; Gou et al., 2003). Peleg's model has the advantage  
263 of allowing the activation energy to be calculated in a simpler way. This has been  
264 pointed out by other authors while studying the drying process (Clemente et al.,  
265 2014).

266 Tables 5 and 6 gather the effective diffusivity values obtained by other authors  
267 working on meat products. As can be observed, they are of the same order of  
268 magnitude as the ones obtained in this study.

269 It must be pointed out that the diffusion of water and nitrite depends on their  
270 direction with respect to the meat fiber. When the results obtained in the present  
271 study by means of the diffusional model are compared with the ones obtained by  
272 Gómez et al. (2015b) for nitrite and water diffusion during curing parallel to the  
273 meat fibers, we can observe that the effective diffusivity for water is greater in this  
274 direction than when it takes place perpendicularly to them; in the case of nitrite,  
275 the opposite is true. This behavior was also observed for nitrate curing (Gómez  
276 et al., 2017). Gómez et al (2017) suggest that when curing parallel to the meat  
277 fibers, greater dehydration is produced, limiting the salt movement. For that  
278 reason, nitrite transport is slower when cured parallel to the meat fibers than when  
279 it takes place perpendicularly.

280 If the results of nitrite diffusion coefficients are compared with the ones found by  
281 Gómez et al. (2017) for nitrates obtained perpendicularly by using the same  
282 model, the nitrite values are higher than the nitrate. Considering that nitrite has a  
283 lower molecular weight than nitrate, a higher diffusion coefficient is expected for  
284 the former.

285 As to the activation energy, the values for parallel diffusion (Gómez et al., 2015b)  
286 were 60.32 kJ/mol for nitrite and 32.24 kJ/mol for water; thus, nitrite needs more  
287 energy for parallel diffusion than for perpendicular. When curing perpendicularly  
288 to the meat fibers, the slower movement of water produces less dehydration,  
289 facilitating the diffusion of nitrites and, consequently, the effective diffusion is  
290 greater than when it takes place parallelly. The same behavior was observed by  
291 Gómez et al. (2017) studying nitrate diffusion. These results underline the  
292 importance of the anisotropy of meat when modelling curing processes, and the  
293 effect of water movement on nitrite diffusion. Nevertheless, further studies are  
294 needed to evaluate the effect of dry curing compared to brine curing.

295 Gómez et al. (2017) found activation energy values of 31.86 kJ/mol for nitrate  
296 and 24.71 kJ/mol for water during nitrate diffusion perpendicular to meat fibers.

297 As pointed out above, due to its lower molecular weight, the diffusion coefficients  
298 for nitrite are higher than for nitrate. As a consequence, if the diffusion is faster,  
299 less activation energy is needed for nitrite than for nitrate. Thus, the salt used  
300 during the curing process has an influence on it.

301

302 **4. Conclusions**

303 A good agreement was found between the experimental curing kinetics and the  
304 values calculated by means of the four models considered. Nevertheless, each  
305 model offered different information.

306 All the models provide information about the influence of the process parameters  
307 on the curing process, except the Zugarramurdi and Lupin model. From both  
308 Azuara's and Peleg's models, the predicted equilibrium moisture content and  
309 equilibrium nitrite content coincided with the experimental values.

310 According to the diffusional model, the perpendicular nitrite diffusion coefficient  
311 was higher than that of nitrate calculated in a previous study.

312 The activation energy for water and nitrite determined from the parameters of  
313 both the Peleg and the diffusional models was similar. However, the Peleg model  
314 had the advantage of simplicity of calculation. The values of the activation energy  
315 and the effective diffusivity confirm the effect of meat anisotropy during curing;  
316 the perpendicular transport of nitrite is easier than the parallel.

317 The above conclusions highlight that when modeling the curing process, it is  
318 important to choose the most appropriate model depending on the objective of  
319 the study.

320

## 321 NOMENCLATURE

$C$	Moisture or nitrite concentration	$\text{kg}\cdot\text{m}^{-3}$
$C_0$	Initial concentration of nitrite or water	$\text{kg}\cdot\text{m}^{-3}$
$C_e$	Equilibrium concentration of nitrite or water	$\text{kg}\cdot\text{m}^{-3}$
$D_e$	Effective diffusivity	$\text{m}^2\cdot\text{s}^{-1}$
$k_{As}$	Azuara's model parameter	$\text{day}^{-1}$
$k_{Aw}$	Azuara's model parameter	$\text{day}^{-1}$
$k_{Zw}$	Zugarramurdi and Lupin's model parameter	$\text{day}^{-1}$
$k_{Zs}$	Zugarramurdi and Lupin's model parameter	$\text{day}^{-1}$

$k_1$	Peleg's model parameter	day*g dry matter*g water <sup>-1</sup>
$k_2$	Peleg's model parameter	g dry matter*g water <sup>-1</sup>
$k_3$	Peleg's model parameter	day*g dry matter* g nitrite <sup>-1</sup>
$k_4$	Peleg's model parameter	g dry matter*g nitrite <sup>-1</sup>
$R$	Radius of the cylinder	m
$r$	Radial coordinate	m
$s$	Nitrite content	g nitrite*(g initial sample) <sup>-1</sup>
$s_e$	Equilibrium nitrite content	g nitrite*(g initial sample) <sup>-1</sup>
$t$	Time (diffusional model)	s
$t$	Time (empirical models)	day
$w$	Moisture content	g water*(g initial sample) <sup>-1</sup>
$w_e$	Equilibrium moisture content	g water*(g initial sample) <sup>-1</sup>
$X$	Moisture content	kg water*(kg dry matter) <sup>-1</sup>
$X_e$	Equilibrium moisture content	kg water*(kg dry matter) <sup>-1</sup>
$X_0$	Initial moisture content	kg water*(kg dry matter) <sup>-1</sup>
$X_s$	Nitrite content	kg nitrite*(kg dry matter) <sup>-1</sup>
$X_{se}$	Equilibrium nitrite content	kg nitrite*(kg dry matter) <sup>-1</sup>
$X_{s0}$	Initial nitrite content	kg nitrite*(kg dry matter) <sup>-1</sup>

322

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326

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