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Additional Information

# 1 **Modelling Osmotic dehydration of lemon slices using new sweeteners**

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7

## 8 **Abstract**

9 Lemon slices were osmotically dehydrated using the following healthy  
10 sweeteners as osmotic agents: tagatose, isomaltulose, oligofructose and  
11 aqueous extract of stevia. A kinetic study using a Fickian approach was  
12 performed, which also analysed the changes in water activity, total mass, mass  
13 of water and mass of soluble solids in lemon slices. The results showed that the  
14 greatest value of effective diffusivity ( $D_e$ ) in osmodehydrated lemon slices was  
15 obtained from a combination of oligofructose and stevia. However, the level of  
16 water activity ( $a_w$ ) reached with this syrup was the highest, meaning that the  
17 product might be less stable. Additionally, isomaltulose favoured the total mass,  
18 whereas tagatose did the opposite. Finally, the syrup recommended for  
19 dehydrating lemon slices would be a combination of tagatose, oligofructose and  
20 aqueous extract of stevia since its  $D_e$  was similar to the value obtained when only  
21 oligofructose and stevia were used, but  $a_w$  values were lower.

22

23 **Keywords:** lemon, tagatose, isomaltulose, oligofructose, stevia, osmotic  
24 dehydration, kinetics.

25

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## 26 **Introduction**

27 Citrus fruits have played an important role in the economy and dietary habits in  
28 Spain. They are a good source of bioactive compounds like ascorbic acid,  
29 polyphenols and carotenoids and have been involved in the prevention of some  
30 diseases such as diabetes, obesity, cancer and cardiovascular diseases  
31 (Devalaraja *et al.*, 2011; Kim *et al.*, 2011). Among these citrus fruits, lemon is the  
32 product consumed the least due to its high acidity. However, the addition of  
33 sweeteners might be an effective way to counteract this feature. Therefore, the  
34 development of new sweet lemon products could promote consumption of this  
35 fruit, and in turn, improve the nutritional health of society. Osmotic dehydration  
36 (OD) might be a suitable technique for obtaining such products, since it has been  
37 widely applied to other fruits, such as oranges (Cháfer *et al.*, 2001; Rubio-Arreaez,  
38 *et al.*, 2015), pears (Park *et al.*, 2002), tomatoes (Azoubel and Murr, 2004),  
39 apples-(Moura *et al.*, 2005; Derossi *et al.*, 2008; Castelló *et al.*, 2009), apricots  
40 (Toğrul and İspir, 2007; İspir and Toğrul, 2009), strawberries (Castelló *et al.*,  
41 2006; Castelló *et al.*, 2010), kiwis (Castro-Giráldez *et al.*, 2011) and cherries  
42 (Silva *et al.*, 2012). Besides, OD has already been studied as an alternative which  
43 gives uses to lemon by-products (Masmoudi *et al.*, 2007). OD consists of placing  
44 foods in a low water activity solution in order to induce water outflows and inflows  
45 of external solutes, resulting in high quality products that can be stored  
46 longer.(Shi, 2008). However, the common use of sugars in the OD stage leads to  
47 an enhancement in the cariogenic property of the final products as well as an  
48 increase in their glycemic index, and can also be linked to different diseases  
49 (diabetes, obesity, etc.). Fortunately, there are other new sweeteners available in  
50 the market which are non-cariogenic and also have other advantages over

51 conventional sugars or sweeteners, such as isomaltulose, oligofructose, stevia  
52 and tagatose (Soto and Del Val, 2002; Goyal *et al.*, 2010). Each one is described  
53 below.

54 Isomaltulose is a reducing disaccharide which is naturally present in honey, and  
55 sugar cane juice, and its taste is similar to sucrose. The physicochemical  
56 properties of isomaltulose enable it to be used a substitute for sucrose in most  
57 sweet foods (Lina *et al.*, 2002; Bui *et al.*, 2009; De Oliva-Neto and Menão, 2009;  
58 Mercali *et al.*, 2011; Peinado *et al.*, 2013). It has a sweetening power of  
59 approximately 42% compare to sucrose and it can be used as an alternative to  
60 sucrose, because its caloric power is similar (Schiweck *et al.*, 1990; Periche *et*  
61 *al.*, 2014).

62 Oligofructose is low in calories, meaning it has multiple health benefits. It is  
63 obtained by partial enzymatic hydrolysis of chicory inulin and it is a soluble dietary  
64 fiber with prebiotic character to enhance the growth of beneficial gut bacteria and  
65 calcium absorption (Rao, 2001; Franck, 2002; Raschka and Daniel, 2005;  
66 Bosscher *et al.*, 2006; Al-Sherajia *et al.*, 2013).

67 Stevia is a plant that has been consumed as a food and also used as a medicine  
68 in some countries such as Japan and Paraguay (Lemus-Mondaca *et al.*, 2012).  
69 The sweetening power of this plant is 15 times greater than sucrose, and it has  
70 multiple therapeutic properties (antioxidant, antimicrobial, anti-fungal activity,  
71 anti-hyperglycemic, anti-hypertensive, anti-inflammatory, anti-tumor, anti-  
72 diarrheal and diuretic effects), but it is calorie free (Chatsudthipong and  
73 Muanprasat, 2009).

74 Tagatose is a fructose isomer in milk and milk products. In comparison with other  
75 sugars, it has numerous health benefits including a lower glycemic index, and low

76 calorie content. It also reduces the symptoms associated with type II diabetes  
77 and it is recommended for patients with obesity or heart diseases (Oh, 2007; Lu  
78 *et al.*, 2009; Gardner *et al.*, 2012; Shourideh *et al.*, 2012; Shankar *et al.*, 2013).  
79 Tagatose exerts greater osmotic pressure, and hence has less water activity than  
80 sucrose at equivalent concentrations (Patra *et al.*, 2009). D-Tagatose is well  
81 suited for confectionery products such as chocolate and candies, fudges,  
82 caramels, ice cream, soft drinks, and breakfast cereals because tagatose  
83 crystallizes easily (FAO/WHO, 2001, 2002, 2003).  
84 In consideration of all the above, the aim of this work is to study the effect of  
85 different combinations of healthy osmotic agents (tagatose, oligofructose,  
86 isomaltulose and stevia) on the kinetic behaviour of lemon slices in order to obtain  
87 mathematical models following second Fick's law. For this purpose, variation of  
88 total mass, soluble solids and water mass changes have been analysed over  
89 time.

90

## 91 **Materials and Methods**

### 92 *Preparation of sample*

93 *Eureka* Lemons of a similar colour, size and ripeness were selected from an  
94 agricultural plot in Lliria (Valencia). The lemons were peeled and cut into 0.5 cm  
95 thick slices using a household slicer (Fagor Delice CF- 150).

96

### 97 *Osmotic dehydration treatment*

98 Tagatose (Tagatesse<sup>®</sup>, Damhert) Isomaltulose (Palatinose<sup>™</sup> PST- N, Beneo  
99 palatinit), oligofructose (Fructalose<sup>®</sup> OFP, Sensus) and an aqueous extract with  
100 1% of dry Stevia leaves (*S. rebaudiana* Raab, Vitalfood, Rohrbach, Germany)

101 were used as agents for osmotic dehydration. Table 1 shows the combinations  
102 of these four sweeteners used in the four syrups considered and the code  
103 assigned.

104 The kinetic study was carried out for 48 hours by analysing samples at 0, 10, 20,  
105 30, 45, 60, 90, 120, 240, 300 minutes and at 24 and 48 hours. The ratio between  
106 syrup and lemon slices was 20:1 (w/w) with constant stirring so as not to modify  
107 the concentration of soluble solids in the syrup.

108

#### 109 *Physicochemical analysis*

110 Soluble solids in the liquid phase in orange slices and syrups were measured by  
111 a refractometer (Abbe Refractometer, Atago), obtaining the results in °Brix and  
112 also expressed as  $z_s$  (kg soluble solids/kg liquid phase). Moisture content ( $x_w$ : kg  
113 water/kg orange slices) was analysed gravimetrically following an adaptation of  
114 the AOAC method, (2000). Water activity ( $a_w$ ) was determined by a hygrometer  
115 (Decagon CX-1). All determinations were carried out in triplicate.

116

#### 117 *Kinetic study and modelling*

118 Variation of total mass ( $\Delta M$ ), mass of soluble solids ( $\Delta M_s$ ), and mass of water  
119 ( $\Delta M_w$ ) were calculated for all times considered in this study. Additionally, a Fick's  
120 model was used to obtain the effective diffusivity ( $D_e$ : m<sup>2</sup>/s) of soluble solids  
121 (Crank, 1975; Barat *et al.*, 1998; Cháfer *et al.*, 2001) depending on the  
122 composition of the syrup used.

123

#### 124 *Statistical analysis*

125 Statgraphics plus (version 5.1) software was used to perform the statistical  
126 analyses, the factor taken into account being the composition of the syrup used  
127 in the osmotic dehydration.

128

## 129 **Results and Discussion**

130

131 The average values of composition and water activity for lemons used in these  
132 experiments were the following:  $83\pm 4\%$  of water,  $15.7\pm 0.4\%$  of soluble solids and  
133  $0.778\pm 0.002$  of water activity. The °Brix of the osmotic solutions used were  
134  $35.15\pm 0.7$  for syrup T,  $29.50 \pm 0.7$  for syrup OS,  $36.30\pm 0.7$  for syrup IT and  
135  $32.90\pm 0.7$  for syrup ITOS. According to these results, tagatose would lead to the  
136 highest driving force.

137 Fig. 1 represents the results of moisture content ( $x_w$ ), water activity ( $a_w$ ) and °Brix  
138 of lemon slices *versus* time of osmotic dehydration depending on the syrup used.

139 As was expected the longer the time of dehydration the higher the concentration  
140 of soluble solids in lemon slices. Other studies with oranges and other fruits  
141 (strawberry, apple, apricot) showed similar results (Cháfer *et al.*, 2001; Castelló  
142 *et al.*, 2006, 2009; Íspir *et al.*, 2009). In the sweeteners studied, noteworthy was  
143 that the samples osmodehydrated with oligofructose and stevia showed the  
144 highest levels of water activity, while syrup composed only by tagatose led to the  
145 lowest increase in soluble solids. However no differences were found in terms of  
146 moisture content among the slices treated with the four syrups studied.

147 The results for variation of total mass ( $\Delta M$ ), water mass ( $\Delta M_w$ ) and soluble solid  
148 mass ( $\Delta M_s$ ) recorded in this study were obtained using the following formulas (Shi  
149 and Fito, 1994; Fito and Chiralt, 1996) shown in Figure 2.

150 
$$\Delta M = \frac{M^t - M^0}{M^0} \quad (1)$$

151 
$$\Delta M_w = \frac{M^t x_w^t - M^0 x_w^0}{M^0} \quad (2)$$

152 
$$\Delta M_s = \frac{M^t x_s^t - M^0 x_s^0}{M^0} \quad (3)$$

153 Where

154  $M^i$ : M mass of orange slices (kg) at time i (i=0 or t)

155  $M_w^i$ : mass of water of orange slices (kg) at time i (i=0 or t)

156  $M_s^i$ : mass of soluble solids of orange slices (kg) at time i (i=0 or t)

157  $x_w^i$ : mass fraction of water (kg of water/kg of orange slices) at time i (i=0 or t)

158  $x_s^i$ : mass fraction of soluble solids (kg of soluble solids/kg of orange slices) at  
159 time i (i=0 or t)

160 As can be seen in Fig. 2 samples dehydrated with syrups containing isomaltulose  
161 (IT and ITOS) showed the greatest losses of total mass, especially when they  
162 were combined with oligofructose and stevia. On the contrary, syrup composed  
163 by oligofructose and stevia (OS) kept the mass of lemon slices constant while  
164 syrup containing only tagatose (T) led to an increase in total mass in all samples  
165 for the whole process. Therefore, tagatose would be more beneficial in the  
166 development of osmodehydrated products, since contrary to what is common in  
167 this process, it led to an increase in mass.

168 Consistent with the values of moisture content registered during the process, no  
169 differences were found with regard to water and soluble solid mass changes.  
170 However, the combination of oligofructose and stevia with or without isomaltulose  
171 gave rise to the highest rates of soluble solid mass intake. This behaviour is  
172 noteworthy since driving forces recorded for these syrups were lower than in the



173 other cases, and consequently, a lower rate of soluble solid intake would have  
 174 been expected. It seems that it was more difficult for tagatose molecules to  
 175 penetrate the structure of the lemon slices whereas for oligofructose it was easier  
 176 to dehydrate this product.  
 177 Moreover, the changes in the composition in the liquid phase of lemon slices were  
 178 modeled using the eq. (4).

$$179 \quad Y_s^t = \frac{(z_s^t - y_s)}{(z_s^0 - y_s)} \quad (4)$$

180 Where:

181  $Y_s^t$ : driving force of soluble solids (dimensionless)

182  $z_s^i$ : soluble solid mass fraction in the liquid phase at time  $i$  ( $i=0$  or  $t$ )

183  $y_s$ : soluble solid mass fraction in the osmotic solution used for dehydration.

184

185 The latter ( $y_s$ ) was considered to be equal to the equilibrium concentration of each  
 186 syrup, having the values mentioned at the beginning of this section.

187 Fig. 3 shows the experimental points  $1 - Y_s$  versus  $t^{0.5}$  to adjust them to a simplified  
 188 Fickian approach for diffusion in a plane sheet, with only one term of the Fick's  
 189 second law series solution for short times (Crank, 1975) (equation 5). In this case,  
 190 the range of time considered corresponded only to the first 120 minutes of  
 191 osmotic treatment. From the fitting of this model, it is possible to obtain the kinetic  
 192 parameter of effective diffusivity ( $D_e$ ) which allows us to predict the process time  
 193 required to achieve a specific concentration of soluble solids in osmodehydrated  
 194 lemon slices (Table 2).

$$195 \quad 1 - Y_s = \left[ \frac{4D_e t}{\pi l^2} \right]^{0.5} \quad (5)$$

196 Where  $t$  is time of processing (s) and  $l$  is half thickness of the dehydrated sample  
197 (m).

198

199 The results of  $D_e$  showed that the best fitting was observed when OS syrup was  
200 used in the dehydration of lemon slices. Furthermore, syrup composed of  
201 oligofructose with stevia extract (OS) presented a higher slope, which  
202 corresponded to the highest value of effective diffusivity, whereas syrup made of  
203 tagatose showed the longest times of osmodehydration since the effective  
204 diffusivity was the lowest. This is consistent with the results relating to water and  
205 soluble solid mass changes. Isomaltulose did not significantly affect the values of  
206  $D_e$  for the syrups composed of tagatose or oligofructose.

207

## 208 **Conclusions**

209 According to this study, isomaltulose gave rise to the highest mass loss values in  
210 the osmodehydrated lemon slices, in contrast with tagatose. However, the level  
211 of concentration of soluble solids reached by tagatose would be lower than in the  
212 case of the other sweeteners and would be reached more slowly. Oligofructose  
213 combined with stevia would lead to a quicker concentration of soluble solids, but  
214 also to the highest level of water activity and therefore would potentially be the  
215 least stable. To solve this problem it would be recommendable to combine  
216 oligofructose, aqueous extract of stevia and tagatose.

217

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223

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364

365



366 **Legends to figures**

367 Figure 1. A) Water activity ( $a_w$ ) *versus* time B) °Brix *versus* time and  
368 C)  $x_w$  *versus* time for the lemon slices dehydrated with different osmotic solutions.

369

370 Figure 2. A) Variation of total mass ( $\Delta M$ ), B) Variation of water mass ( $\Delta M_w$ ) and  
371 C) Variation of soluble solid mass ( $\Delta M_s$ ) for the lemon slices dehydrated with  
372 different osmotic solutions.

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374 Figure 3. 1-Driving force of soluble solids ( $Y_s$ ) vs.  $t^{0.5}$  (square root of time of  
375 dehydration) for the lemon slices dehydrated with different osmotic solutions.

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Table 1. Percentage of sweeteners in the syrups used in the study of osmotic dehydration of orange slices.

	<b>Tagatose</b>	<b>Isomaltulose</b>	<b>Oligofructose</b>	<b>Aqueous solution containing 1% of Stevia</b>	<b>Water</b>
<b>Syrup T</b>	30%	-	-	-	70%
<b>Syrup OS</b>	-	-	30%	35%	35%
<b>Syrup IT</b>	10%	20%	-	-	70%
<b>Syrup ITOS</b>	10%	10%	10%	10%	50%

Table 2. Values of effective diffusion coefficient ( $D_e$ ) and correlation coefficients ( $R^2$ ) of Fick's equation for an infinite sheet (Crank, 1975).

	$D_e \times 10^9$ (m <sup>2</sup> /s)	$R^2$
<b>Syrup T</b>	2.27±0.15 <sup>a</sup>	0.87±0.05
<b>Syrup OS</b>	10.2±0.3 <sup>c</sup>	0.95±0.01
<b>Syrup IT</b>	4.7±1.9 <sup>ab</sup>	0.91±0.03
<b>Syrup ITOS</b>	9±3 <sup>bc</sup>	0.91±0.02

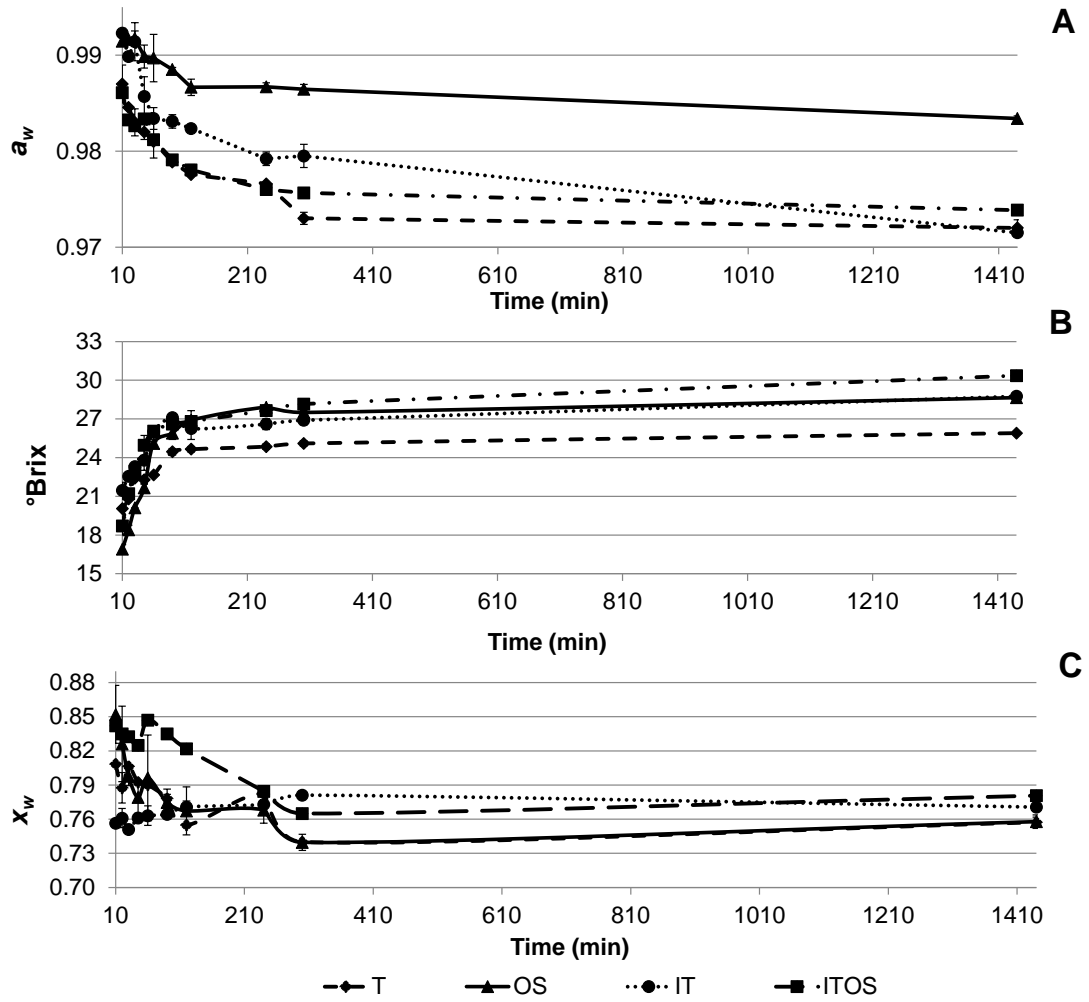


Figure 1. A) Water activity ( $a_w$ ) versus time B)  $^{\circ}\text{Brix}$  versus time and C)  $x_w$  versus time for the lemon slices dehydrated with different osmotic solutions

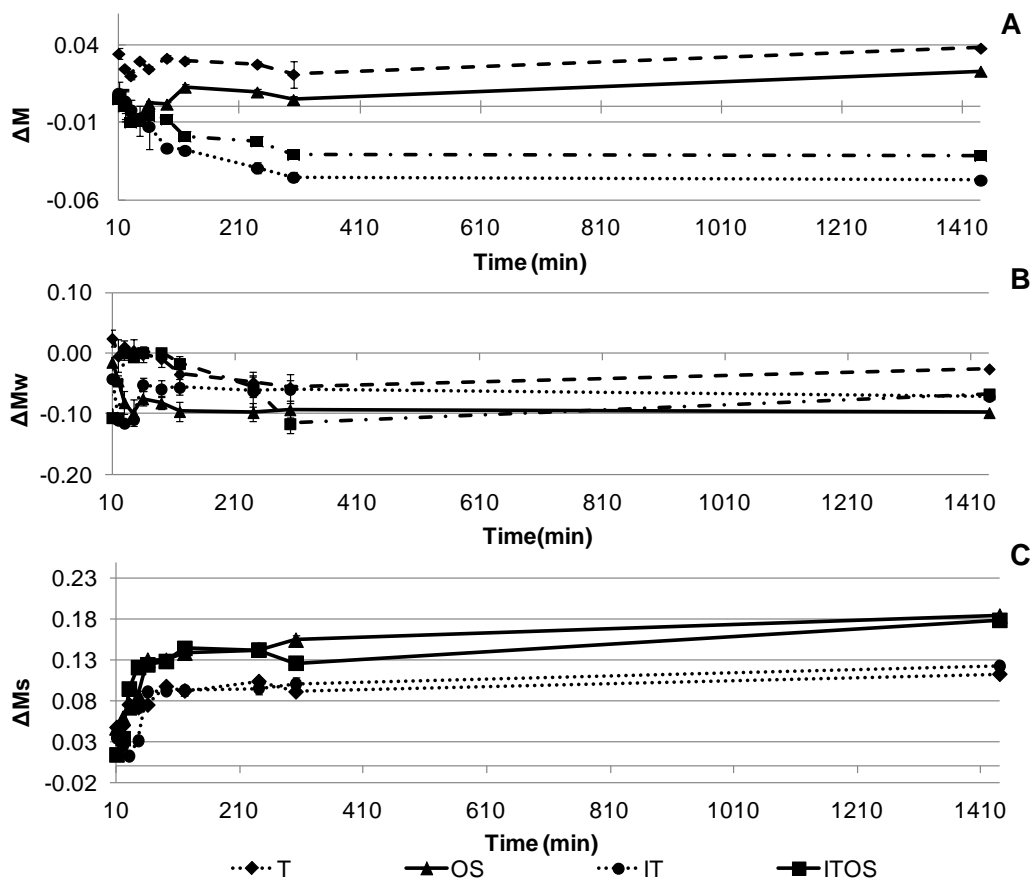


Figure 2. A) Variation of total mass ( $\Delta M$ ), B) Variation of water mass ( $\Delta M_w$ ) and C) Variation of soluble solid mass ( $\Delta M_s$ ) for the lemon slices dehydrated with different osmotic solutions

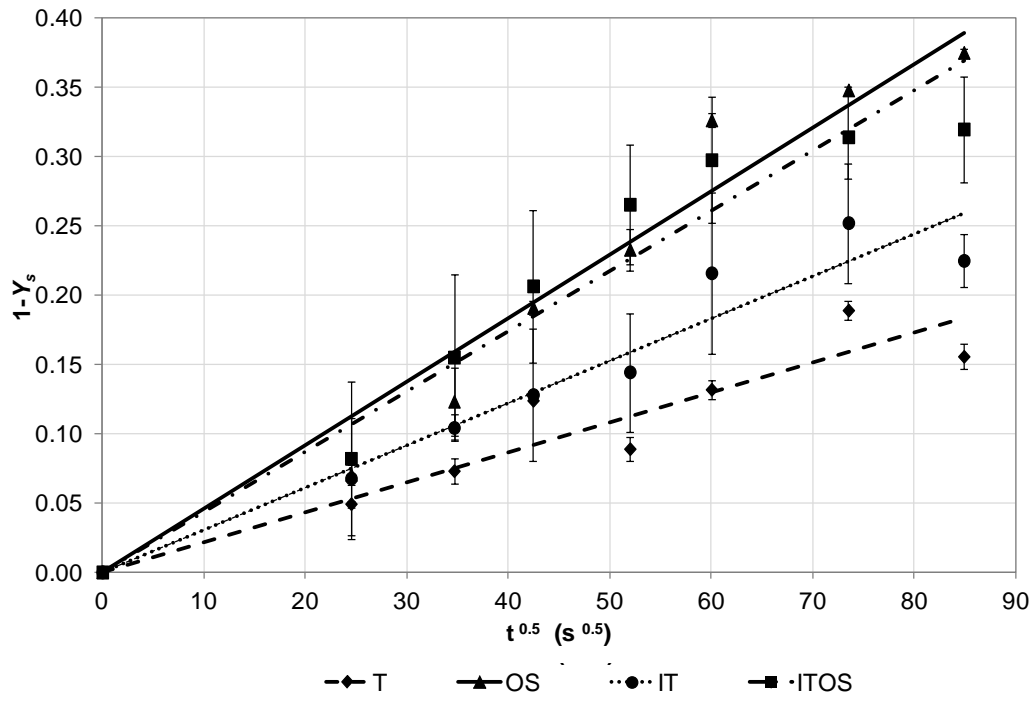


Figure 3. 1-Driving force of soluble solids ( $Y_s$ ) vs.  $t^{0.5}$  (square root of time of dehydration) for the lemon slices dehydrated with different osmotic solutions