

Application of Plackett-Burman Design to Identify Factors Affecting Significantly the Osmotic Dehydration

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Abstract : The aim of this study was to identify factors that have a significant influence on diffusion phenomena that take place during osmotic dehydration. To do this, nine (9) factors were investigated, based on the literature. An experimental design of Plackett-Burman was used to screen for these factors. The study was conducted by fixing for each factor, two levels: low and high. The experimental data were fitted using the chi square test (p-value) and the mean relative deviation (MRD) in order to conclude on factors affecting water loss or solute gain during the osmotic dehydration. Following this work, the molecular weight, temperature and concentration have been identified as factors having a significant influence on water loss (p-value>0.05 ;MRD <10%). The molecular weight, temperature, concentration, and rinsing of the sample have been identified as factors having a significant influence on solute gain for the sample (p-value>0.05 ;MRD <10%).

Keywords: Chi square test ; osmotic dehydration; Plackett-Burman design; solute gain ; water loss;.

I. INTRODUCTION

Osmotic dehydration is a process used for partial removal of water from the plant tissue by immersion in a hypertonic solution (El-Aouaret *al*, 2006 ;Torreggiani *et al*, 1987). This technique is used to produce fruit chips, candied fruits, fruit jams, ... (Sablani *et Rahman*, 2003 ; Agnelli *et al*, 2005). It offers many benefits that are: the conservations of color, flavor and texture of the fruit; preservation of nutritional, sensory and functional properties of the fruit; reducing the cost of energy used for convective drying; improving the solute content of the fruit and conservation of the texture and stability of the pigments; the increasing of yields in terms of production (Silveira *et al*, 1996 ; Sablani *et al*, 2002 ; Rahman *et perera*, 1999).

During osmotic dehydration, the food product is in contact with the osmotic solution. A bidirectional transfer of materials is achieved at the end of this contact. There is a massive water outlet from the plant tissue to the osmotic solution. This movement of water is often accompanied by substances dissolved of the product (sugars, vitamins, pigments, flavors, ...) (Corzo *et Gomez*, 2004; Azoubel *et Murr*, 2003 ; Lenart *et flink*, 1984). The driving force for the diffusion of product water to the solution is provided by the osmotic pressure which is higher in the osmotic solution (Rastogi *et Raghavarao*, 2004). The water diffusion is often accompanied by simultaneous diffusion of counter-current solutes from the hypertonic osmotic solution to dehydrated plant tissue (Rastogi *et Raghavarao*, 2004 ; Sablani *et al*, 2002 ; Flourey *et al*, 2008). The rates of water diffusion and solute in plant tissue depend on several factors such as: the concentration of the osmotic solution, molecular weight of the solutes, the osmosis temperature, thickness of the samples, the combination of solutes, the ratio solute/ product, the type of fruit dehydrated or vegetable, the rinsing of the samples, the agitating of the osmotic solution (Corzo *et Gomez*, 2004 ; Rastogi *et Niranjana*, 1998 ; Rastogi *et al*, 1997). Among these factors, some might not have a significant influence on the phenomena of the diffusion that take place during osmotic dehydration. Thus, to identify the important factors that actually influence the diffusion, the Plackett-Burman design which is a experimental design commonly used was applied for screening factors.

The usefulness of this experimental design is the fact that the effects of the study of one factor does not alter the effects of another factor by interaction so that the effects of each factor on the phenomenon can be independently determined. This experimental design also offers a limited number of experiments to determine the effects of each factor and to rank them in order of importance. (Yu *et al*, 1997 ;Goupy, 1990 ; Haalad, 1989 ; Plackett - Burman, 1946).

II. MATERIALS AND METHODS

2.1- Plant material

Samples of mango and papaya have been selected on the site of the great market of Yamoussoukro district. The varieties selected were: Kent variety for mango and solo variety for papaya. These fruits are washed, peeled and cut into slices of diameter 30 mm at thicknesses ranging from 15 to 40 millimeters. These slices have homogeneous surfaces to allow regular contact between this sample and the osmotic solution. The solutes used were: commercial sugar (sucrose) and glucose. (Floury et al, 2008; Rastogi and Raghavarao, 2004)(Floury et al, 2008 ;Rastogi et Raghavarao, 2004)

2.2. Osmotic dehydration

The slices of fruit were identified and wrapped in absorbent paper to remove water on the surface of these slices. Then they were weighed. To achieve the osmotic dehydration, these slices have been immersed in osmotic solutions defined according to the Plackett-Burman table. The slices of fruit are removed and weighed every 15 minutes up to 7 hours osmosis time. (Mauro et Menegalli, 2003 ; Nieto et al, 2004 ; Lenart, 1996).

The percentage of weight loss (WL) of the sample is determined by the following equation:

$$WL (\%) = \frac{M_o - M_t}{M_o} \times 100 \quad (1)$$

The solute content of the liquid phase of the sample was determined by measuring the brix of the sample with a refractometer at room temperature (AOAC, 1998). The percentage of solute gain (SG) of the sample is determined by the following equation:

$$SG (\%) = \frac{D_{bo} - D_{bt}}{D_{bo}} \times 100 \quad (2)$$

Where M_o and M_t were the initial and final time of the weights of the samples; D_{bo} and D_{bt} brix degrees of osmotic solution to the initial and final time (Wang et Brennan, 1995 ; Khoyi et Hesari, 2007).

2.3. Plackett-Burman design

The Plackett-Burman design is a square and orthogonal matrix in which each factor has two levels: a low level (-1) and a high level (1). Since there are 9 factors to consider, the Hadamard matrix or Plackett - Burman for which the number of experiences is a multiple of 4, near and above 9, is 12. So for this study, the Plackett-Burman matrix was comprised of 12 experiences and 11 factors including 9 factors assigned (independent variables) and 2 factors unassigned (Claeys-Bruno et al, 2009 ; Horadam, 2007 ; Rao et Diakar, 2001 ; Ghanemet al, 2000 ; Yu et al, 1997 ; Goupy, 1995; Davies, 1993 ; Goupy, 1990 ; Plackett et Burman, 1946). The nine (9) factors tested in this work during osmotic dehydration of fruits were: concentration, temperature, thickness, agitation, salt content, type of fruit dehydrated, ratio solute/ product, molecular weight and rinsing. Table 1 illustrate the independent variables and their corresponding level and value used in the Plackett-Burman design

Table 1: Factors and their corresponding level and value used during osmotic dehydration

| Independent variables | Symbol | Experimental value | |
|-----------------------|--------|--------------------|-------------|
| | | Low (-1) | High (+1) |
| Molecularweight | X1 | Sucrose | Glucose |
| Agitation | X2 | Yes | No |
| Salt content | X3 | 10% | 0% |
| Concentration | X4 | 70% | 30% |
| Température | X5 | 60°C | 30°C |
| Thickness | X6 | 40 mm | 15 mm |
| Ratio solute/ product | X7 | 1 / 9 (p/p) | 1 / 4 (p/p) |
| Type of fruit | X8 | Mango | Papaya |
| Rinsing | X9 | Yes | No |

The table 2 gives the Plackett-Burman design for 12 runs after replacing the codes level by their corresponding value (Khosraviet al, 2003 ; Rao et Diakar, 2001 ; Goupy, 1995).

Table 2: The Plackett-Burman design matrix experimental

| Runs | Variables | | | | | | | | |
|------|-----------|-----|--------|--------|---------|--------|-------|--------|-----|
| | X1 | X2 | X3 (%) | X4 (%) | X5(° C) | X6(mm) | X7 | X8 | X9 |
| 1 | Sucrose | Yes | 10 | 70 | 60 | 40 | 9 / 1 | Mango | Yes |
| 2 | Glucose | Yes | 00 | 70 | 60 | 40 | 4 / 1 | Papaya | No |
| 3 | Glucose | No | 10 | 30 | 60 | 40 | 9 / 1 | papaya | No |
| 4 | Sucrose | No | 00 | 70 | 30 | 40 | 9 / 1 | Mango | No |
| 5 | Glucose | Yes | 00 | 30 | 60 | 15 | 9 / 1 | Mango | Yes |
| 6 | Glucose | No | 10 | 30 | 30 | 40 | 4 / 1 | Mango | Yes |
| 7 | Glucose | No | 00 | 70 | 30 | 15 | 9 / 1 | papaya | Yes |
| 8 | Sucrose | No | 00 | 30 | 60 | 15 | 4 / 1 | Mango | No |
| 9 | Sucrose | Yes | 00 | 30 | 30 | 40 | 4 / 1 | papaya | Yes |
| 10 | Sucrose | Yes | 10 | 30 | 30 | 15 | 9 / 1 | papaya | No |
| 11 | Glucose | Yes | 10 | 70 | 30 | 15 | 4 / 1 | Mango | No |
| 12 | Sucrose | No | 10 | 70 | 60 | 15 | 4 / 1 | papaya | Yes |

2.4 Statistical analysis

Data analysis and figures were done using the softwares Microsoft Office Excel 2010 and Statistica 7.1. The chi square (χ^2) goodness-of-fit test and the mean relative deviation (MRD) were used to statistically evaluate if there is a significant difference in water loss or solute gain between the two levels of each variable during the osmotic dehydration. For the chi square test the difference is significant when the p-value is less than 0.05. The MRD value is given in percentage and may be estimated as follows:

$$E(\%) = \frac{100}{N} \sum_{i=1}^N \frac{|X_{exp} - X_{cal}|}{X_{exp}} \quad (3)$$

where values below 10 % are indicative of good fitness (Lomauro et al., 1985 ;Akmel et al, 2015). If that is not the case, there is a significant difference in water loss or solute gain between the two levels for the variable.

III RESULTS AND DISCUSSION

3.1. Influence of the molecular weight

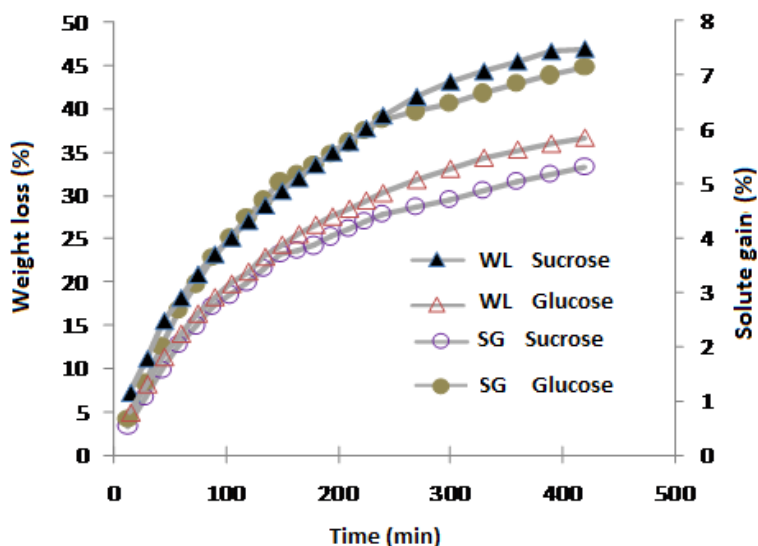


Figure 1: Water Loss (PE) and solute gain (GS) as a function of molecular weight

The weight loss and the solute gains are increasing (Figure 1). After 420 min (7 hours) osmosis time, there is a loss of 47% of the weight of the sample with sucrose and 36% with glucose. After the same time the solute

gains of dehydrated fruits in the glucose solution (6.15%) is higher than those dehydrated in sucrose solutions (5.35%). These differences are confirmed statically by chi square test for the weight loss (0.023) and for the solute gain (0.021) that are less than 0.05 and also by the MRD for the mass loss (25.77%) and for the solute gain (21.35%) that are higher than 10% (Table3).

The molecular weight of the solutes could be taken as a factor that significantly affects the water and solute diffusion in the fruits. These results were confirmed by the works of Ade-Omowaye (2003), Yao and Le Maguer (1997) which showed that these diffusions are influenced by the molecular weight of the solute.

Table 3 : statistical analysis results of chi square test and MRD

| Factor | Weightloss | | Solute gain | |
|-------------------------|------------|---------|-------------|---------|
| | p- value | MRD (%) | p- value | MRD (%) |
| Molecularweight | 0.0231 | 25,77 | 0,0215 | 21,35 |
| Agitation | 0,9991 | 9,87 | 0,2731 | 7,86 |
| Salt content | 0,6158 | 6,21 | 0,0618 | 5,61 |
| Concentration | 0,0001 | 27,75 | 0,003 | 22,46 |
| Temperature | 0,0001 | 27,97 | 0,0045 | 21,25 |
| Thickness | 0,9996 | 9,76 | 0,1379 | 6,12 |
| Ratio solute/ product P | 0,9999 | 3,89 | 0,8923 | 4,57 |
| Type of fruit | 0,8442 | 7,73 | 0,5122 | 6,16 |
| Rinsing | 0,2649 | 5,84 | 0,0173 | 19,83 |

3.2. Influence of the agitation

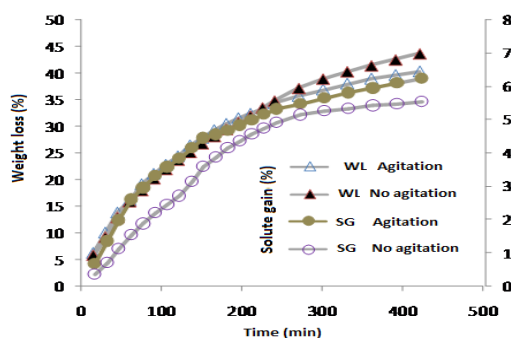


Figure 2: Water Loss (WL) and solute gain (SG) as a function of agitation

The kinetics of weight loss and solute gain versus agitation or without agitation of the osmotic solution are increasing. After 420 minutes of osmosis time, the solute gain and weight loss reach 6.23% and 44% respectively for the samples agitated against 5.50% and 42% for samples without agitation (Figure 2). The statistical results show that agitation does not significantly affect the osmotic dehydration because no significant difference was observed on the solute gain and the weight loss, based on the values of chi square test (p-value>0.05) ; and MRD (<10%) (Table 2).

3.3. Influence of the salt content

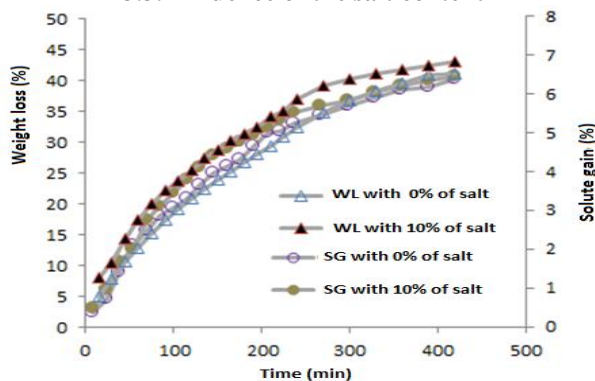


Figure 3: Water Loss (WL) and solute gain (SG) as a function of salt content

The kinetics of the sample weight loss in the 10% solution of salt (43%) is higher than that of weight loss (40%) in the salt-free solutions. After 420 min of osmosis time, it is found that the increasing rate of gain in solute of the sample reaches 6.85% for 10% solutions of salt and 5.36% for the salt-free solutions (Figure 3). The statistical results show that salt content does not significantly affect the osmotic dehydration because no significant difference was observed on the solute gain and the weight loss (p-value>0.05 ;MRD <10%) (Table 2).

3.4. Influence of solute concentrations in the osmotic solution

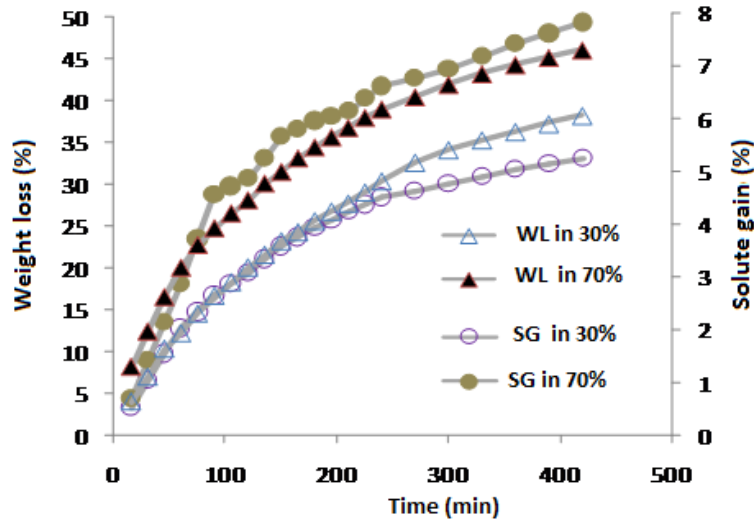


Figure 4: Water Loss (WL) and solute gain (SG) as a function of concentration

The curves relating to fruit solute gains, show that the gains in the solutions of 70% concentration are above those of the solutions of 30% with a gain of 7.82% against 5, 26%. Regarding the weight loss, fruit dehydrated in the solutions of 70% concentration have higher weight loss of 46% after 420 min osmosis time than those dehydrated in the solutions of 30% concentration which lost 38% of thier weight (Figure 4). Equally, statistical analysis shows very highly significant differences among the weight loss (p-value<0.001 ;MRD>10%) and between the solute gain (p-value<0.001 ;MRD >10%) due to the influence of solute concentrations (Table 2). These results are consistent with those of Andrade et al (2007), Alves and Coelho (2006), Nieto et al (2004) and Mauro and Menegalli (2003) that have shown that the concentration influences the osmotic dehydration.

3.5. Influence of the temperature

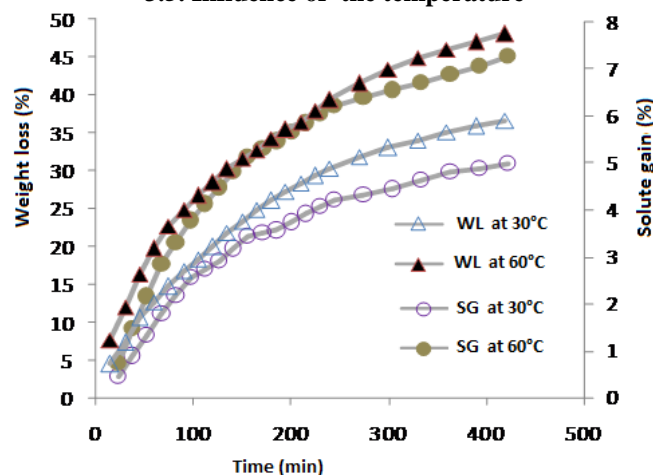


Figure 5: Water Loss (WL) and solute gain (SG) as a function of temperature

The weight loss are more important at 60 °C (48% of loss) after 420 min of osmosis time than what obtain at 30 °C (37% of loss). Concerning solute gain, it is observed that the solute gains at 60 °C (7.26%) are above the

solute gains at 30 °C(4.98%) (Figure 5). According statistical analysis there a very highly significant differences among the weight loss (p-value<0.001 ;MRD>10%) and between the solute gain (p-value<0.001; MRD >10%) due to the influence of Temperature (Table 2). These results are consistent with those of Chanloet al (2006) ,Madhiyanonet al (2006), Khoyi and Hesari (2007) and Janaiet al (2008) that have shown that the temperature of osmotic solutions significantly influences these diffusions.

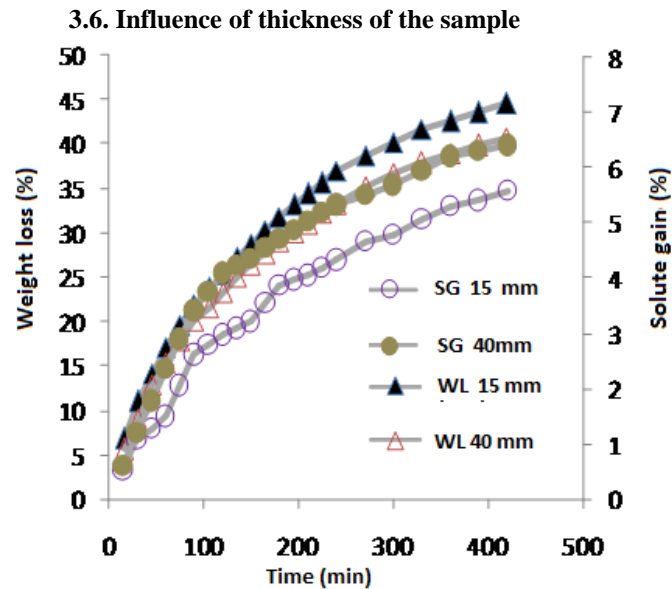


Figure 6: Water Loss (WL) and solute gain (SG) as a function of thickness of the sample

The weight loss curves of the samples from 15 and 40 mm in thicknesses are increasing and almost confused. Concerning the solute gain, the samples from 40 mm in thicknesses have a gain solutes of 6.41% above than those from 15 mm in thicknesses with 5.09% gain after 420 min osmosis time (Figure 6). The statistical results show that thicknesses does not significantly affect the osmotic dehydration because no significant difference was observed on the solute gain and the weight loss (p-value>0.05 ;MRD<10%) (Table 2).

3.7. Influence of ratio solute/ product

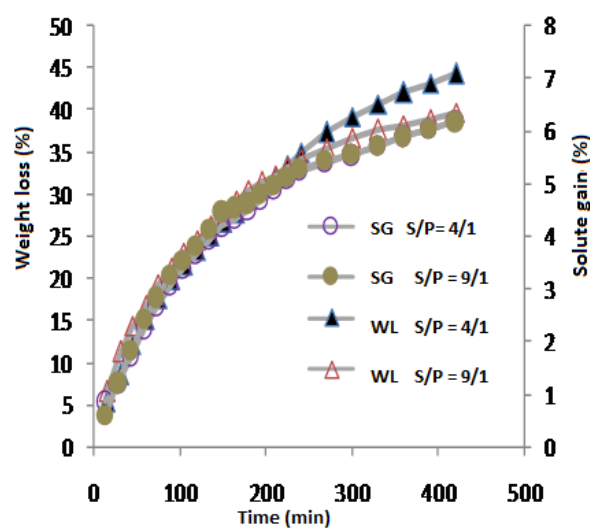


Figure 7: Water Loss (WL) and solute gain (SG) as a function of ratio solute/ product

The samples weight losses and their solute gains from the ratio solute / product are growing and almost confused (Figure 7). However, the samples dehydrated in 9/1 solutions have water losses and solutes gains

which are slightly above. The statistical results show that there is no significant difference among the solute gain and among the weight loss ($p\text{-value}>0.05$; $MRD<10\%$) (Table 2). The observed variance between the curves after 420 min of osmosis time are quite minor. Therefore, the ratio solute/product does not affect significantly the osmotic dehydration.

3.8. Influence of type of fruit

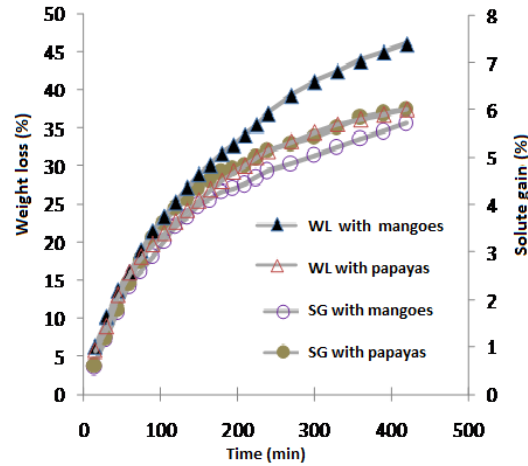


Figure 8: Water Loss (WL) and solute gain (SG) as a function of type of fruit

The observation of the curves show that weight losses of mangoes are slightly above those of the papayas. Concerning solute gains, the kinetics are almost confused (figure 8). These observations are consistent with the statistical results ($p\text{-value}>0.05$; $MRD<10\%$) (Table 2). Therefore, the type of fruit does not affect significantly the osmotic dehydration.

3.9. Influence of rinsing

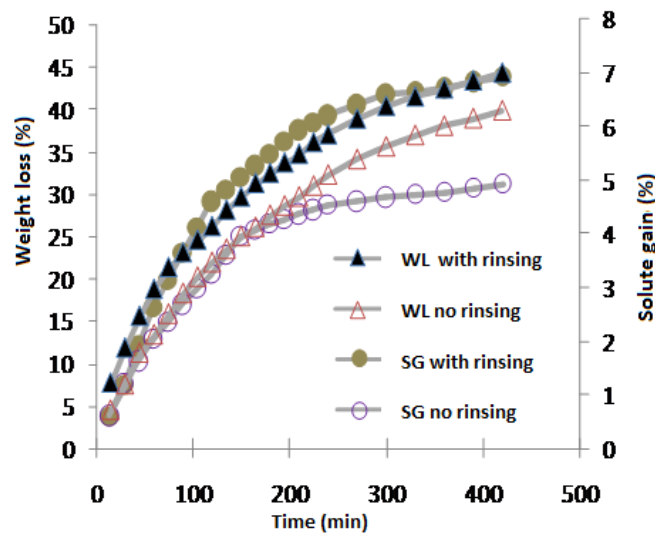


Figure 9: Water Loss (WL) and solute gain (SG) as a function of of rinsing

The analysis of figure 9 show that the gains are more important for rinsed samples (6.93% of gain) after 420 min of osmosis time than samples which are not rinsed (4.92% of gain). This is consistent with the statistical results which show a significant difference among the solute gains ($p\text{-value}<0.05$; $MRD >10\%$). Concerning the weight loss, the figure 9 shows that the weight loss of samples rinsed or not are growing up to 44% of weight loss for rinsed samples and 40% for samples not rinsed. As in the previous cases, the statistical results show that there is no significant difference among the weight losses ($p\text{-value}>0.05$; $MRD<10\%$) (Table 2) .The observed variance between the curves after 420 min of osmosis time are quite

minor. Therefore, the rinsing does not affect significantly the osmotic dehydration, more precisely at weight loss level.

Indeed, The results obtained about agitation, salt content, thickness, ration solute/product, type of product and rinsing are consistent with the works of Mavroudis et al (1998) et Heng et al (1990) that have shown that these factors cannot be used as factors influencing significantly the diffusons.

IV- CONCLUSION

It appears from this study that the weight losses and solute gains of the dehydrated fruits increase with the time for all factors. As regards mass loss, only factors such as molecular weight of the solutes, the concentration of the osmotic solution and the temperature affect significantly the water diffusion and hence the osmotic dehydration of the fruits. As the regards solute gain, factors such as the molecular weight of the solutes, the concentration of the osmotic solution, the temperature and the rinsing affect significantly the water diffusion and hence the osmotic dehydration of the fruits. Concerning the other factors, they have not an impact significantly on the osmotic dehydration because the observed variance between the curves after 420 min of osmosis time are not statistically significant.

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