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Moisture diffusivity during hot-air drying of red prickly pear peel *(Opuntia streptacantha)*

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ABSTRACT Red prickly pear (*Opuntia streptacantha*) is an important fruit in Mexico. Peel is usually discarded, however, biocompounds should be extracted from it in order to give added-value to this by-product. Hot air drying keeps as an easy, cheap and suitable method to preserve organic material, for further extraction of compounds. The aim of this work was to study and model the hot air drying of red prickly pear peel using two temperatures (80 and 85 °C), during 6 h. The water activity in fresh and dry pulp was measured, as well as the moisture content and total soluble solids in fresh pulp. For drying, the air velocity was 2.27±0.04 m/s, for batches of 70±2 g. The peel was cut in strips with thickness of 0.009 m. The relative humidity (RH) was determined. Several empirical (Newton, Page modified, among others) and diffusional models were applied to describe the drying. Also, the biocompounds concentrations were evaluated. Fresh pulp had 0.9607±0.003 for water activity, 89.3±1.6% (w.b.) for moisture content and 11.5ºBx. The best correlation coefficient was achieved with the empirical Page model. The diffusion coefficients were 8.75 and 9.88×10^{-9} $m^{2/}s$ for 80°C and 85°C, respectively, at 28.1% RH. Activation energy was 11.95 kJ/mol. Dry peel had water activity between 0.29 and 0.30, and moisture content between 9.22 and 12.26% (w.b.). In dry peel (per gram of dry matter): total phenolic compounds were 18-27 mg equivalent gallic acid/g, total flavonoids 40-53.7 mg quercetin/g, betalains 0.5-0.8 mg/g and for anthocyanin 3.4-22.8 mg equivalents cyanidin 3-glucoside/g.

Keywords: Mathematical models, Red prickly pear peel, Drying, Diffusion.

INTRODUCTION The prickly pear fruit belongs to the *Opuntia* specie and to the *Cactaceae* family, it usually grows in dry weather (Sumaya-Martinez, 2010). The main countries where it grows are Mexico, Argentina, Peru, Bolivia among others (Sáenz *et al.,* 2004). Mexico is considered as the main source of *nopal* (*Opuntia* spp.) and therefore, the prickly pear fruit as well; it is the country with the highest quantity of species of prickly pear. One of these species is the red prickly pear fruit (*Opuntia streptacantha*) (Méndez & García, 2006), which is one of the most consumed. Therefore, the quantity of waste (peels) that are created is high. Nowadays, the interest towards the prickly pear fruit has increased due to the nutrients and antioxidants that it may provides. The nutritive importance is due to the ascorbic acid, fiber, amino acids, phenolic compositions and the presence of betalains found in both the pulp and the peel (Piga *et al*., 2003). Nevertheless, the components in the peel have not been reported (only the bio-components in the pulp). The substances obtained from the peel can be used in the food industry in order to offer value added products. One alternative to keep the waste could be through a drying procedure (Nóbriega *et al.,* 2013).

The drying process is one of the most important methods used in the food industry to the preservation of food; which reduces the water activity levels and the humidity. Thus, this avoids the pollution and deterioration of the food (Kaymak-Ertekin, 2002). There exist different methods to dehydratase food such as solar-drying, spray-drying, osmotic-drying and drying in hot air among others. The main objectives of drying are the volume and weight reduction or as a pervious treatment in some techniques or to the concentration of nutrients in some food. For instance, the bioactive components which are found in fruits, vegetables and plants (Vega *et al.,* 2007).

There are diverse factors affecting the drying in hot air in food, for instance, the air velocity and temperature, the water diffusion through the food, the thickness and the shape of the food. However, the water removal affects the nutritional and sensitive quality of the food. Some mathematic models have been proposed to describe the drying process. When the drying in hot air process is controlled by the transference of inner mass, the drying model is done through diffusion equations based on the second law of Fick, which objective is to determine the effective coefficient of the water diffusion; which is directly related with the drying conditions such as velocity and temperature within the dryers (Vega *et al.,* 2007). The empirical equations frequently used for drying kinetic models include, Newton, Page modified, logarithmic, Henderson and Pabis among others (Vega *et al.,* 2007). The objective of this research is to determine the best drying temperature for the red prickly pear fruit peel so the concentrations of bioactive compounds (total phenols, total flavonoids, betalains (betanins, betaxanthins) anthocyanins) remains as the minimum. Moreover, the modelling of the drying kinetic models and the mass transfer during the hot air drying of the red prickly pear fruit peel was analyzed, besides the influence of the temperature over the variables of the proposed models.

MATERIALS AND METHODS Red prickly pears (*Opuntia streptacantha*) were obtained in the Central Market located in Irapuato city in the state of Guanajuato, Mexico in the months of September-November 2016. They were washed and peeled and the recovered peels were cut in stripes.

Red prickly pear peel characteristics Dimensions were measured using a Vernier. Total soluble solids (expressed as ºBrix in the pulp) were measured with a refractometer (model HI 9680, Hanna instruments, Romania). Water activity was determined in an electronic hygrometer (Aqualab, Decagon Devices, USA) and the moisture content was done with the method 925.10 (AOAC, 1996). These assays were done by triplicate before and after the drying process of the peel.

Hot air-drying process A sample of 70 ± 2 g were placed in a drilled trial to perform the drying process, using a tunnel hot air dryer with a electric resistance of 2000 W, equipped with a fan of 170 W and an airflow of 233 m^3/h; the dryer was designed and built by the Universidad de Guanajuato's Food Department. The drying process was done using three different temperatures

(85, 80 y 65 ± 2 °C during 6 h for the first two temperatures and during 9 h for the last temperature, the stripes were collocated with the inner part facing up. The weight of the peels was registered every 20 min.

To determine the weights of the stripes, it was used a digital scale (VELAB balances, model VE-5000). Finally, the air velocity was measured using an anemometer (AIRFLOW, model TA3, United Kingdom) which gave us the result of 2.27±0.04 m/s.

RH assay Both dry bulb and wet bulb temperatures were measured every 20 min within the drying process. We obtained an average every drying round for every temperature and it was determined the RH using a psychrometric chart of 1 atm. In order to obtain the final RH it was obtained the average of the RH of the others drying rounds.

Drying models The moisture ratio was obtained using the following equation:

$$MR = \frac{(M - Me)}{(Mo - Me)} \tag{1}$$

In order to select the appropriate model that describes the drying process of red prickly pear peel stripes, the drying wheels were adjusted to 10 drying process, the evaluated models are presented in the Table 1. The nonlinear regression analysis was done using Excel, where the correlation was the most important parameter to choose the best model.

Table 1. Mathematic models used to estimate the kinetics of drying.

Modelos	Ecuación
Newton	XR= exp (–kt)
Page Modificado	XR= exp [-(–kt)^n]
Logarítmico	XR= a*exp (–kt) + c
Henderson and Pabis	XR= a* exp (–kt)
Dos términos (Henderson)	XR= a*exp (–kt) +b * exp (–gt)
Wang and Singh	XR= at^2 + bt + 1
Thompson	XR= a*[ln(XR)]^2 +b*Ln (XR)
Aproximación de difusión	XR= a*exp (–kt) + (1-a) exp (-kbt)
Midilli- Kucuk	XR= a*exp (–kt^n) + bt
Verma	XR= a* exp (–kt) + (1-a)* exp (-kt)

In order to follow the diffusivity of the moisture during the drying process, Fick´s equation for particles with plate geometry as applied:

$$MR = \frac{8}{\pi^2} \exp\left(\frac{-\pi^2 Defft}{L^2} \right) \tag{2}$$

Assay of the concentration of bioactive compounds The assays were done on fresh and dry peels at 80 and 85 °C, for the 4 tests of assay of the concentrations of bioactive compounds: total phenolic compounds (TFC), total flavonoids (TF) anthocyanins and betalains. We did a previous extraction through peel maceration both fresh and dry using 10.0 ml of methanol at 80% for the TFC and TF and 8.0 ml for the betalains and 0.2 g of dry peels or 1.0 g of fresh peel depending on the type of simple, this was for the total phenolic compounds, flavonoids and betalains; for the anthocyanins, the methanol was substituted for cold acetone. All the assays were tripled done.

Total phenolic compounds assay. This assay was done by the Folín-Ciocalteu method with assistance of a spectrophotometer (Perkin Elmer, model: Lambda XLS). It was measured the absorbance with a wavelength of 765 nm. We used a calibration curve of gallic acid in order to determine the total phenolic concentration (Slinkard y Singleton, 1977).

Total Flavonoid Assays. It was determined through a spectrophotometric method, to measure the absorbance it was used a wavelength of 415 nm. It was used a calibration curve of quercetin to estimate the concentration of total flavonoids (Khanam *et al.,* 2012).

Total anthocyanins assay. It was done through the pH Differential method, to determine the absorbance it was used a spectrophotometric method with a wavelength of 515 and 700 nm (Pasko *et al.,* 2009).

Total betalains assay. For this assay, betacinanins (betanins) and betaxanthins (vulgaxanthin-1) were quantified using a spectrophotometric method reported by Nilsson (1970). They will be measured with a wavelength of 538 nm, 470 nm respectively.

Statistics analysis It was done an ANOVA, a way to determine the drying effect over the physicochemical characteristics, as well as the effect over the bioactive compounds concentrations. It was done a Tukey test with a significance level of 95%. The data was analyzed using the Software analysis known as Statgraphics Centurion.

RESULTS AND DISCUSSION DISCUSSION In Table 2 there are presented the physicochemical results of the red tuna peel before and after the thermal treatment that was applied in order to dry it. In terms of the percentage of the humidity, it is presented that the composition of the fresh peel presents high amounts of water. The treatment presents significant statistics differences in the samples of 85 and 65 with the one of 80°C. The humidity percentage registered in the tuna peel shows an inversely proportional relation with the drying time applied to such samples. However, the results show that the drying temperature also influenced the humidity. This is because we obtained a lower value of humidity with a higher drying temperature at the same time.

For the water activity, it is also observed a considerable reduction of the value before and after the treatment. However, there are not significant statistical differences between the treatments ($p \geq 0.05$). Total soluble solids of the pulp were of 11.5 °Bx. The result was lower in comparison with the obtained by Stintzing *et al.* (2005), who reported 14.8 °Bx for the juice of a ripe red prickly pear fruit.

The Relative Humidity (RH) for drying, observed by the use of psychometric chart in 1 atm, was 28.1%.

Table 2. Comparison of physicochemical properties between fresh red prickly pear peel and dried at three temperatures.

Parameter	Fresh peel	Dried peel		
		65 °C	80 °C	85 °C
Humidity (%)	89.33 ± 1.6	8.86 ± 0.33 ^{a}	12.26 ± 0.87^{b}	9.22 ± 0.34^{a}
Water activity	0.970 ± 0.003	0.297 ± 0.004^{a}	0.307 ± 0.009^{a}	0.294 ± 0.015^{a}
Dimensions (mm)	9.7 ± 0.09		1.5	

Statistical Analysis only was applied to the samples with thermal treatment.
Bracketed Literal with different values show a significant difference ($p \leq 0.05$).

Bioactive compounds concentrations The assay of bioactive compounds concentrations (phenolic compounds, total flavonoids, betalains and anthocyanins) before and after the drying process was done in order to know the effect that the hot air-drying process has over the bioactive compounds in the peel.

In the Figure 1 it is presented a graphic of the total phenolic compounds concentrations (TPC) reported in a dried base, where it is observed that the drying process had a negative effect due to the treatment, This is because it was observed a reduction of the bioactive compounds after the thermal treatment, presenting the highest value the fresh peel with 2,7.73 mg similar to the gallic acid (GAE)/ g dried weight peel as the dried weight peel at 80°C and finally the dried at 85 °C, which means that when the temperature is raised there exist and increases the loss of TPC. Therefore, it actually exists a significant difference ($p \leq 0.05$) between the three types of samples previously mentioned.

This reduction of TPC caused by the temperature mirrors the study reported by Nóbrega *et al.* (2015), who worked with acerola waste, a very common fruit in Brazil and its results prove that during the thermal treatment to the waste there was a reduction of almost 80% of the TPC. These results are also analogic of the study done by Mejía-Meza *et al*. (2010) who reported that the content of TPC of raspberry had a significant reduction after being dried with hot air under similar low conditions. This decreasing effect in the concentration of TPC can be justified because the compounds are susceptible to the thermal reduction. However, contrasting the results in the study done by Ching-Hui *et al.,* (2006) it is presented an increase in the total phenolic compounds or more than 11% in tomatoes after being dressed regarding the fresh tomato.

Figure 2 describes the concentrations of the total flavonoids, these results present the same inverse behavior than the TPC, this means that it is presented a higher value in the results of the peel previously dried in comparison with the peel without thermal treatment. The peel dried at 85°C presented a maximum value with the concentration of 53.74 mg similar to the quercetin/ g dried weight, this value was very closed to the result obtained with the dried peel at 80°C, which there is not presented a statistical difference among them. Nevertheless, with the fresh peel it is presented a significant statistical difference because the obtained result was almost ten times lower than the one of the dried peel at 85 °C. Therefore, we can conclude that the temperature does not affect the TF, in contrast, it increases the concentration when there is presented a reduction in the water.

The behavior presented in the dried red prickly pear peel was similar to the one presented in the studio done by Ching-Hui *et al.* (2006), where it was evaluated the flavonoid concentration and similar to the results obtained in this study, it is also presented an increase in the content of the total flavonoids when it was done the treatment of hot air drying. This can be explained by the biochemical reactions that occur as in the peel as in the pulp during the drying process, which results in the increase of TF.

The concentrations of anthocyanins could be observed in the Figure 3, where are presented in the concentrations obtained after the drying treatment (80 and 85 °C). These results do not present significant statistical differences since they are very similar to the values until 4.49 mg equivalent to the cyanidin 3-glucosido/ g of dried weight. In the research done by Nóbrega *et al.* (2013) the acerola was dried at 70 and 80°C, in its results it is presented a reduction in the concentration of anthocyanins of 20 % when the drying is at 80 °C compared with the one at 70 °C; therefore, the fact that are not significant statistical differences in this study can be because the temperature range is lower compared to the study by Nóbrega *et al.* (2013).

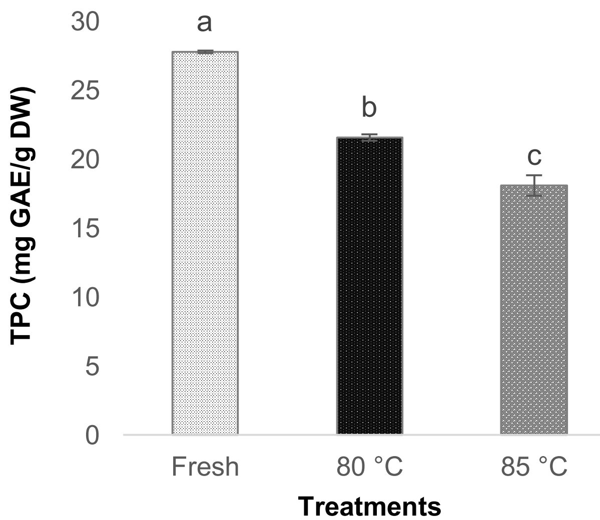

Figure 2. Concentration of TPC (dry weight) in fresh and after drying treatment (80 and 85 ° C) red prickly pear peel The error bars indicate the standard deviation, where n = 3. The different literals indicate significant difference (p≤0.05)

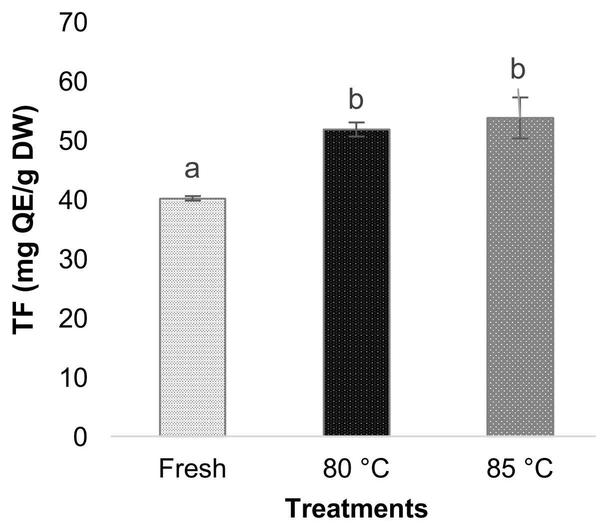

Figure 1. Concentration of FT (dry weight) in fresh and after drying treatment (80 and 85 ° C) red prickly pear peel. The error bars indicate the standard deviation, where n = 3. The different literals indicate significant difference (p≤0.05)

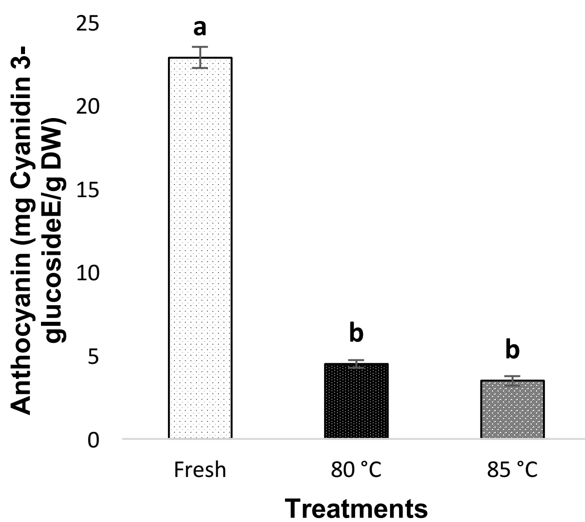

Figure 3. Concentration of anthocyanins in fresh and after drying treatment (80 and 85 ° C) red prickly pear peel. The error bars indicate the standard deviation, where n = 3. The different literals indicate significant difference (p≤0.05).

In the Figures 4 and 5 are presented the concentrations of betacianins (betanins) and betaxanthins (vulgaxanthins-1). Referring to the betacianins are presented statistical differences between the two treatments, with the 85 °C the one that presents the higher quantity of betacianins with 0.71 mg similar to the ones of betanins/ g of dried weight of dried peel. Referring to the betaxanthins (vulgaxanthins-1) is also using the temperature of 85°C was when is presented a highest concentration of 0.12 mg similar to the vulgaxanthins-1/ gr of dried weight of dried peel. Obtaining a total of 0.83 mg of betalains/ g of dried weight of dried peel, when it is dried at 85°C y 0.50 mg of betalains/ g of dried weight of dried peel when it was dried at 80°C. The fact that is presented a higher concentration of betanins, it was an expected result, this is because these are the ones that provide the reddish color to the fruits where it is presented.

In the study done by Gokhale and Lele (2012) they tested two drying temperatures at 80 and 100 °C and they determined that the assays of betalains (betacianins and betaxanthins) in beetroot, in their results they observed that the temperature at 100 °C there is a reduction in the concentrations of betacianins, in a contradiction with the results of this study, where it is used a higher temperature there are presented higher levels of concentration; nevertheless, this can be due to the high temperature used in the beetroot. Referring to the betaxanthins, the behavior was similar to the study done by Gokhale and Lele (2012), because similar to their study, there was an increase in the concentration when it was used the highest drying temperature, which means that the betaxanthins are less sensitives to the higher drying temperatures. This is why they are better kept during the drying process

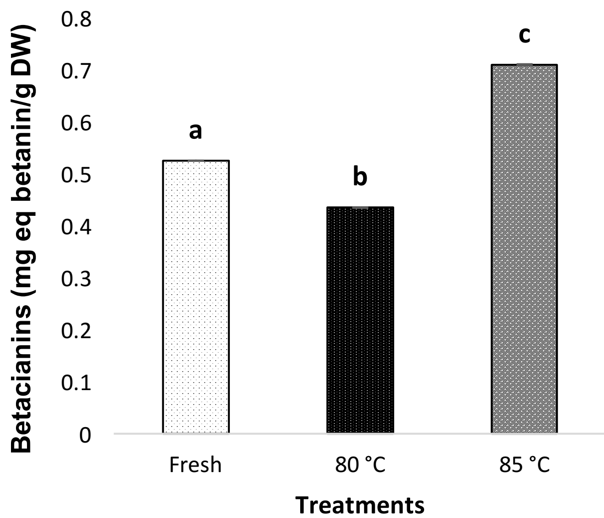

Figure 4. Concentration of betacianins in fresh and after drying treatment (80 and 85 ° C) red prickly pear peel. The error bars indicate the standard deviation, where n = 3. The different literals indicate significant difference (p≤0.05).

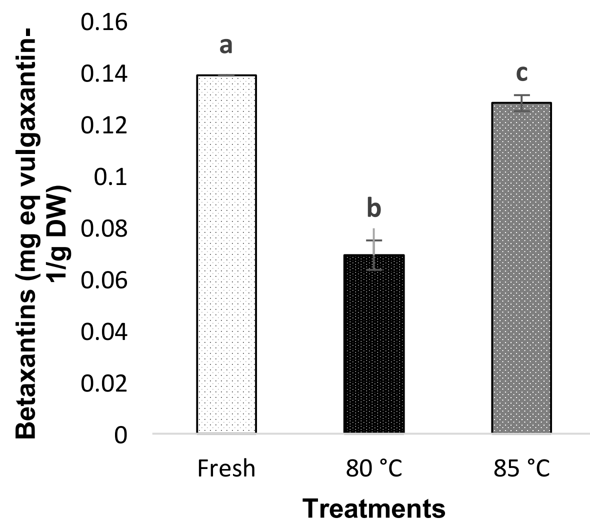

Figure 5. Concentration of betaxantins in fresh and after drying treatment (80 and 85 ° C) red prickly pear peel. (The error bars indicate the standard deviation, where n = 3. The different literals indicate significant difference (p≤0.05).

Mathematic models In the Figure 6 there are presented the drying curves of the red prickly pear peel with the three drying temperatures, the moisture ratio was plotted in the function of time. Moisture decreased continuously with the drying time, taking 300 and 360 min to complete the drying.

The correlations obtained among the experimental values and the calculated (R^2) was satisfactory for eight out of 10 applied models (Table 3). Nevertheless, the treatments where are presented the values of (R^2) lower to the dried peel at 65 °C since it is present the correlation coefficients of 0.95 in the Henderson and Pabis model. However, it is still considered that it adapts to that model. Similarly, the percentage of variance of the dried peel at 85 °C was higher presenting a good percentage of variance for the other two treatments taking the value of even 99.59% and a value of R^2 of 0.99 being almost a perfect adjustment with the models. Mosqueda *et al.* (2016) adjusted the drying kinetics with hot air in nopal and tomato with the same models; however, the kinetics that obtained only were adjusted to five models, being the one of Midilli-Kucuk.

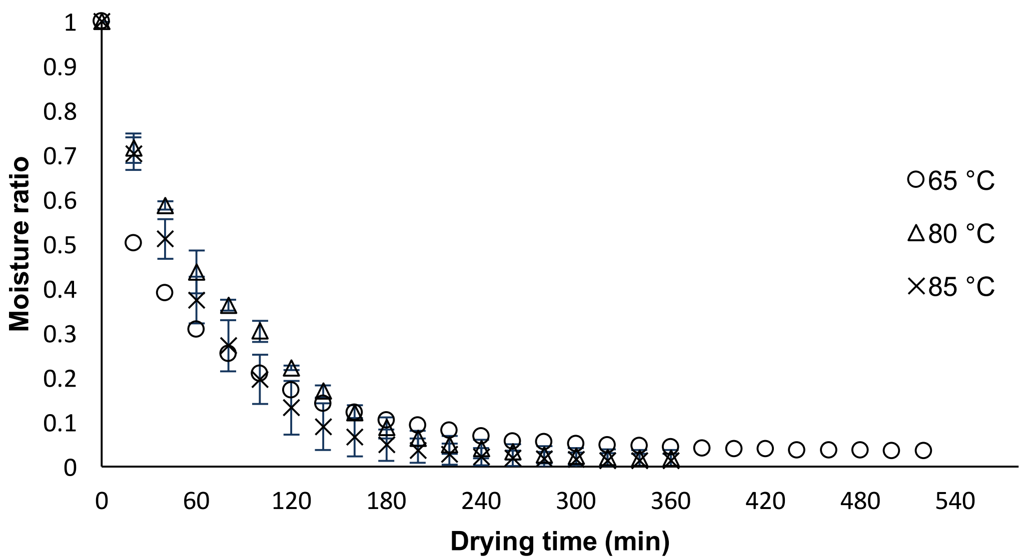

Figure 6. Drying curves of the three drying treatments, with their respective standard deviations

The diffusion coefficients during drying of the red prickly pear peel processed at 65, 80 and 85 °C are presented in the Table 4. At 85 °C, the highest value was observed.

The diffusivity depends on the temperature and it can be represented by the Arrhenius equation. The diffusivity logarithm presents an almost linear behavior over the absolut factor of temperature when it is presented a minimum R^2 value minimum of 0.94 (Table 4). The obtained values in the coefficient diffusion were lower to the obtained by Mosqueda *et al.,* (2016) which were of 8.65×10^{-7} and 2.45×10^{-7} for tomato and nopal respectively. This can be due to the quantity of water presented in the red prickly pear peel is lower compared to the quantity of nopal and tomato; this is why the diffusivity is lower. Nevertheless, the results obtained in this Project are very similar to the ones reported by Vega *et al.* (2007) which was of 6.9×10^{-9} drying at 60 °C and 11.2×10^{-9} drying at 80 °C. Comparing the results obtained we can deduce that the diffusivity of water of the red prickly pear peel is very similar to the diffusivity of the red pepper.

Table 3. Comparison of the mathematic models for the drying kinetics at different temperatures.

Model	Dried peel at 65 °C		Dried peel at 80 °C		Dried peel at 85 °C	
	% variance	R^2	% variance	R^2	% variance	R^2
Newton	71.65	0.98	99.20	0.99	99.90	0.99
Page modified	71.66	0.96	99.20	0.99	99.90	0.99
Logaritmic	90.76	0.96	99.34	0.99	99.91	0.99
Henderson and Pabis	78.11	0.95	99.34	0.99	99.90	0.99
Two terms (Henderson)	99.17	0.99	99.59	0.99	99.92	0.99
Diffusion aproximation	99.70	0.99	99.59	0.99	99.92	0.99
Midilli-Kucuk	80.40	0.95	99.23	0.99	99.88	0.99
Verma	71.65	0.96	99.20	0.99	99.90	0.99

Table 4. Coefficients of water diffusion in red prickly pear peel obtained at three different temperatures.

Temperature (°C)	Diffusion Coefficients (m^2/s)	(R^2)
85	$9.88306E-^{9} \pm 3.63E^{-9}$	0.94
80	$7.6512E^{-9} \pm 0.0$	0.97
65	$0.76512E^{-9}$	0.95

The activation energy required was of 11.95 kJ/mol, lower than reported by Vega *et al*.(2007) for drying of the red pepper was of 39.70 kJ/mo.

CONCLUSION The temperature of 85 °C was the most appropriated to perform the drying, showing the highest values of total flavonoids, betacianins and betaxanthins (betalains).

The drying kinetics with the temperature of 85 °C had well-adjust to mathematical models, therefore, it is still the best to predict the drying process and to perform the drying of the red prickly pear fruit peel. This temperature turned to be the one that presented the highest diffusivity of water.

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