

Determination of Impregnation Values of Some Postharvest Apple Kinds Under Different Vacuum Pressures

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Abstract: In this study, Golden Delicious, Starking Delicious and Granny Smith kind apples were observed under different vacuum pressures. The effect of vacuum pressure and vacuum period was investigated and results were evaluated graphically and statistically.

The biggest X (Impregnation liquid fraction) values were determined in Starking Delicious(11.02), and the lowest values were determined in Golden(3.74) and Granny(4.31) Apples. According to observations suitable vacuum pressures for each one was evaluated. For Golden, 50 kPa and 60 kPa($R^2 \geq 0.99$), Starking 50 kPa($R^2 \geq 0.98$) and Granny 60 kPa ($R^2 \geq 0.98$) was determined. For each of these apples working under these vacuum pressures would give more favorable results.

Keywords: Postharvest apple, vacuum pressure, infiltration, impregnation, calcium

INTRODUCTION

Low fruit calcium levels have been associated with reduced postharvest life and physiological disorders(Wills et al., 1998). For example, low levels have been correlated with physiological disorders of avocados(Chaplin and Scott, 1980), papaya(Qiu et al., 1995), apples (Conway et al., 1992) and mangoes(Van Eeden, 1992). Delayed ripening in response to increased fruit calcium levels has been obtained with apples(Klein and Lurie, 1994) and avocados(Wills and Tirmazi, 1982). Pressure infiltration of $CaCl_2$ solutions mainly affects Ca concentration of tissue near the surface of treated fruit. This effect varies depending upon the sampled tissue, with the greatest increase in Ca concentration being in the cortex near the epidermis (Conway and Sams, 1987).

Postharvest Ca treatments used to increase Ca content of the cell wall were effective in delaying senescence, resulting in firmer, higher quality fruit (Sams et al., 1993) that were less susceptible to disease during storage (Conway et al., 1991). Many techniques to increase Ca content in the cell walls of

fruit tissue after harvest have been developed. These include heat treatment (Klein et al., 1997), dipping (Conway and Sams, 1983), vacuum infiltration (Rajapakse et al., 1992), pressure infiltration (Conway and Sams, 1985), surfactants or coating agents (Saftner et al., 1998), or a combination of these techniques (Klein et al., 1990).

Calcium chloride concentration of 2% was sufficient for maximum Ca accumulation in the tissue and cell wall. Between 4 and 6 months storage, significant changes in the cell wall occurred, probably resulting in fruit with lower quality and increased susceptibility to fungal diseases.(Catherine et al., 2002) Dipping fresh-cut products in solutions of 0.5–1.0% calcium chloride is very effective at maintaining product firmness (Ponting et al., 1971, 1972).

Very few studies have been carried out by using impregnation techniques for the development of functional foods with vegetable matrices. Anino et al.(2001) compared two impregnation methods(vacuum and atmospheric) for the incorporation of calcium in apple tissue. Fito et al.(2001) proposed a mathematical model to calculate

the physiologically-active food components concentration of the impregnation medium in order to formulate functional foods with the addition of different Ca and Fe salt and satisfy a specific percentage of the Recommended Daily Intake. These studies suggest that these impregnation methods could be used for the incorporation of physiologically-active food components in fruits and vegetables matrices.

Vacuum impregnation technology can be used to effectively incorporate solutes into porous, solid food matrices containing air, such as fruits and vegetables. This method involves holding food under vacuum to enhance air extraction from pores, then, immersing it in the desired solution. As a result of capillary forces, a limited amount of liquid penetrates the porous space adjacent to the liquid–solid interface; the bulk of liquid penetration, however, occurs following vacuum release. Vacuum impregnation (VI) technology, therefore, allows an impregnation solution to replace a portion of the air initially contained in the porous structure of food, as a result of the positive pressure differential that develops when atmospheric pressure conditions are restored. The hydrodynamic mechanism (HDM) enhancing liquid penetration has been precisely described and modelled (Fito, 1994; Fito et al., 1996). Vacuum impregnation has been used as an alternative to impregnation under atmospheric pressure to increase efficiency and save time. In previous studies (Degraeve et al., 2003; Guillemain et al., 2006),

Vacuum impregnation efficiency has been reported to depend on process parameters, including vacuum level and time, holding time in the impregnation solution after pressure release (Hoover and Miller, 1975); on the food matrix, including effective porosity (Mujica-Paz et al., 2003a) and tortuosity; and on impregnation solution properties, such as osmolarity (Mujica-Paz et al., 2003b) and viscosity (Barat et al., 2001; Saurel, 2004).

The X values were calculated from sample weight before and after each treatment. Loss of native liquid was neglected, so the calculated X value for mushrooms would represent the total external liquid penetrated into the tissue. An analysis of variance and multiple range tests were applied to establish significant differences between vacuum treatment.

From the mean values of X obtained, theoretical values of the calcium content finally achieved in the matrix was calculated taking into account the amount incorporated because of treatment and the amount initially present in raw material.(C.F. Ortiz, Salvatori and Alzamora, 2003)

This product composition as well as its physical and chemical properties may be changed to improve the properties of the final product. The impregnated liquid fraction (X) means the pore fraction of the matrix that can be penetrated by the external solution at the mechanical equilibrium status, and is a function of the product porosity and the applied vacuum pressure.

An additional of this micronutrient might be achieved by impregnation soaking processes. Vacuum impregnation treatment of a porous material consists of exchanging the internal gas or liquid occluded in open pores for an external liquid occluded in open pores for an external liquid phase (of controlled composition) due to pressure changes. Fruit and vegetables have a great number of pores occupied by gas or native liquid and offer the possibility of being impregnated by a determined solution.(Fito and Chirald, 2000)

MATERIALS and METHODS

Sample Preparation for Impregnation

Treatments

'Golden Delicious'(a_w = 0.99; 11.1–13.2 °Brix and pH 3.49–3.88), 'Starking Delicious'(a_w = 0.99; 11.3 – 12.4 °Brix and pH 3.1–3.5) and 'Granny Smith'(a_w = 0.98; 10.4 – 12.2 °Brix and pH 3.11–3.27) types of apples were harvested from a commercial garden in the province of Yalova,Turkey. The harvested fruits were classified with dividing into 7 groups according to their weights. (160-180 g) .%2 (w/v) CaCl₂ (99% CaCl₂.2H₂O Sigma Chemical Co.)(Carl E. Sams et Al. 2003) solution was prepared.

Vacuum Impregnation Treatments

An iation system was established in order to make the apples absorbe calcium under vacuum. The system has a capacity of 20 liters. The system consisted of elestrical motor, vacuum pump, vacuum chamber in which the apples were placed, manometers, valves hoses and the container with the

calcium solution inside. Schematic representation of laboratory type calcium impregnation system with vacuum is given in Figure 1.

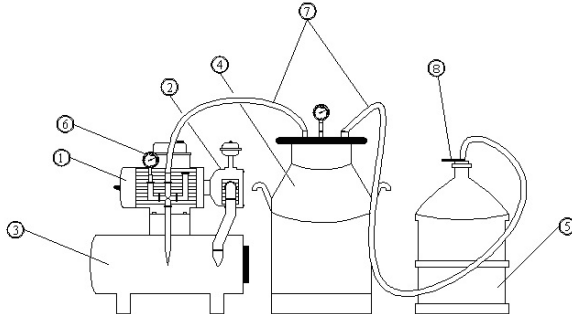


Figure 1. Schematic representation of laboratory type calcium impregnation system: (1) single-phase electrical motor; (2) vacuum pump; (3) vacuum store; (4) vacuum chamber in which the apples were placed; (5) container for CaCl₂ solution; (6) manometer; (7) transparent plastic hose; (8) spherical valve

The operating principle of calcium infiltration system is given in Figure 2(a-d).

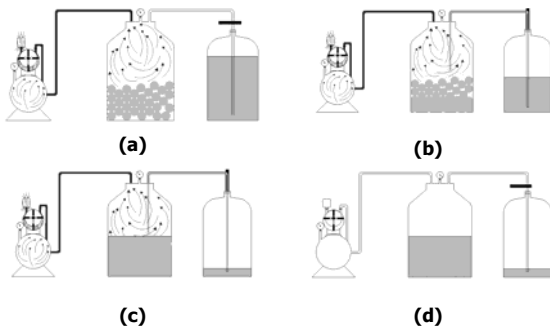


Figure 2. The operation of vacuum calcium infiltration system: (a) the desired infiltration pressure is obtained on the apples in the vacuum chamber; (b) and (c) the desired pressure (vacuum) value is reached in 5–10 s; apples are kept at this vacuum value for 2–4 min after the desired value is reached; thereafter, the valve on the calcium solution container is opened and the solution is pulverised onto the apples under vacuum in vacuum chamber; (d) then the valve and vacuum system on the calcium solution container is closed and the apples are held in the solution for 1, 3, 5, 7, 9 and 11 min

Prepared fruits, was put in impregnation vacuum system at the temperature of 22 degrees. At first ; fruits were weighed one by one in the scale of 1/1000 sensitivity, then their volumes were calculated.. 20, 30, 40, 50, 60 and 70kPa.(Del Valle et al. 1998) pressure was applied to the fruits grouped of seven

and this process was repeated 3 times. Also , They were kept in vacuum pressure in 2 and 4 minutes. (Scott and Wills, 1977, 1979). Every group of fruit was kept in a solution of %2 (w/v) CaCl₂ (99% CaCl₂.2H₂O Sigma Chemical Co.)(Carl E. Sams et Al. 2003), for a period of 1, 3, 5, 7, 9 and 11 minutes. After this process was finished, Fruits removed from impregnation system were put on a kraft paper for 60 minutes. Then fruits were weighed one by one. 648 attempts were made for a total of 3 kinds of fruit. The volume of the fruit impregnated with the IS (X) was determined as reported by Salvatori (1997).

$$X = \frac{M_f - M_i}{\rho_s V_0}$$

- X : volume of the fruit impregnated by an external liquid (cm³ liquid/cm³ fruit)
M_f : final mass of the fruit (g)
M_i : initial mass of the fruit (g)
V₀ : initial volume of the fruit (cm³)
ρ_s : density of the solution (g/cm³)

Design and Statistical Analysis

A design was used to determine the effect of the vacuum pressure and impregnation time on the quantity of the solution impregnated in the fruit . Experimental conditions are presented in Table 1. For each of the experimental points, after the impregnation process, a 60 min relaxation period at atmospheric pressure was applied.

Table 1. Experimental conditions

							Vacuum Time(min)
Solution Dipping Time(min)(2% CaCl₂)	1	3	5	7	9	11	2
Vacuum Pressure(kPa)	20	30	40	50	60	70	
Solution Dipping Time (min)(2% aCl₂)	1	3	5	7	9	11	4
Vacuum Pressure(kPa)	20	30	40	50	60	70	

Analysis of variance (ANOVA) was used to detect treatment effect. Mean separation were performed by using least significance difference(LSD) at the

$p \leq 0.05$. The statistical analysis was performed using Statistical Analysis System (SAS) JMP 7 to obtain the coefficients of the polynomial, the error probabilities $p(F)$, and the explained variability percentage (R^2), which allows the evaluation of the global fitting of the model to the experimental values of independent variables.

RESULT and DISCUSSION

Experimental Results

X values which Salvatori (1997) and Guerrero (1996) reported for apple seems between 0.135 and

0.273. The most important reason of the difference and X values multiplied by coefficient of 1000 in the experiments is that not having been used a cubic part of an apple, having been used a whole apple between 160 and 180 grams. So, study material is completely different. In Table 2 the pressure applied to the "Golden Delicious", "Starking Delicious" and "Granny Smith" types of apples' X values according to their vacuum time and solution immersion time Least Significance Difference(LSD) was applied

Table 2. Vakum basınçları(20, 30, 40, 50, 60 ve 70 kPa), vakum zamanları(2 ve 4 dakika) ve %2'lik CaCl₂'lü çözeltiye daldırma Zamanlarına göre hesaplanan X değerlerine göre çıkan sonuçların değerlendirilmesi.

		Response, X (m ³ liquid/m ³ fruit)*1000																	
Time min	kPa	Golden Delicious Dipping Time(min)						Starking Delicious Dipping Time(min)						Granny Smith Dipping Time(min)					
		1	3	5	7	9	11	1	3	5	7	9	11	1	3	5	7	9	11
2	20	0.12 ^c	0.19 ^d	0.25 ^e	0.44 ^d	0.5 ^c	0.5 ^d	0.73 ^d	0.83 ^c	1.21 ^c	1.32 ^d	1.42 ^d	1.45 ^c	0.47 ^b	0.49 ^c	0.57 ^d	0.69 ^c	0.71 ^d	0.58 ^d
	30	0.58 ^{bc}	0.7 ^c	0.8 ^d	1.07 ^c	1.57 ^b	1.38 ^{cd}	1.29 ^c	1.22 ^d	1.39 ^c	1.64 ^d	1.71 ^d	1.77 ^e	0.44 ^b	0.56 ^c	0.51 ^d	0.56 ^c	0.8 ^d	1.52 ^c
	40	1.19 ^b	1.17 ^b	1.31 ^c	2.04 ^{ab}	1.98 ^b	2.19 ^{bc}	1.36 ^c	3.27 ^b	3.63 ^b	3.63 ^c	3.19 ^c	3.15 ^d	0.50 ^b	0.37 ^c	0.57 ^d	0.63 ^c	0.67 ^d	0.66 ^d
	50	0.87 ^b	1.38 ^b	1.30 ^c	1.77 ^b	2.03 ^b	2.15 ^{bc}	2.33 ^{ab}	2.92 ^b	3.47 ^b	3.53 ^c	4.49 ^b	4.09 ^c	0.79 ^b	1.16 ^b	1.19 ^c	1.46 ^b	1.46 ^c	1.24 ^c
	60	1.29 ^b	1.47 ^b	1.95 ^b	2.23 ^{ab}	2.67 ^a	2.69 ^{ab}	2.76 ^a	3.38 ^{ab}	4.54 ^a	4.82 ^a	4.97 ^a	5.1 ^b	1.56 ^a	1.86 ^a	2.62 ^b	3.25 ^a	3.54 ^b	4.18 ^b
	70	2.04 ^a	2.11 ^a	2.38 ^a	2.56 ^a	2.88 ^a	3.62 ^a	2.16 ^a	3.62 ^a	3.21 ^b	4.24 ^b	4.32 ^b	5.9 ^a	1.72 ^a	2.05 ^a	4.39 ^a	3.64 ^a	4.48 ^a	4.69 ^a
4	20	0.19 ^d	0.31 ^e	0.65 ^c	0.72 ^e	1.37 ^d	1.46 ^c	1.28 ^c	1.64 ^e	1.34 ^f	1.79 ^d	1.7 ^d	1.98 ^c	0.60 ^c	0.61 ^d	0.64 ^d	0.75 ^e	0.77 ^d	0.67 ^d
	30	1.01 ^c	0.91 ^d	1.04 ^c	1.34 ^d	2.86 ^b	1.57 ^c	1.20 ^c	1.63 ^e	2.05 ^e	2.12 ^d	2.33 ^d	2.49 ^c	1.44 ^b	1.51 ^c	2.58 ^b	2.44 ^b	2.57 ^b	2.30 ^c
	40	2.41 ^a	2.52 ^a	2.14 ^b	2.62 ^{bc}	3.11 ^b	3.1 ^{ab}	1.99 ^b	2.13 ^d	2.55 ^d	4.17 ^c	4.82 ^c	4.65 ^b	0.39 ^c	0.74 ^d	1.43 ^c	1.94 ^c	2.29 ^b	2.01 ^c
	50	1.77 ^b	2.01 ^{bc}	2.34 ^{ab}	2.71 ^b	3.06 ^b	3.32 ^a	1.98 ^b	2.72 ^c	6.5 ^a	6.66 ^b	6.27 ^b	5.15 ^b	0.56 ^c	0.84 ^d	1.51 ^c	1.40 ^d	1.43 ^c	1.17 ^d
	60	1.36 ^c	1.68 ^c	1.92 ^b	2.23 ^c	2.47 ^c	2.57 ^b	2.24 ^b	3.20 ^b	4.46 ^c	4.66 ^c	5.17 ^c	5.21 ^b	1.81 ^{ab}	2.95 ^a	3.15 ^a	3.42 ^a	3.82 ^a	4.31 ^a
	70	2.24 ^a	2.49 ^{ab}	2.92 ^a	3.74 ^a	3.65 ^a	3.35 ^a	3.5 ^a	3.62 ^a	6.11 ^b	7.39 ^a	10.2 ^a	11.2 ^a	2.06 ^a	2.36 ^b	2.52 ^b	3.49 ^a	3.70 ^a	3.71 ^b

Within the table, means in the same column with the same letters were not significantly different ($P < 0.05$).

In Table 2 calculated experimental x values are in 'Golden Delicious' type 0.12 ile 3.62, 'Starking Delicious' apple type 0.73 ile 5.9, 'Granny Smith' apple type 0.47 ile 4.69 and they have 2 minutes vacuum time. Calculated experimental x values in 4 minutes vacuum time are 'Golden Delicious' apple type 0.19 ile 3.74, 'Starking Delicious' apple type 1.20 ile 10.22, 'Granny Smith' apple type 0.60 ile 4.31 . In all apple varieties, the increase of x values are less under low pressure (20, 30, 40 ve 50 kPa) but under high vacuum pressure (60 ve 70 kPa) there was a clear difference. The LSD test also showed that difference by 'lettering difference'.

Statistical Analysis

The analysis of variance showed that the are well adjusted to the experimental data for all the studied fruits Table 3, Table 4 and Table 5. These results indicated that the models were sufficiently accurate for predicting the volume of each fruit impregnated with an X, for any combination of independent variable values within the ranges studied. Statistical analysis in Table 3, Table 4 and Table 5 presents the significance of the regression coefficients of at $p \leq 0.10$.

Table 3. Analysis of variance for volume the "Golden Delicious" impregnated by a X (The impregnation liquid fraction)

Vacuum Time (min)	Dipping Time (min)	Vacuum Pressure (kPa)	R ²	F	p(F)
2	1	20	0.93	20.31	0.01
	3	30	0.95	32.1	0.0095
	5	40	0.96	42.4	0.062
	7	50	0.94	27.4	0.01
	9	60	0.96	44	0.006
	11	70	0.98	136	0.001
4	1	20	0.96	36.3	0.008
	3	30	0.85	8.8	0.05
	5	40	0.80	6.23	0.08
	7	50	0.99	159	0.0002
	9	60	0.99	454	0.0002
	11	70	0.82	7.09	0.07

As can be seen in Table 3, according to R² test results in 'Golden Delicious' type of apple 50 and 60 kPa vacuum pressure is the most appropriate. Conway et al. (1983) study of 68.95 kPa pressure is different but it's understood that they can study in 60

kPa pressure. 4 minutes' vacuum time is not different from 2 minutes' vacuum time.

Table 4. Analysis of variance for volume the "Starking Delicious" impregnated by a X (The impregnation liquid fraction)

Vacuum Time (min)	Dipping Time (min)	Vacuum Pressure (kPa)	R ²	F	p(F)
2	1	20	0.95	33.19	0.009
	3	30	0.95	32	0.009
	5	40	0.96	42	0.006
	7	50	0.89	13.1	0.03
	9	60	0.96	40.1	0.006
	11	70	0.94	25.7	0.01
4	1	20	0.69	3.48	0.16
	3	30	0.96	39.07	0.007
	5	40	0.89	12.7	0.03
	7	50	0.98	96.6	0.002
	9	60	0.94	27.7	0.01
	11	70	0.96	41.2	0.006

As can be seen in Table 4, according to R² test results in 'Starking Delicious' type of apple 50 kPa vakum basıncı vacuum pressure is the most appropriate. And it's understood that 4 minutes' vacuum time is not different from 2 minutes' vacuum time.

Table 5. Analysis of variance for volume the "Granny Smith" impregnated by a X (The impregnation liquid fraction)

Vacuum Time (min)	Dipping Time (min)	Vacuum Pressur (kPa)	R ²	F	p(F)
2	1	20	0.86	9.9	0.04
	3	30	0.84	8.3	0.05
	5	40	0.91	16.5	0.02
	7	50	0.83	7.81	0.06
	9	60	0.96	114.3	0.006
	11	70	0.77	5.13	0.10
4	1	20	0.81	6.8	0.07
	3	30	0.83	7.77	0.06
	5	40	0.87	10.08	0.04
	7	50	0.70	3.62	0.15
	9	60	0.98	141.3	0.001
	11	70	0.91	15.30	0.02

As can be seen in Table 5, according to R² test results in 'Granny Smith' type of apple 60 kPa vacuum pressure is the most appropriate. And it's understood that 4 minutes' vacuum time is not different from 2 minutes' vacuum time.

Graphical Analysis

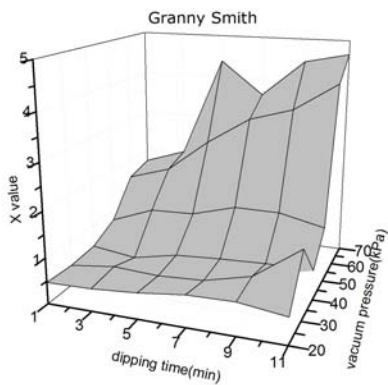
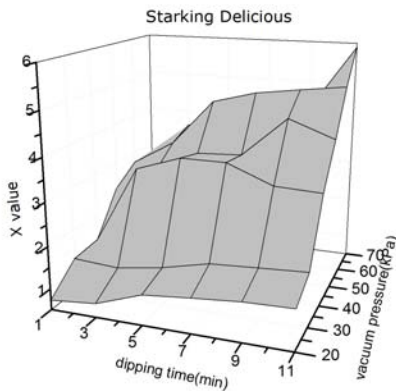
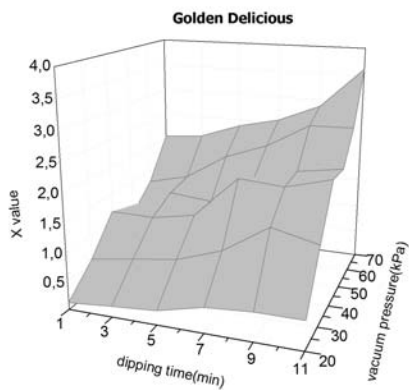


Figure 3. 3 D graphs of X values with respect to dipping time and vacuum pressure for 2 minutes vacuum time

In Figure 3 graphs of the results of apple varieties 'Golden Delicious', 'Starking Delicious' and 'Granny Smith' were given the X values as 3-dimensional by putting in place in the formula which Salvatori(1997) reported according to their 2 minutes Vacuum Time, Vacuum Pressure and Solution Dipping Time.

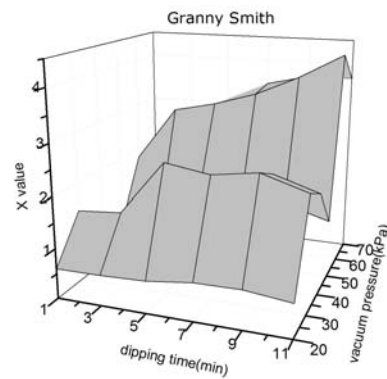
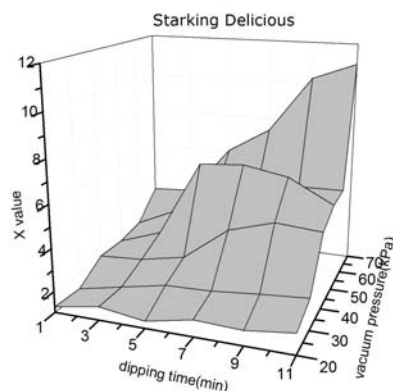
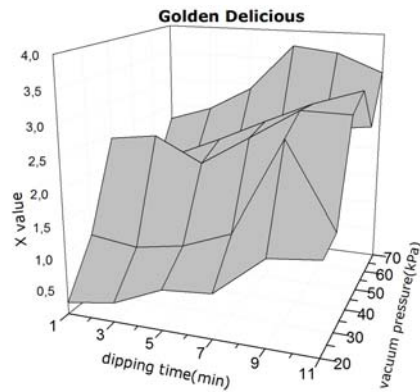


Figure 4. 3 D graphs of X values with respect to dipping time and vacuum pressure for 4 minutes vacuum time

In Figure 4 the X values " were given as 3-dimensional by putting in place in the formula which Salvatori(1997) reported according to their 4 minutes Vacuum Time, Vacuum Pressure and Solution Dipping Time.

According to the graphes obtained from 2 and 4 minutes vacuum time; For Golden type of apple after 40 kPa pressure absorption increased. Especially in 40 kPa(4 minute) vacuum pressure very successful results were obtained. The highest X Values were

obtained in 70 kPa vacuum pressure, the lowest X values were obtained in 20 kPa vacuum pressure. In addition to the study of Conway et al. (1983) at 68.95 kPa pressure it was understood that 50 and 70 kPa pressure is also suitable.

As it can be understood from the graph, in Starking type of apple absorption was obtained even in low pressure. The highest absorption values were obtained in 2 minutes vacuum time at 60 kPa vacuum pressure, in 4 minutes vacuum time at 70 kPa vacuum pressure. As it can be understood from the graph, in Granny type of apple, absorption was calculated in very low values both 2 and 4 minutes vacuum time, low vacuum pressure ((20, 30, 40 ve 50 kPa). But absorption was seen too high at 60 and 70

kPa pressure and showed that study is suitable for this fruit at these pressure ranges.

CONCLUSIONS

Vacuum pressure level and its application time had an important effect on the impregnation in the studied fruits. Especially in infiltration experiments performed under vacuum pressure fruits were cutted cubic or cylindrically and quite good results were obtained from these experiments. In this paper, in all fruits having weight between 160 g and 180 g good results were obtained and these results were supported by statistical and graphical parameters.

It should be kept in mind that at long vacuum impregnation times, deformation of the fruit tissue may occur at high vacuum pressure.

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