

# Evaluating The Effect of Time and Temperature on The Ph, Titratable Acidity & Brix of Pasteurized Low-To-No Nectar Orange Drinks Packed in Paper Based Aseptic Packages

**Azeen Kamal Al-Deen Mohammad**

Kurdistan Institution for Strategic Studies & Scientific Research  
Sulaimaniya, Iraq  
azeen.mohammad@kissr.edu.krd

**Nashmil Ali Abdul**

Kurdistan Institution for Strategic Studies & Scientific Research  
Sulaimaniya, Iraq  
nashmil.abdul@kissr.edu.krd

**Mohabad Hussien Jalal**

Kurdistan Institution for Strategic Studies & Scientific Research  
Sulaimaniya, Iraq

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## ABSTRACT

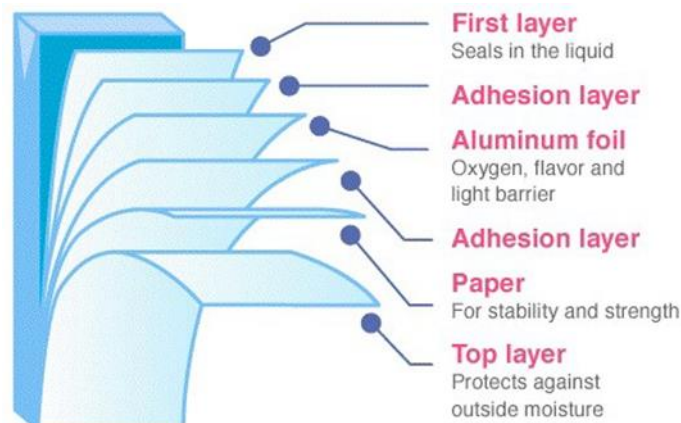
*Orang drink is the most popular flavored drink consumed in Iraq, and it can be found in markets of all sizes and in all regions, exposing it to a wide range of storage temperature and storage time. In this work, the impact of temperature and storage duration was studied on three different local brands of Orange drink with Low-to-No nectar content; all were packed in Laminated Aseptic Cartons (LAC). Samples were stored in a fixed and controlled temperature environment ranging between 10°C and 50°C for a period of 5 weeks and the samples were tested for the values of Brix, Titratable Acidity (TA) and pH at the beginning and at the end of each week. The findings revealed that there was no statistically significant difference found ( $p > 0.05$ ), which showed that there is low-to-no evidence that the selected range of storage temperature and time in this study significantly affected the values of Brix, TA and pH.*

## 1. INTRODUCTION

Extending the shelf life of any consumed product is an ever-ending quest of the fast moving consumable goods (FMCG) industry. Shelf life is defined as the time, under defined storage conditions, during which food remains safe, retains desired sensory, chemical, physical and biological characteristics as well as complies with any label declaration. [1] In addition to the storage conditions, there are many more factors influencing the shelf life of FMCG products. Some of these factors are related to the products themselves (Intrinsic factors), and others are related to the processes of manufacturing, moving, packing and storing the products; which are factors the final product encounters as it moves from manufacturing to retailing and consumption (Extrinsic factors). Intrinsic factors include Water activity, pH value, Titratable Acidity, Available oxygen. Extrinsic factors include Time–temperature profile during processing, Temperature control during storage and distribution, Exposure to light (UV and IR) during processing, storage and distribution, Consumer handling. [2]

Similar researches have been conducted on the effect of storage conditions, these researches focused in general on High nectar juices or concentrated juice. They also focused on Can, Glass and PET bottles that could lead to different results compared to laminated aseptic cartons (LAC). In addition, the type of juice and environment (geographical location) might have an impact on the results. Little research was found on Orange Drink packed in LAC and produced or stored in an environment and conditions similar to that found in Iraq.

One research was done on the effect of packaging material and storage conditions on the value of vitamin C and the pH Value of a pasteurized Cashew Apple Juice, the juice was packed in PET bottles and high-density polyethylene sachet, and it was stored at 30°C and 4°C for 4 months. There were significant differences ( $p > 0.05$ ) in the value of vitamin C (48mg/100ml – 159mg/100ml) and pH (5.0– 6.2) of the juice stored at  $30 \pm 1^\circ\text{C}$  as compared with those samples stored at 4°C. [3] Another research was done to evaluate several properties of pasteurized Roselle-fruit juice blends packed in plastic bottles, the samples were stored at 28°C and 4°C for 6 months. Over that period the pH ranged from 2.34 to 4.37 (at 28°C) and 2.34–3.38 (at 4°C), Titratable Acidity ranged from 3.12 to 1.28 (at 28°C) and 3.12–1.24 (at 4°C), the Reducing Sugar (RS) value For Roselle-fruit ranged from 2.95 to 9.92 mg/100 g (at 28°C) and 2.95–9.32 mg/100 g (at 4°C). [4]



**Figure 1:** Different layers of LAC. [10]

The type of packaging used was also shown to affect the Intrinsic chemical properties. Selecting one packaging materials across all samples is important to eliminate the packages as a variable. In general, there are four types of packaging materials used for packing liquid FMCGs, paperboard cartons, Cans, Plastic, and glass. In addition to complying with current rules and regulations, it is critical to carefully select the correct packaging material to provide the required protections for the packed products. [5] Plastic sachets and plastic bottles are not

suitable for products that need to have long shelf life. As the material has lower resistance to sunlight penetration and heat, unwanted changes in the packed product properties happen at a faster rate. Laminated Aseptic Carton (LAC) packages provide better protection against sunlight and heat penetration in comparison with the other types of packages. It is not unusual for products packed in sachets to have a shelf life of less than one month while products packed in LAC can have a shelf life that exceeds 9 months. [6] [7]

For this study LAC packaging was selected, as it is a common type of packaging used for drinks in Iraq. The packing process of Drinks in LAC includes pasteurization of the Drink followed up by packaging the drink using Aseptic Packaging Methods. To produce the final product the formed drink first passes through a pasteurization station where it is commonly heated up to 95–98°C for a period of 10–30s, followed by a cooling cycle. The pasteurized product is then packed into LAC packages and sealed using Aseptic filling and packing machines in a closed Aseptic environment. The LAC normally consists of seven layers as shown in Fig. 1. Each layer has specific properties and is used to protect against a certain Extrinsic factor including Light, Oxygen, Odor, and moisture. A sealed LAC also keeps the product sealed away from surrounding environment until the package is opened (i.e. seal is broken) [8] [9] [10]



**Figure 2:**Laminated Aseptic Carton (LAC) Package. [11]

In Iraq, the most consumed juices (as called commercially) are Low-to-No Nectar drinks. We will refer for both Low Nectar Juice and No Nectar Drink as "Drink" in this study. Moreover, within the many flavors, Orange is the Iraqi consumers' favorite. The main sales channels to end consumers consists of small to medium sized markets and grocery stores. These markets normally store the drinks in storage conditions that could prevent the packages from direct exposure to sunlight, rain, winds but not necessary the local temperatures. This is especially true for aseptic packaged drinks as the most companies with products packed aseptically in carton packages, Fig. 2, claim that the technology does not demand that drinks are stored in a controlled temperature but can be stored in ambient temperature. [7]

Preventing changes in the properties of packed juices and drinks is one of the major concerns of the mass production beverage industries; especially when the shelf life is expected by retailers and consumers to become longer, and the storage conditions are becoming more diverse as the same product can be shipped to different countries having different environmental conditions, or can go through different seasons of the year during its shelf life. In this study, The effect of two Extrinsic factors on a set of intrinsic chemical properties of a selected FMCG product was examined and tested, these factors are the storage temperature and storage time. The product selected is Low-to-No nectar Orange drink packed in LAC packages and the set of Intrinsic properties tested are the pH value, the Brix value, and the Titratable Acidity value of the Drinks. These three properties are submitted for testing as per Iraqi regulations and the results must be within a certain threshold for allowing the product to be sold for consumers. Understanding the change in these values during the shelf life of the

products will indicate if they may deviate from initial conditions enough to fall beyond the accepted thresholds. In addition, studies suggest that these values relate directly to the product test and Consumer acceptability. [12] [13]

## 2. METHODS AND MATERIALS

### 2.1 Materials

The samples consisted of Orange drink commercially packed in LAC packages. Samples were bought from local wholesalers. The wholesalers stored the drinks in warehouses without controlled temperature (ambient weather), and the production date of any sample did not exceed 45 days (shelf life of products is 12 months). Three local brands were selected, each belonging to a different local company/factory in order to evaluate if there would be significant variation in the results between different brands when all are subjected to the same test procedures. Having three companies allows to progress with further study in cases where significant variations were observed which might not be possible to understand without examining factors related to the manufacturing stages as well. One brand is known by the wholesalers for having high quality standards, one brand is known for having moderate quality standards, and one brand is known for having low quality standards. The standards differentiation was labeled based on the feedback of the wholesalers and the distributors. Attention was paid to selecting samples with no signs of damages to the packages to minimize any variables which could result from the damages. The ingredients of each brand, as labeled on the packages by the companies which produced the filled packages, are:

- Brand 1 Ingredients: Water, Sugar, Orange Juice from Improving Materials, Citric Acid, Sodium Benzoate, Fruit Pectin E-440, Vitamin C Beta-carotene (Pro-vitamin A) Natural Identical Orange Flavour.
- Brand 2 Ingredients: Water, sugar, citric acid, Stabilizer: GMC, Natural Orange Flavour, permitted colors: E110 - E102. Potassium sorbate sodium benzoate Xanthan gum.
- Brand 3 Ingredients: Filtered water, sugar, orange concentrate, citric acid, thickeners, orange flavour.

### 2.1 Methods

126 samples were tested in total over a period of 5 weeks, 42 samples from each brand. A 5 week incubation period is an average period used in other studies conducted for similar objectives. All samples kept sealed and unopened until the testing time. The pH, Brix, and Titratable Acidity (TA) tests were done for the orange drinks from each individual sample. For each test two samples from each brand were tested individually in order to have repeated results for the same brand under the same conditions to highlight any extreme variations in the results between both samples of each brand which might be a result of a defected sample or testing errors. At the start of the study, two samples from each brand were tested and the average results were recorded as the Initial Conditions before subjecting the samples to the selected temperatures and incubation times. For subjecting the samples to the selected temperatures and conditions, ten samples from each brand were grouped together and placed in a controllable fridge having a set temperature of 10°C. The same was repeated 4 more times and each group was placed in a controlled temperature of 20, 30, 40, and 50°C respectively using controllable incubators. The range of 10-50°C was selected to mimic the known weather temperatures in Iraq throughout the year. The samples were incubated for a total period of 5 weeks, at the end of each week, 2 samples from each brand were taken out, tested for the values of pH, Brix and TA, then the average result was recorded. There were extra samples to be used randomly either to confirm tests or to replace damaged samples. The diagram below, Fig. 3, illustrates the samples distribution over the incubation period and the selected temperatures. pH, TA, and Brix testing methods were as follows:

- pH value was measured using a calibrated digital pH Meter
- Titratable Acidity, expressed as % citric acid, was calculated using the simple titration method [14]:

$$\frac{\text{Titratable Acidity}(\%) \cdot T_v \cdot N_n \cdot V \cdot W_e \cdot 100}{V_t \cdot W_s \cdot 100} \quad (1)$$

Tv = Titration value, Nn = Normality of NaOH, V = Vol. madeup, We = Equivalent weight of citric acid, Vt = Volume taken for titration, Ws = weight of sample.

- Brix was measured using a Brix Refractometer

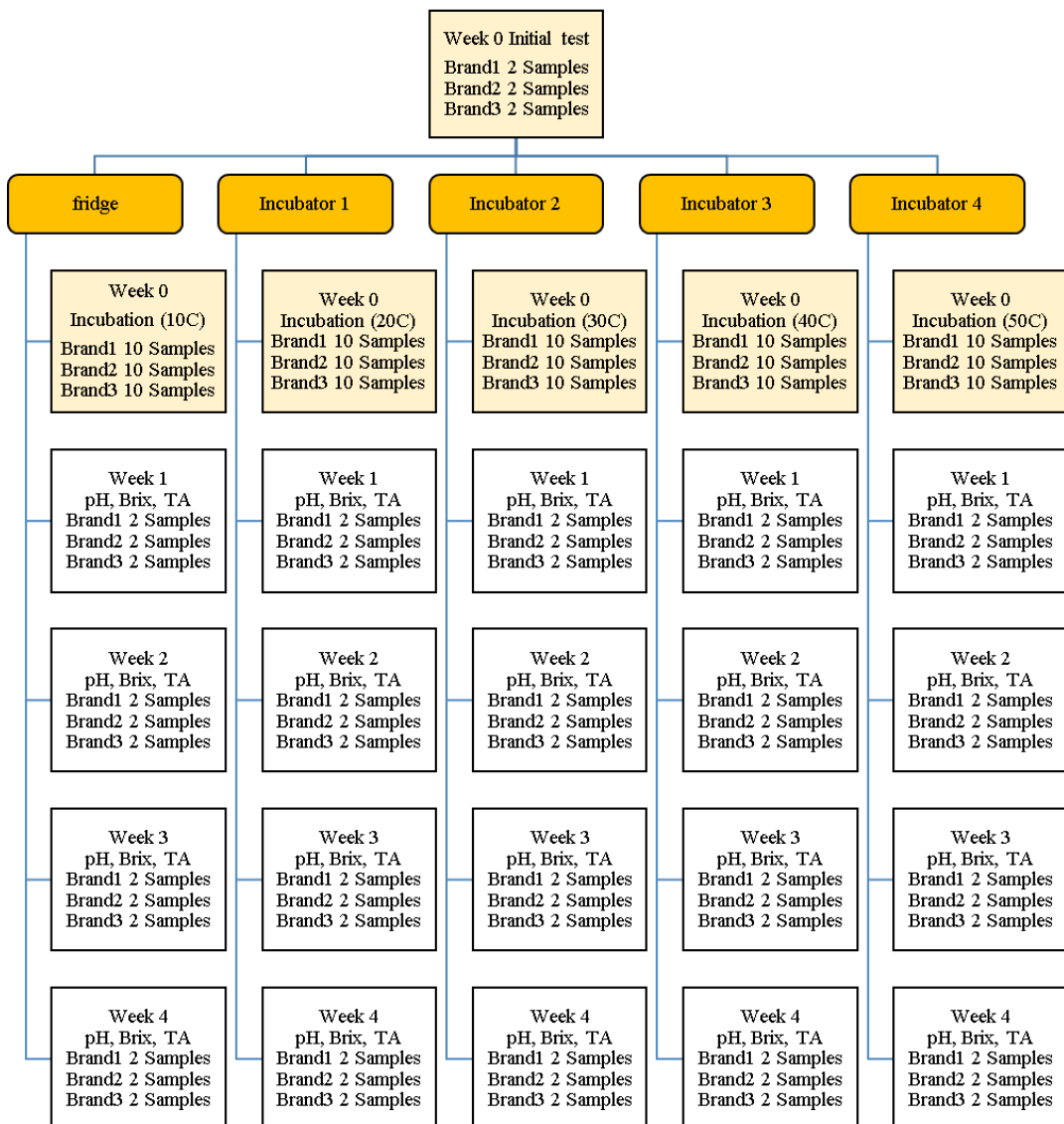


Figure 3. Complete characterization process

### 3. RESULTS AND DISCUSSION

To highlight the overall changes in results and to minimize human and testing method errors, the results were calculated for each test individually then the averages were calculated once over the range of selected incubation temperatures and the range of selected incubation times. Table 1 shows the records of the tests. In Table 1, the columns list the incubation temperatures and tests, and the rows list the incubation times and brands. Any selected column and row will intersect at the test result for a desired incubation temperature, brand and incubation time. An example is the value of pH test at 20°C for brand 2 after 3 weeks of incubation time which is 4, identified by crossing Column No. 5 and Row No. 9.

**Table 1:** Average test results for all the samples throughout the incubation times and temperatures

Column ref. No.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	Row Ref No	
Temperature °C	10			20			30			40			50				
Test	TA	pH	Brix	TA	pH	Brix	TA	pH	Brix	TA	pH	Brix	TA	pH	Brix		
<b>Brand 1</b>	Week 0	0.26	3.8	4.5	0.26	3.8	4.5	0.26	3.8	4.5	0.26	3.8	4.5	0.26	3.8	4.5	1
	Week 1	0.26	3.89	4.5	0.26	3.79	4.5	0.19	3.71	4.5	0.33	3.6	4.5	0.28	3.9	4.5	2
	Week 2	0.25	3.6	4.5	0.25	3.6	4.62	0.22	3.48	4.6	0.3	3.6	4.5	0.29	3.9	4.5	3
	Week 3	0.25	4.23	4.6	0.25	4.23	4.55	0.25	4.24	4.6	0.26	3.4	4.5	0.26	3.5	4.5	4
	Week 4	0.25	3.8	4.5	0.26	3.81	4.5	0.24	3.81	4.5	0.25	3.6	4.5	0.26	3.5	4.5	5
<b>Brand 2</b>	Week 0	0.26	4.1	10	0.26	4.1	10	0.26	4.1	10	0.26	4.1	10	0.26	4.1	10	6
	Week 1	0.26	4.01	10	0.26	4.02	10	0.26	3.93	10	0.3	3.8	10	0.24	4.05	10	7
	Week 2	0.26	4.2	10	0.26	3.96	10	0.12	3.83	10	0.29	3.9	10	0.29	3.85	10	8
	Week 3	0.25	4.25	10	0.25	4	10	0.25	4.1	10	0.26	3.95	10	0.26	3.9	10	9
	Week 4	0.26	4.04	10	0.25	4.02	10	0.26	3.87	10	0.25	3.8	10	0.26	3.9	10	10
<b>Brand 3</b>	Week 0	0.26	3.6	9	0.26	3.6	9	0.26	3.6	9	0.26	3.6	9	0.26	3.6	9	11
	Week 1	0.26	3.54	9	0.26	3.5	8.9	0.26	3.47	8.9	0.3	3.6	8.6	0.29	3.5	9	12
	Week 2	0.24	3.83	8.8	0.26	3.63	9	0.19	3.52	9.5	0.3	3.8	9	0.27	3.7	8.5	13
	Week 3	0.25	3.5	9.5	0.25	3.7	9.5	0.25	3.98	9.5	0.25	3	8.5	0.25	3	8.5	14
	Week 4	0.28	3.58	9	0.3	3.59	9	0.3	3.58	9	0.25	3.7	8.5	0.25	3.6	8.6	15

#### 3.1 Titratable Acidity

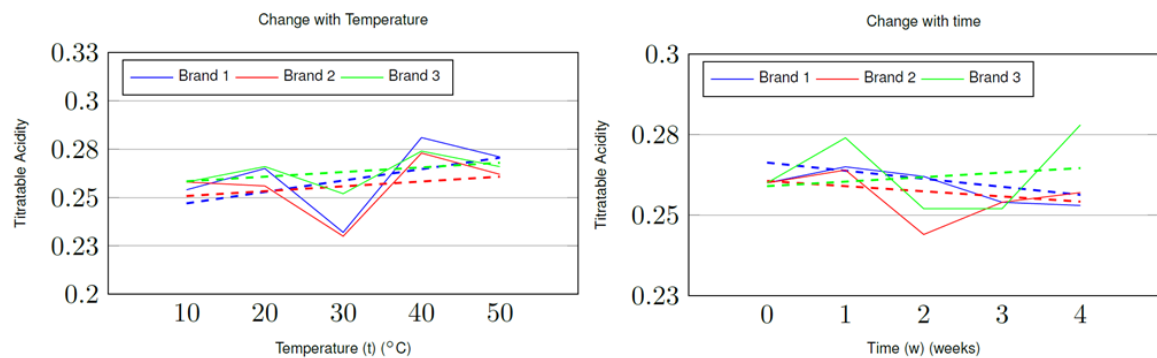
Table 2 lists the calculated average results for Titratable Acidity (TA) as a function of temperature and as a function of time. These results are used to plot two graphs as shown in Figure 4, one graph shows the average change of TA in relation to temperature and the other graph shows the average change of TA in relation to incubation time. The solid lines on the graphs are the plots of the real results. The dashed lines on the graphs are linear regressions used to better visualize how the TA value changes in relation to temperature and in relation to time. Each Brand in Figure 4 is labeled with a unique color.

The results show that TA was affected more in relation to temperature (i.e. as incubation temperature increased) and less as a function of time (i.e. as incubation time increased). The value of TA for all brand was increasing with the increase of both incubation time and incubation temperature. The standard deviation for the TA ranged from 8.7% to 11.9%. The linear regression (LR) equations, Table 3, are useful to mathematically define both the magnitude of the slope for the LR lines drawn in the Graph (Figure 4) and the average change

in test results per time unit or temperature unit. The percentage change for TA values ranged from a minimum of %0.54 throughout the temperature and incubation time ranges to a maximum of %1.01 with the exception of brand 1 change with time that had a slop of %2.45. To farther explore the effect of the incubation time and incubation temperature on the results the P-Value (P) was calculated from the average results. All the TA results were found to be not statistically significant ( $p > 0.05$ ) which suggests that there is no evidence of the results being significantly affected by the change in the incubation time and incubation temperature.

**Table 2:** Average Results for Titratable Acidity as a function of Temperature and Time

Temp °C	Brand 1	Brand 2	Brand 3	Time	Brand 1	Brand 2	Brand 3
10	0.254	0.258	0.258	Week0	0.26	0.26	0.26
20	0.256	0.256	0.266	Week1	0.26	0.26	0.27
30	0.232	0.23	0.252	Week2	0.26	0.24	0.25
40	0.281	0.273	0.274	Week3	0.25	0.25	0.25
50	0.271	0.262	0.266	Week4	0.25	0.25	0.27



**Figure 4:** The Average change in Titratable Acidity Value for each brand

**Table 3:** linear regression equations for Titratable Acidity

$A(w) = -0.0025w + 0.2663$	TA (A) Linear regression as function of time (w) (brand 1)	(2)
$A(w) = -0.0016w + 0.2606$	TA (A) Linear regression as function of time (w) (brand 2)	(3)
$A(w) = 0.0014w + 0.259$	TA (A) Linear regression as function of time (w) (brand 3)	(4)
$A(t) = 0.0059t + 0.2411$	TA (A) Linear regression as function of temperature (t) (brand 1)	(5)
$A(t) = 0.0025t + 0.2483$	TA (A) Linear regression as function of temperature (t) (brand 2)	(6)
$A(t) = 0.0024t + 0.256$	TA (A) Linear regression as function of temperature (t) (brand 3)	(7)

### 3.2 pH

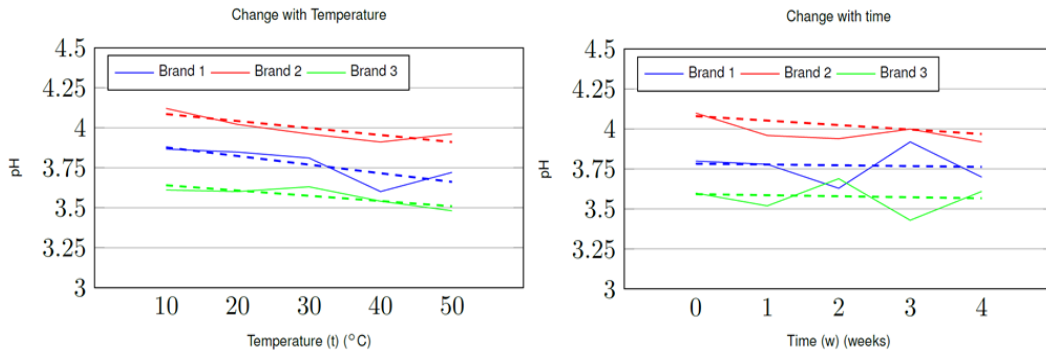
Table 4 lists the calculated average results for pH as a function of temperature and as a function of time. These results are used to plot two graphs as shown in Figure 5, one graph shows the average change of pH in relation to temperature and the other graph shows the average change of pH in relation to incubation time. The solid lines on the graphs are the plots of the real results. The dashed lines on the graphs are linear regressions used to better visualize how the pH value changes in relation to temperature and in relation to time. Each Brand in Figure 5 is labeled with a unique color.

The results show that pH value was affected more in relation to temperature (i.e. as incubation temperature increases) and less as a function of time (i.e. as incubation time increases). The value of pH was decreasing with the increase of both incubation time and incubation temperature. This is inverse compared to TA results, the reverse behavior of TA and pH is

expected, if one increases the other decreases. The magnitude of change in TA was higher than the magnitude of change in pH for both temperature and time and for all brands. The Standard deviation for the pH ranged from 3% to 5.8%. The linear regression (LR) equations, Table 5, are useful to mathematically define both the magnitude of the slope for the LR lines drawn in the Graph (Figure 5) and the average change in test results per time unit or temperature unit. The percentage change for pH values ranged from a minimum of %0.13 throughout the temperature and incubation time ranges to a maximum of %1.06 with the exception of brand 1 change with time that had a slope of %1.37. The results were found to be not statistically significant ( $p > 0.05$ ) which suggests that there is no evidence of the results being significantly affected by the change in the incubation time and incubation temperature.

**Table 4:** Average Results for pH as a function of Temperature and Time

Temp °C	Brand 1	Brand 2	Brand 3	Time	Brand 1	Brand 2	Brand 3
10	3.86	4.12	3.61	Week0	3.8	4.1	3.6
20	3.84	4.02	3.6	Week1	3.78	3.96	3.52
30	3.81	3.96	3.63	Week2	3.63	3.94	3.69
40	3.6	3.91	3.54	Week3	3.92	4.04	3.43
50	3.72	3.96	3.48	Week4	3.7	3.92	3.61



**Figure 5.** The Average change in pH Value for each brand

**Table 5:** linear regression equations for pH

$P(w) = -0.005w + 3.7836$	pH (P) Linear regression as function of time (w) (brand 1)	(8)
$P(w) = -0.0279w + 4.081$	pH (P) Linear regression as function of time (w) (brand 2)	(9)
$P(w) = -0.0064w + 3.593$	pH (P) Linear regression as function of time (w) (brand 3)	(10)
$P(t) = -0.0539t + 3.9303$	pH (P) Linear regression as function of temperature (t) (brand 1)	(11)
$P(t) = -0.0437t + 4.1284$	pH (P) Linear regression as function of temperature (t) (brand 2)	(12)
$P(t) = -0.0328t + 3.6722$	pH (P) Linear regression as function of temperature (t) (brand 3)	(13)

### 3.3 Brix

Table 6 lists the calculated average results for Brix as a function of temperature and as a function of time. These results are used to plot two graphs as shown in Figure 6, one graph shows the average change of Brix in relation to temperature and the other graph shows the average change of Brix in relation to incubation time. The solid lines on the graphs are the plots of the real results. The dashed lines on the graphs are linear regressions used to better visualize how the Brix value changes in relation to temperature and in relation to time. Each Brand in Figure 6 is labeled with a unique color.

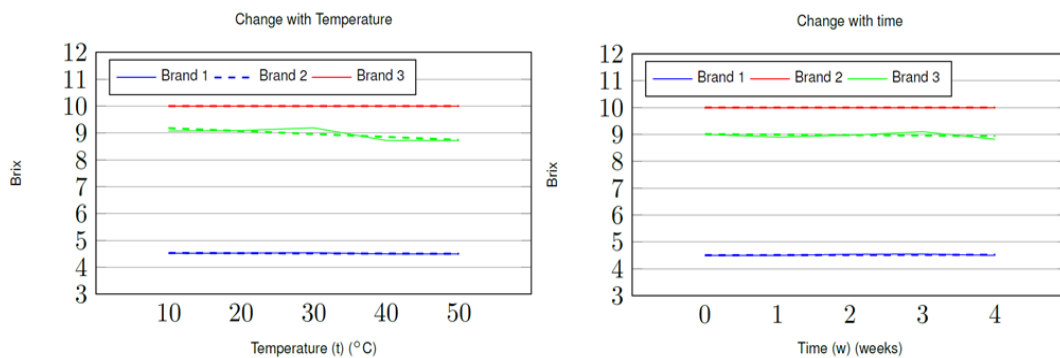
The Brix results showed the lowest change both in relation to temperature and in relation to time. Brix value was almost constant for all tested samples; and when it was not constant, the biggest standard deviation from the mean value in any test was no more than 3%. The linear



regression (LR) equations, Table 7, are useful to mathematically define both the magnitude of the slope for the LR lines drawn in the Graph (Figure 5) and the average change in test results per time unit or temperature unit. The percentage change for Brix values ranged from a minimum of %0 throughout the temperature and incubation time ranges to a maximum of %0.18 with the exception of brand 3 change with time that had a slop of %1.17. The results were found to be not statistically significant ( $p > 0.05$ ) which suggests that there is no evidence of the results being significantly affected by the change in the incubation time and incubation temperature.

**Table 6:** Average Results for Brix as a function of Temperature and Time

Temp C	Brand 1	Brand 2	Brand 3	Time	Brand 1	Brand 2	Brand 3
10	4.52	10	9.07	<b>Week 0</b>	4.5	10	9
20	4.53	10	9.09	<b>Week 1</b>	4.5	10	8.9
30	4.54	10	9.19	<b>Week 2</b>	4.54	10	8.9
40	4.5	10	8.72	<b>Week 3</b>	4.55	10	9.1
50	4.5	10	8.72	<b>Week 4</b>	4.5	10	8.8



**Figure 6.** The Average change in Brix Value for each brand

**Table 7:** linear regression equations for Brix

$B(w) = 0.005w + 4.504$	Brix (B) Linear regression as function of time (w) (brand 1)	(14)
$B(w) = 10$	Brix (B) Linear regression as function of time (w) (brand 2)	(15)
$B(w) = -0.0165w + 9.0095$	Brix (B) Linear regression as function of time (w) (brand 3)	(16)
$B(t) = -0.0075t + 4.5415$	Brix (B) Linear regression as function of temperature (t) (brand 1)	(17)
$B(t) = 10$	Brix (B) Linear regression as function of temperature (t) (brand 2)	(18)
$B(t) = -0.1085t + 9.2855$	Brix (B) Linear regression as function of temperature (t) (brand 3)	(19)

#### 4. CONCLUSION

The results from this study suggested there is little to No evidence that the selected extrinsic factors, incubation time and temperature, had any effect on the examined intrinsic chemical properties. It can be concluded from the results that the Low-to-no nectar orange drinks packed aseptically in Laminated Aseptic Cartons (LAC) experience no significant changes in the values of Brix, Titratable Acidity and pH when incubated at temperatures within the ranges 10°C-50°C for incubation time up to 5 weeks.

The following can be considered for future work:

- Longer incubation time: for this study a total incubation time of 5 weeks was decided which is on average 1 week longer than other studies conducted on the same subject. Changes in the intrinsic chemical properties, if any, would be more significant if the incubation time was extended and the average of the results would be less effected with human errors, if any.

- Use medium to high nectar products: the results from this study could be different depending on the product ingredients and contents, a higher nectar concentrate could result in results that change more significantly with the extrinsic factors.

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