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Determination of Freeze Drying Behaviors for Various Thicknesses of Apples by Using Taguchi Method

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Abstract

Freeze drying process the best drying technology regarding quality of the end product but it is an expensive method and the high costs of process limit its application to industrial scale. Also, the freeze-drying process is based on different parameters, such as drying time, pressure, sample thicknesses, chamber temperature, sample temperatures and relative humidity. Hence, the determination of drying behaviors, such as moisture content, moisture ratio and drying rate of the freeze-drying process are too complex. In this article, to help this freeze drying of complex process, the use of Taguchi method has been proposed. Taguchi method has been developed as per Taguchi's L9 orthogonal array with each experiment performed for the prediction of drying behaviors, such as moisture content, moisture ratio and drying rate of apples in the freeze drying process. Signal-to-noise ratio analysis and analysis of variance have been carried out in order to determine the effects of process parameters and optimal factor settings. Finally, confirmation tests verified that Taguchi method has been achieved for moisture content, moisture ratio and drying rate.

Keywords: Freeze drying, Taguchi method, ANOVA

Farklı Kalınlıklardaki Elmaların Dondurarak Kurutma Davranışlarının Taguchi Metodu Kullanılarak Belirlenmesi

Öz

Dondurarak kurutma sonuçta elde edilen ürünün kalitesine göre en iyi kurutma teknolojisidir. Fakat, bu yöntem pahalıdır ve işlemin yüksek maliyeti nedeniyle endüstriyel ölçekte uygulanması sınırlıdır. Ayrıca, dondurarak kurutma işlemi, kurutma süresi, basınç, numune kalınlıkları, hazne sıcaklığı, numune sıcaklıkları ve bağıl nem miktarı gibi farklı parametrelere bağlıdır. Bu yüzden dondurarak kurutma işlemi için nem içeriği, nem oranı ve kurutma oranı gibi kurutma davranışlarının belirlenmesi oldukça karmaşıktır. Bu makalede dondurarak kurutmanın karmaşık yapısı için Taguchi metodu önerilmiştir. Taguchi metodu, Taguchi'nin her L9 dikey sırası ile gerçekleştirilen her deneyin, dondurarak kurutma işleminde elmaların nem içeriği, nem oranı ve kurutma davranışları tahmini için geliştirilmiştir. İşlem parametrelerinin etkisi ve optimal unsur ayarlarını belirlemek için sinyal görüntü oranı ve varyans analizi gerçekleştirilmiştir. Son olarak, nem içeriği, nem oranı ve kurutma oranı için Taguchi metodunun onaylama testleri gerçekleştirilerek başarıya ulaştığı doğrulanmıştır.

Anahtar Kelimeler: Dondurarak kurutma, Taguchi metodu, ANOVA

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1. Introduction

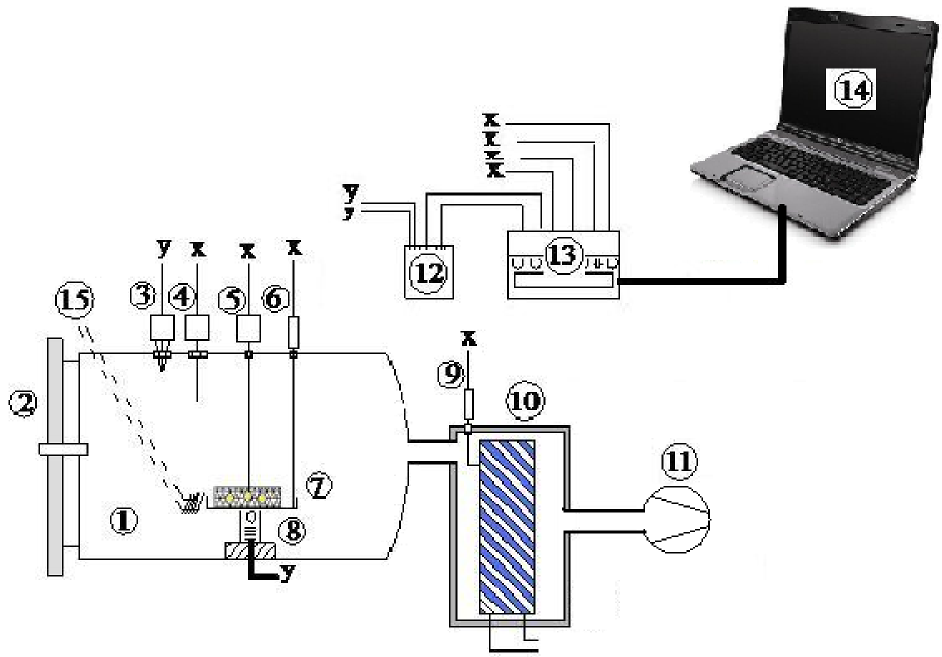
Freeze drying, also known as lyopbilization is a separation process, common method to produce stable food products with the highest quality and long shelf life which are used widely in biotechnology, fine chemicals, food, and pharmaceutical industries [1]. Freeze-drying process has some important advantages which are compared with conventional processes. These are protection against chemical degradation, low water activity, and the easier rehydration the protection of highly sensitive parts. Over and above freeze-drying owing to high equipment costs and high costs of relatively long drying times compared with other drying techniques [2].

In this study, we have used the apples since apples are significant raw materials for a lot of food products for freeze drying. Also, apple is contains large amounts of minerals and vitamins. Like most other moist fruits; it also contains large amounts of moisture, up to 85.5% of its weight, and 12-14% of carbohydrates [3]. Dried apple products are also quite popular in daily life, and can be used as secondary raw materials in food industry as well. Dried apples can have a long shelf life, as drying process removes water from the foodstuff to prevent microbial spoilage and chemical alteration [4]. Several papers have been published on freeze-drying process, analyzed the drying kinetics of the apples so far. Many experiments have been done by researchers on the behaviors of freeze-drying of apples [5 – 13]. Taguchi technique is a high-powered and generally applied method for modelling high quality systems [14, 15]. This paper investigates with the difference of past studies to forecast the moisture content (MC), moisture ratio (MR), and drying rate (DR) values by means of Taguchi method in order to obtain significant process parameters and optimum factor combinations.

2. Meterials and Method

Experimental Setup

In this study, the schematic diagram of a freeze drying experimental rig installation is given in Fig. 1. Freeze drying experimental rig setup composes of four parts including the drying chamber, a weighing system, a vacuum pump, a condensing unit, and the measurement apparatuses [16]. Freeze drying experimental rig consists of mainly the drying chamber, a condensing unit, a vacuum pump, a weighing system and the measuring equipments. The drying chamber is a cylindrical shape, with 370 mm inside diameter by 450 mm length, with a Plexiglas door 40 mm thick allowing visual inspection of the apples. Condensation unit of a vapor compressed refrigeration system prevents the vacuum pump from humidity. This unit is comprised of two condensers (one is for condensing unit and the other is for air-cooling), a 1 HP hermetic-type compressor (R-404A) and an expansion valve. The vacuum in the drying chamber was provided by a hybrid vacuum pump with $2x10^{-3}$ mbar pressure, 5.6 m^3/h (at 50/60 Hz) flow rate and 0.37 kW pumping power. Observation of the samples' weight loss was made with settling a load-cell in the center of the drying chamber. The capacity of the load-cell was 1000 g while its sensitivity was 0.01 g. Pressure transmitters were used to measure the change in pressure in the drying chamber (accuracy: ±0.075 %, range: $1x10^{-3}$ mbar ~ $2x10^{-3}$ mbar, feed: 10.5 - 42 V-DC and output: 4 – 20 mA). In order to get the results of the chamber pressures and sample weight loss, the transmitter and the load-cell were connected to a signal exchanger E-700/ELİMKO, then to a data-logger E-680/ELİMKO and to a personal computer by using EManager 5.1/ELİMKO. The required sublimation heating was provided by a 1 kW current controlled electrical heater. The heater is put at a 4 cm-distance from side of sample tray. The dimensions of the stainless steel sample tray (0.5 mm thickness and 9 mm bore diameter) were 200 x 200 mm was located on the load-cell [16].



1 Drying chamber	9 Thermocouple for condenser surface
2 Plexiglas door	10 Condensing unit
3 Transmitter	11 Vacuum pump
4 Thermocouple for drying chamber	12 Signal converter/E-700
5 Thermocouple, inserted into apples	13 Data-logger/E-680
6 Thermocouple, inserted onto tray	14 Notebook/EManager 5.1
7 Tray	15 Electrical heater
8 Load-cell	

Figure 1. Experimental setup of the freeze-drying of apples

Fresh apples were procured from a local supermarket. Elimination of unripe and rotten apples was made before apples have been peeled, their center’s was taken away and then split up 5 mm, 7mm and 10 mm, and sliced apples have been frozen at -30 °C in a deep-freezer (UĞUR deep-freezer, UDD 300-BK, Nazilli, Aydin, Turkey). Afterwards, the apples have been cut into 5 mm, 7 mm and 10 mm thick slices. Before the freeze-drying process, the taken as the sample sliced apples have been dried at 70 ± 3 °C. The weight measurements were recorded every half an hour during the drying time. Absolute dry weight was taken into account to be accomplished with the condition, which the weight changed less than 1% at the end of two sequential drying level. The initial moisture contents (dry basis) of the apples were obtained by using the Eq. (1) below [17]:

$$MC_{db} = \frac{M_i - M_d}{M_d} \tag{1}$$

Three times experimental analysis was made and the average of the moisture content (MC) values were obtained at each test. Then the results of drying obtained were used to determine the moisture ratio (MR) using Eq. (2) [18]:

$$MR = \frac{M - M_e}{M_o - M_e} \tag{2}$$

The drying rate (*DR*) calculation was made by using the Eq. (3) below:

$$DR = \frac{M_{t+dt} - M_t}{dt} \tag{3}$$

Where M_o, M, M_e, M_t and M_{t+dt} are initial, intermediate time (t), and final equilibrium moisture contents, moisture contents at a time t and $t+dt$ respectively [19].

Experimental Data

The 5 mm, 7 mm and 10 mm apple samples with an initial average moisture containing 8.47, 8.45 and 8.45g water/g dry matter were dried to 0.085, 0.099 and 0.10g water/g dry matter. In Fig. 2, 3, and 4, the variation of MC, MR and DR obtained from the experiments with sample thickness and drying time are presented. As it is shown in Fig 2, 5 mm, 7 mm and 10 mm apple samples were dried to 0.085, 0.099 and 0.10g water/g dry matter moisture contents in 660 min, 900 min, and 1110 min respectively. The time to reach the same moisture content, 7 mm and 10 mm samples required more time than 5 mm samples. It is clear that a linear relationship was obtained between freeze drying time and product thickness. Drying rate is defined as the quantity of removed water.

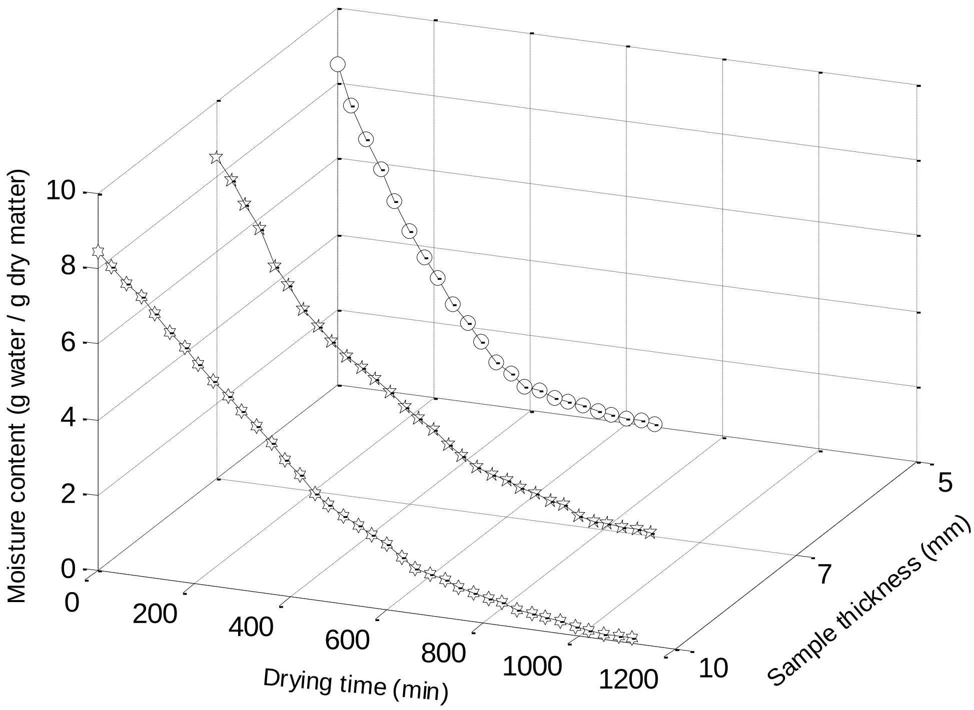


Figure 2. Changing of moisture amount according to sample thickness and drying time

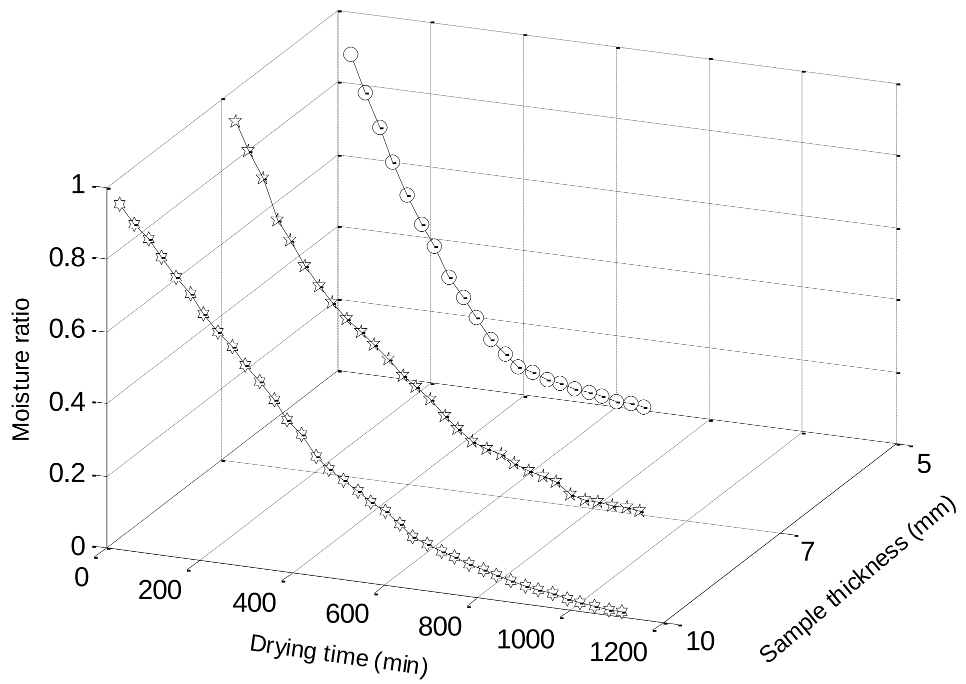


Figure 3. Changing of moisture ratio according to sample thickness and drying time.

The change in drying rate according to the sample thickness and drying time are given in Fig. 4 for 5 mm, 7 mm and 10 mm apple samples. While the drying rate is higher initially it declines steadily with drying time as seen in Fig.4. It is obtained that there is no constant rate drying period from the curves. In Fig. 5, moisture ratio obtained versus drying rate obtained is shown. Drying rate is decreasing with decreasing moisture ratio.

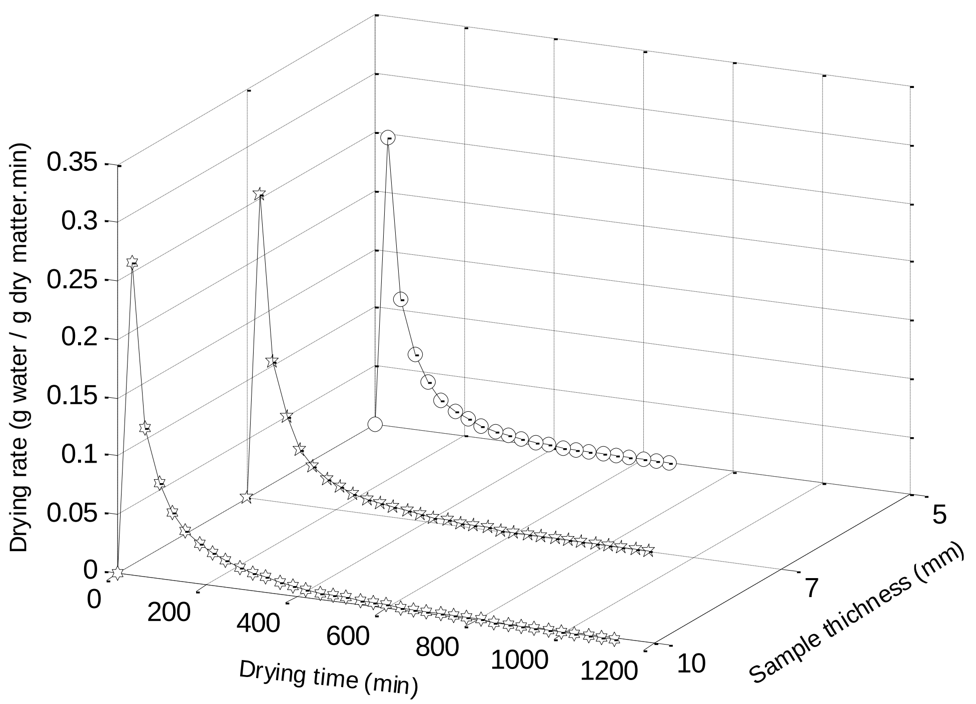


Figure 4. The variation of drying rate with sample thickness and drying time

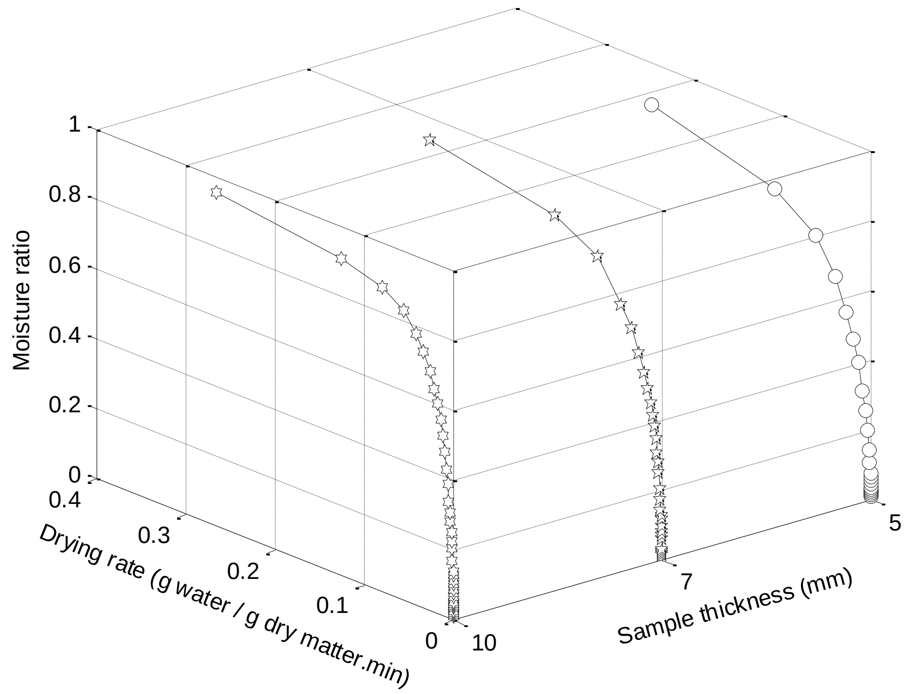


Figure 5. Changing of moisture ratio according to drying rate and sample thickness

Application of the Taguchi Method

Dr. Genichi Taguchi proposed methods for combining experimental design techniques with quality assessments. Investigations of performance can be made by taking into account considered factors using these methods. System design, parameter design and tolerance are the significant subjects in Taguchi methods [20]. Additionally, it gains a good bit popularity due to small numbers of trials, obtaining the effects of process parameters on quality characteristics and their optimum values.

Moisture content of drying on the different time moments expressed as (MR) values. According to experiments MR's possible factors affects; (1) Time, (2) Thickness as determined. The experiments are designed for the two factors with three levels are selected and used L9 matrix experiment. The experimental design consists of 9 trials (each row in the L9 orthogonal array) and the columns of the OA are assigned to factors and their interactions.

In this study, upon applying the steps above, the effects of process parameters, such as the moisture content (MC), moisture ratio (MR), and drying rate (DR) values were determined and optimum factor levels were obtained. The designated factors, their levels and experiment outputs are illustrated in Table 1. By using Taguchi technique based upon orthogonal arrays, the number of experiments which may result in an enhancement in time and cost can be decreased. In order to find out the all parameters space with a small number of experiments a specific design of orthogonal arrays were used.[15, 21].

Table 1. The specified factors, their levels and experiment responses

Exp. No	Factor A: Time (minute)	Factor B: Thickness (mm)	Response 1 MC	Response 2 MR	Response 3 DR
1	180	5	3.713384	0.438432	0.020159933
2	180	7	4.77178	0.563394	0.025962753
3	180	10	6.239583	0.736695	0.034090909
4	360	5	0.907828	0.107185	0.002286756
5	360	7	2.875947	0.339557	0.007715173
6	360	10	4.012311	0.473725	0.010858586
7	540	5	0.257576	0.030411	0.000320333
8	540	7	1.190341	0.140541	0.002021956
9	540	10	2.116477	0.249888	0.003728255

3. Results and Discussions

A common method used to determine important parameters on response and measuring their effects is ANOVA. F-test is the ratio between the variance of the process parameter and the error variance and it states whether the parameter has an important effect on the quality characteristic. This process is conducted by comparing the F test value of the parameter with the standard F Table value (F 0.05) at the 5% level of significance. If the F-test value is greater than F 0.05, the process parameter is taken into account. Depending on it, it can be seen that all the factors and their interactions on temperature gradient are significant. According to Taguchi experimental design matrix and ANOVA analysis results, the comparison with predicted and actual values based on statistical models which are the data obtained from the experiments. The comparison graphs are shown in Fig. 6, 7 and 8.

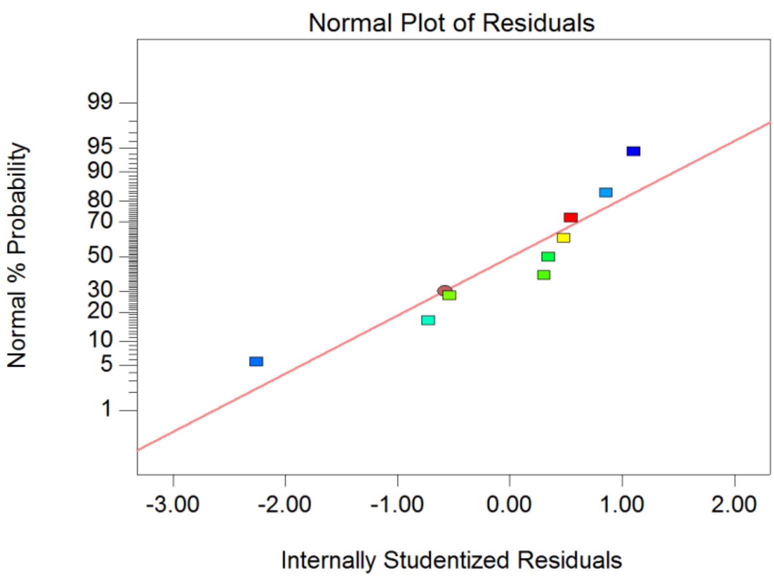


Fig.6. The comparison with predicted and actual based on statistical data for MC values

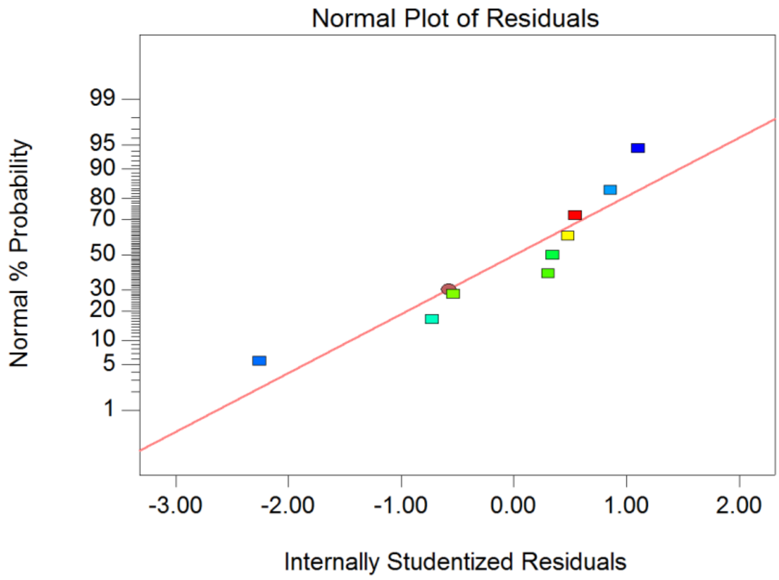


Fig.7. The comparison with predicted and actual based on statistical data for MR values

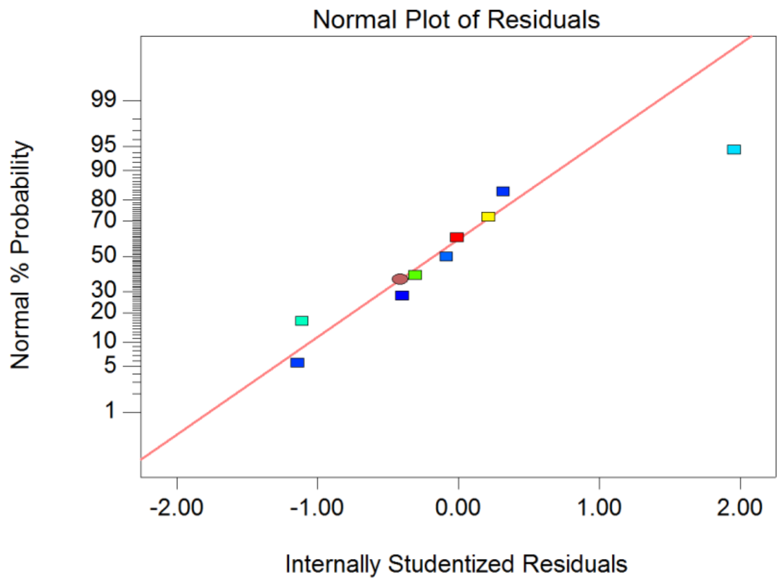


Fig. 8. The comparison with predicted and actual based on statistical data for DR values

The relationship with the thickness and drying time factors and drying rate is given in Fig. 9. According to the three-dimensional graphics when time is increasing, the drying rate decreases logarithmically, at the same time when the thickness of the material increasing drying rate increases linearly. The relationship with the thickness and drying time factors and moisture content is given in Fig. 10. According to the three-dimensional graphics when time is increasing, the moisture content decreases linearly, at the same time when the thickness of the material increasing moisture content increases linearly. The relationship with the thickness and drying time factors and moisture rate is given in Fig. 11. According to the three-dimensional graphics when time is increasing, the moisture rate decreases linearly, at the same time when the thickness of the material increasing moisture rate increases linearly.

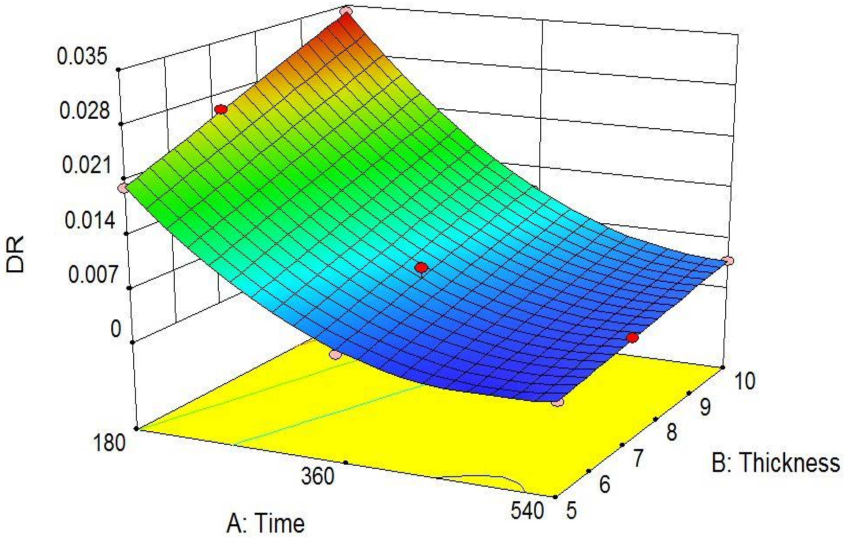


Fig. 9. Relations with the thickness and drying time factors and drying rate

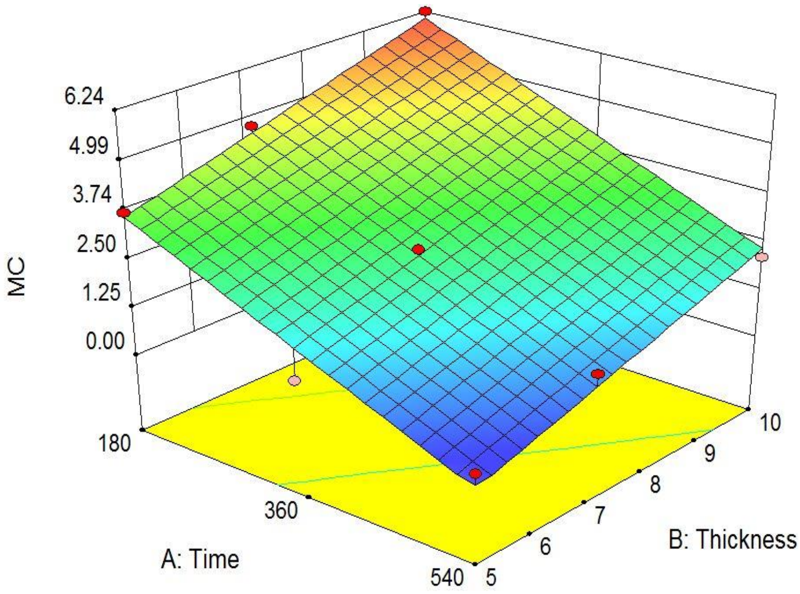


Fig. 10. Relations with the thickness and drying time factors and moisture content

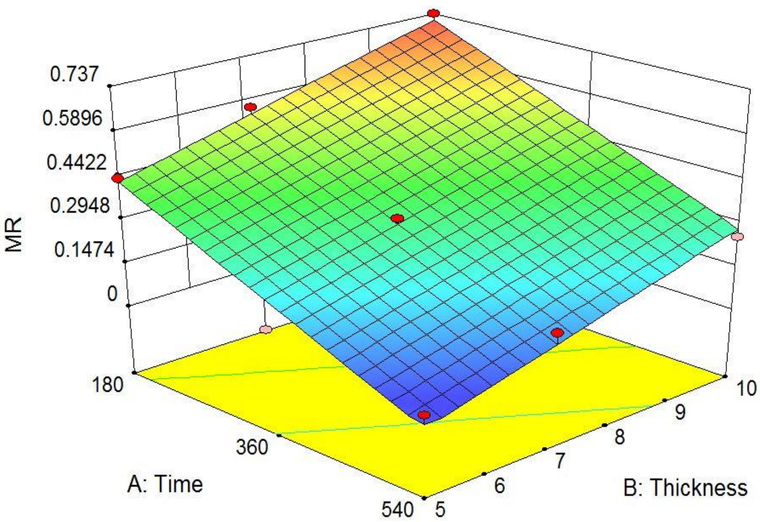


Fig. 11. Relations with the thickness and drying time factors and moisture rate

4. Conclusions

In this paper, the output variables including moisture content (MC), moisture ratio (MR) and drying rate (DR) values belonging to freeze drying behaviors of apples have been modeled and predicted with scaled input attributes comprising the sample thicknesses and drying time were investigated by using Taguchi technique. The aim of prediction algorithms is to approach the predicted (forecasted) results to experimental results. The unit of S/N ratio is decibel. The experimental results and corresponding S / N ratios are given and the ANOVA results are illustrated in Table 2. The standard deviations obtained from the ANOVA it is calculated as respectively MC 0.4338, MR 0.0512 and DR 0.0008. Because of the standard deviation of the low level, it is an indication of accuracy on obtained each successive test results and statistical calculations is the guarantee of accurate realization. According to the standard deviation especially it is seen that the sensitivity desired for a drying rate. C.V. % value means that the coefficient of variation from model and shows the error percentage. The lower value is made solutions that are highly sensitive. Obtained this analysis C.V. % value calculated as respectively MC 14.96, MR 14.96 and DR 6.99. R-Squared value is the total number of variations can be explained by the model. Adjusted R-Squared value is the total number of variations can be explained by the model when taken important factors. Predicted R-Squared value is the total number of variations of the model can explain the added predictability of new data. According to the results ANOVA, the model can explain the observed values of drying rate 99.76%. This value is obtained from statistical models show that highly accurate.

Table 2. The ANOVA analysis results which is applied Taguchi experimental matrix

	Response 1 MC **Moisture content**	**Response 2 MR** **Moisture Ratio**	**Response 3 DR** **Drying Rate**
Linear F-Test Values	< 0.0001 (selected)	< 0.0001 (selected)	0.0018
Quadratic F-Test Values	0.2514	0.2514	0.0013 (selected)
Standard Deviations.	0.433820453	0.051220286	0.000833119
C.V. %	14.96779781	14.96779781	6.998084973
R-Squared	0.963625501	0.963625501	0.997622737
Adjusted R-Squared	0.951500668	0.951500668	0.995245475
Predicted R-Squared	0.924077773	0.924077773	0.992910388
Signal to Noise(S/N)	24.65157114	24.65157114	54.09249997

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