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Abstract

In order to determine the optimal conditions for vacuum drying the olive cake, experiments based on the Taguchi method are carried out using an orthogonal $L_1s[9]$ three-factor table each having three levels [1]. The parameters studied are the depression exerted under the olive cake layer, the drying time and the thickness of the layer[2].

The test bench used to carry out the experiments is adopted from the patenting carried out by A.LAJDEL and M.MAZOUZI [3 , 4]. It comprises a vacuum pump sucking the air contained in a tank linked to a throat on which the olive cake layer is placed in position as shown in Figure 1.

The results obtained show that the optimal combination minimizing the water content of olive pomace is a thickness of 5mm, a drying time of 100 minutes and a negative pressure of -130 mbar which was identified as the most influencing factor.

Introduction

Morocco is known, like other Mediterranean countries, for its industry related to the extraction of olive oil. The latter poses serious environmental problems [5]. Residues from olive oil mills (olive cake and vegetable water) do not undergo any treatment and are often dumped into sewerage, stored in evaporation ponds or spread directly on the ground. This results in a negative impact on the environment which results in the pollution of soil and water (surface and underground) [6,7].

Olive cake is composed of skins, pulp residues, stone fragments, water and a significant amount of residual oil [9]. This biomass represents a renewable energy source for the production of thermal energy, electricity etc. Its recovery can thus represent both environmental and economic advantages.

The important techniques for valuing olive cake, such as the extraction of residual oil by solvents, its use as an additive for animal feed, their conversion into compost [10] and also its use as fuel in boilers [11] for various purposes (production of hot water, production of electricity, etc.), first require the reduction of its water content [12]. The drying process is therefore an essential step in its recovery.

Olive cake represents about 25% of the olives processed. Its water content at the outlet of the oil mill depends on the olive processing system. The olive cake produced by the pressure system contains a moisture content of about 30%. On the other hand, the olive cake obtained from centrifugation systems (3 and 2 phases) has a humidity level of between 45 and 66% [13]. Its recovery (solvent extraction of the residual oil, livestock feed, energy source, etc.) first requires lowering its water content to values between 5 and 12%. Drying is therefore an important step in its recovery.

Design of experiments is a methodology of choice which makes it possible to optimize the development of a product or an industrial process. In general, this methodology will seek to determine and establish the links between input variables of the process and a quantity of interest, called response. The variables, physical quantities modifiable by the experimenter, are supposed to influence the variations in the response. With the design of experiments, one obtains the maximum of information with the minimum of experiments.

There are many types of design of experiments which differ in the way they deal with the factors studied, the levels associated with these factors and the interactions that may exist [1]. The Taguchi methodology for implementing design of experiments, developed by Genichi Taguchi in Japan in the 1960s, uses fractional design

of experiments [1,13]. These are optimal designs that take into account the effects of factors and their interactions while dramatically reducing the number of tests to be performed. Taguchi's experimentation method uses a set of notions including the orthogonal table, the controlled factor, the noise factor, the signal-to-noise ratio, etc. Nowadays, this method is widely used in the industrial field [1,14].

The objective of this study is to optimize by the Taguchi method parameters such as the depression under the thin layer of the olive cake, the thickness of the layer and the drying time.

Signal-to-noise ratio and analysis of variance (Anova-Pareto) are used to interpret the experimental results.

Material and methods

Plant material

The olive cake samples were taken mainly at the level of the two-phase trituration unit located in the province of Taourit, region of the Oriental. This province is known for an important olive production, this is due to the large areas of olive tree plantation as well as to the characteristic climatic conditions. The olive cake that was the subject of this study was collected during the olive growing season November - March 2018.

The samples were taken directly from the outlet of the olive cake to the storage basin and must be homogeneous, and obtained without modification of their characteristics. The analytical results and their interpretation depend closely on the way of collection, packaging and the duration of storage. For this, our samples were taken in clean containers and then hermetically sealed without leaving air bubbles in the container. Then, transported as soon as possible to the laboratory to store them and subsequently perform the necessary tests [8].

The main characteristics of the olive cake studied are an initial humidity equal to 66% (wet basis) and a density equal to 0.9090 g.ml-1.

Drying

Drying is one of the most important preservation operations for many food products. It is often the last operation (after generally the evaporation operation) of the manufacturing process of a product. It is a thermal separation operation which consists in removing all or part of a liquid impregnating a so-called "wet" body by vaporization of this solvent. The final product is a solid qualified as "dry" even if it contains residual moisture.

The most widely used drying method in the food industry is entrainment drying: the temperature remains below that of boiling water and the entraining gas is generally air. It is the air which gives up its heat to the product and receives the moisture extracted from it. Drying is then defined as being a double transfer of heat and mass.

Understanding the phenomena involved in the drying of food products is mainly based on: (i) air-product balances; and (ii) the kinetics of drying and transformation of the product.

To evaporate water from a product, two mechanisms can be implemented: boiling and entrainment.

Physical principles of boiling drying

Drying by boiling consists in transmitting to the product brought to its boiling temperature a heat flow through a latent heat exchange surface. The evaporation of water is directly proportional to the energy input (latent heat of vaporization). In practice, this supply is carried out by conduction through the heat exchange surface in contact with the product by steam, the temperature of which is between 130 and 150 °C.

According to Fourier's law, the heat transfer takes place proportional to the temperature difference between the heat transfer fluid and the boiling liquid at the pressure considered.

Physical principles of entrainment drying

Entrainment drying involves placing a moist body in a stream of air (or other gas) that is sufficiently hot and dry. Under these conditions, a difference in temperature and partial pressure of water is spontaneously established between the body and the gas, such as:

- Heat is transferred from the air to the product, under the effect of temperature
- Water transfer takes place in the opposite direction due to the partial pressure of water between the air and the product surface (Figure 1)

Drying by entrainment is the most universal means of drying, all products and food sectors combined (milk, eggs, cold meats, cereals and vegetable products, fruits, etc.).

Figure 1: Transfer between air and product surface

Drying experiments

The diagram of the device for vacuum drying a thin layer of olive pomace is given in Figure 2.

It is composed of a sleeve (2) on which the thin layer is deposited, linked to a reservoir (4) put under vacuum by a vacuum pump (3). The depression under the layer is adjusted using a valve (1) included between the tank and the throat. The vacuum is measured by a vacuum gauge (6) placed on the tank. Once the drying depression is reached, the sample to be dried is placed on the top while starting the stopwatch. The measurements of the weight of the whole (support + sample) are carried out using a precision balance $(0.01 g)$. The dried sample is then brought to a temperature of 630 ° C using a dryer for 30 min to determine its dry mass (ms). The temperature of the room is kept constant throughout the experiment.

Figure 2 : Experimental device for vacuum drying olive cake

Water content of the product

The water content of the olive cake expressed relative to the dry matter (ms), denoted X (kg water $/$ kg ms), is given by the relation:

$$
X = \frac{mh - m}{ms}
$$

mh : wet mass of olive cake (kg) and ms its dry mass (kg). Reduced water content, noted X^* , is defined by the relation: $Xt-$

$$
X^* = \frac{Xt-}{Xc-Xe}
$$

Xt is the water content at time t (kg eau.kg⁻¹ms), Xe is the water content at equilibrium (kg eau.kg⁻¹ms) and Xc is the critical water content (kg eau.kg⁻¹ms).

In the case of organic products, Xc is equal to the initial water content Xo (due to the absence of phase 1 drying) and Xe is very low compared to Xt and Xo [15,16]. Thus, the expression of X $*$ can therefore be simplified as follows:

$$
X^* = \frac{xt}{x_0}
$$

It is this expression of the reduced water content that will be used as a criterion to be optimized by the Taguchi method in drying olive pomace under vacuum.

Taguchi method

The Taguchi method is used for optimization of process parameters and to identify the optimal combination of parameters for the desired responses.

Choice of factors and orthogonal table

Taguchi divides the factors into two categories: controllable factors and noise factors. The controllable factors of vacuum drying of olive cake examined in this study are: depression under the thin layer of the olive cake, thickness of the thin layer and drying time. The vacuum under the olive cake layer is adjusted using the valve (1) separating the tank (4) and the throat (2). The value of the depression under the layer of olive cake corresponding to the first

controllable factor is consistent with the value recorded within the reservoir indicated by the vacuum gauge (6), these two values are inversely correlated. When the first one argues, the other shrinks and lives towards it. The levels of each factor (3 in number), in coded values and in real values, are given in Table 1.c

With regard to the number of factors selected (03) and the levels assigned to each of the factors (03), the appropriate fractional factorial design for this study is an orthogonal Taguchi L18 matrix [9].

Signal to noise ratio

For the analysis of the experimental results, the Taguchi method uses a statistical measure of performance called the signal-to-noise ratio (Signal-to-noise, noted [S / N]). The relationship that allows its evaluation is different depending on the nature of the criterion to be optimized: targeting "Nominal-the-better", minimizing "Lower-thebetter" or maximizing "Higher-the-better" [17]. In this study, the aim is to minimize the reduced water content (X^*) of olive cake by convective drying. The relation of $[S/N]$ which makes it possible to determine the optimum corresponding to the minimum value of the response (Smaller-the-better) is:

$$
\left[S/N\right] = -10 \times log \left[\frac{1}{n} \sum_{j=1}^{n} \frac{1}{y_{ij}} \right]
$$

yij : value of the response obtained at the i experiment at its j repetition. n : number of repetitions of the test.

Results and discussion

The aim of this study is to optimize the parameters of vacuum drying of olive cake by minimizing its water content.

Kinetic drying curves

The kinetic curves of convective drying of olive cake $[X^* = f(t)]$ for different levels of depression and thicknesses of the thin layer of olive cake are shown in Figure 3.

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Figure 3 : Kinetic curves of vacuum drying of olive cake a) $e = 5$ mm; b) $e = 10$ mm; c) $e = 15$ mm

The appearance of the graphs suggests that the drying is rapid at the start of the process, then less and more quickly until reaching a value of the reduced water content of the olive cake of about 12% [15]. These results are in agreement with the results found for different plant products [16, 17, 18, 19].

The results obtained indicate that the depression and the thickness of the thin layer of the olive cake influence the duration of the drying process. To achieve a reduced moisture content of the olive cake of about 12%, the drying time is reduced by 55.0%, 42.0% and 40%, respectively, for thin film thicknesses of 5, 10 and 15 mm when the vacuum within the tank goes from -250 mbar to -130 mbar.

Optimization of drying parameters

The L18 orthogonal table used is given in Table 2. The experiment matrix is made up of 3 columns corresponding to the selected factors and 18 rows representing the 18 drying tests to be carried out. In the last two columns are reported, respectively, the experimental values of the reduced water content and the calculated values of the signal/noise ratio.

Table 2: LIS standard orthogonal table and experimental results						
Test n°				A B C Designation	X^*	[S/N]
			$-1 - -1$	AIBIC1	0,9145	1,339
				AIB2C2	0,8559	0.67
				A1B3C3	0.8336	0.79

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The results obtained indicate that the experiment n \degree 16 which gives the highest value of the ratio [S / N] (5.31) appears to be the optimum value. The corresponding value of X^* is equal to 0.2973, recommended value for the valuation of olive cake. The value of the lowest ratio (0.2711) is that corresponding to experiment n \degree 11.

Figure 4 shows the values of the average signal to noise ratio ($[S/N]$ m) as a function of the levels of the factors that affect the reduced water content of olive cake. A high value of [S / N] m indicates a large effect of the parameter on the reduced water content.

Under the experimental conditions studied, the results obtained show that the effect of the depression under the olive cake layer (A) on X * is the most important, followed by the effect of the thickness of the thin layer of the olive cake (B) and of the effect of time (C). The optimal drying conditions (minimum value of reduced water content) obtained are a negative pressure in the tank of -130 mbar (level 3), a layer thickness of 5 mm (level 1) and a drying time of 100 minutes (level 3). Thus, the optimal combination of input parameters that minimize the reduced water content of vacuum drying olive cake is: A3B1C3.

Figure 4 : Ratio Graph [S/N]m

Variance analysis (ANOVA- Pareto)

Analysis of variance (ANOVA-Pareto) was applied to analyze the contribution of each of the optimized parameters. It is a simplified method of the analysis of variance method, which is based on Pareto principles.

Anova-Pareto is an easy and fast analysis technique that does not require a Fisher test. The analysis of the drying results by this method using the S / N ratio data is given in Table 3. The Pareto chart is then plotted using the contribution ratio and the cumulative contribution (Figure 4).

Table 3 : Variance analysis (Anova-Pareto) of drying results

These results clearly show the predominance of the depression under the olive cake layer (factor A) with a contribution of 46% followed by the thickness of the thin layer of the sample (29%) and the drying time (26%).

Conclusion

In this study, the Taguchi method was used to optimize the vacuum parameters of olive cake. The L18 standard orthogonal table was used to conduct the drying tests. The results obtained and analyzed by the signal / noise ratio and the analysis of variance (Anova-Pareto) indicate that the depression under the olive cake layer is the most influential factor of the drying process with a contribution of 46%. The optimum values of the parameters determined by the [S / N] ratio are a depression within the reservoir of -130 mbar, a thickness of the thin layer of the olive cake equal to 5 mm and a drying time equal to 100 min.

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