



Contribution to the validation of thermal pasteurization treatment of green olives canned

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Abstract

This work is interested in the process of the validation of the thermal Scale applied by the company for canning of green olives of table. Scale conceived presents a tender of the product to a temperature T° constant of 90°C during at least 20min "Stage of T° ". In the course of the processing time, recordings of temperatures in the autoclave were carried out, mainly at the critical or cold points which present the center of the product and the medium of the 4 shopping carts arranged on a well-defined plane. Calculations of the pasteurization values and the parameters of transfers of heat in the product are given for the product in each shopping cart. The data obtained show that shopping carts 1.2 and 3 are located in a colder zone compared to the shopping cart 4 which is located in the zone which receives the most heat. However, the pasteurization values determined in the four shopping carts located at various places of the autoclave are all higher (VS1:34.6; VS2: 36.9; VS3: 34.9; VS4: 40.6) with the value $F_0=30\text{min}$ reference necessary for the pasteurization of green olives of table to $T^{\circ}=90^{\circ}\text{C}$. This confirms the attack of commercial sterility and the validity of the Scale. Organoleptic analyses of parameters are carried out to confirm nutritional and medical quality at the same time preserves of green olives produced by the company

1. Introduction

Canning is among the methods of preservation of green olives, it is a process which combines the preparation of food in limp of preserve tight and their pasteurization by heat. This heat treatment is applied in order to inactivate the enzymes and to destroy the micro-organisms, whose presence could make food unsuitable to consumption. However, absolute sterility does not exist; what one searches when a food is sterilized is commercial sterility where the risk of contamination is statistically improbable. In this objective, the food-preserving industry did not cease developing and seeking to diversify its methods and to make evolve its products [1]. The green table olives constitute the vegetable food product fermented most popular in Morocco [2]. Indeed the olive-growing sector, participant in 5% of the national GDP, with an annual production of 1 500 000 tons olives and 160 000 tons oil, remains one of these principal sectors [3].

Moreover, in the companies of preservation of green olives, stabilizing pasteurization is the heat treatment applied, especially for the acid products, to destroy the pathogenic germs. But this destruction of the microorganisms is made to the detriment of the nutritional and sensory qualities [4]. In fact, many vitamins and proteins are not very stable at the strong temperatures and of the tastes of cooked and the modifications of texture can also appear. Otherwise, the ideal heat treatment is that which guarantees at the same time the medical and nutritional quality of food. Thus, the current will of the canners is to move towards a reduction of the scales of sterilization or pasteurization to preserve final quality of the products, but by maintaining the security microbiological.

In the Moroccan companies of preservation of green olives, the heat treatment of olives is considered a point of risk to be controlled. Thus, the aims of this article is to validate the scale of canning applied by the company to green olives and to evaluate its effect on organoleptic and nutritional quality of these pasteurized olives.

2. Material and Methods

This study was carried out in a company located in the Marrakech-Tansift El Hawz region. Among the company's activities is the pasteurization of green olives. For this purpose, according to the HACCP approach, the thermal pasteurization scale has been identified as a critical point to be mastered in order to

meet both the health and nutritional quality of the product [5]. The treatment designed by the company corresponds to a temperature plateau of 90 °C for about 20 minutes.

The metal cans containing the olives are arranged in layers in baskets and then introduced into an autoclave, the start-up procedures of which have been established. Thus, the temperature evolution in the boxes judged in the coldest zones in the autoclave or the points having the most unfavorable conditions for the penetration of heat was recorded. In the baskets containing the olives boxes, the heat transfer characteristics were determined and the sterilization value was calculated in each box, using mathematical models (Combination of the methods of Ball et al. [6] and Bigelow [7] cited by Couvert [8]).

The pasteurization cycle in the autoclave was carried out in 3 steps: a first step of raising the temperature from about 20 to 25 min up to the operating temperature of 90°C; Followed by a pasteurization step of 20 min at 90°C; Then a cooling step of 30 min up to the temperature of 34°C.

The temperature in the autoclave is monitored by a HITEMP 140 recorder which uses the Evidencia Transit software to program, start, stop and download the data to be recorded. The test probes and the recorder are next to the mercury thermometer located in the middle of the autoclave, above the level of the baskets. The recorders were placed in the box in the center of the basket.

2.1. Calculation method

The pasteurization value represents the theoretical heating time of a required product at a constant reference temperature ($T_{ref} = 90^{\circ}\text{C}$, with $Z = 10$) equivalent to the time of the 3 phases (temperature rise, plateau and time Cooling) in order to reduce the microbial load in the desired proportions. First, for successive time intervals of duration $t_i = 1$ min, the temperatures T_i :

$T_i = \frac{T_{n-1} + T_n}{2}$; are measured in the product. Thereafter, the lethality value L is calculated at the temperature T_i for one minute:

$$L = 10^{\frac{[T_i - 90]}{Z}}$$

Z : temperature difference allowing reducing the time of the heat treatment by a factor of 10 for the same efficiency. The value of Z is conventionally set at ten (with reference to the Clostridium botulinum value). Thus, for each time interval $t_i =$ one minute at a temperature T_i , the pasteurization value VP_i can be calculated by the following equation:

$$VP_i = t_i * 10^{\frac{[T_i - 90]}{Z}}$$

VP_i : pasteurization value obtained at temperature T for one minute.

The calculation of the total pasteurization value for the entire heat treatment is done by integrating the rectangles of the temperature measurement curve using time / temperature pairs (Bigelow's rectangles method [5], according to Formula:

$$VP = \sum VP_i$$

2.2. Methods of nutritional analysis

The principles of the analytical methods used to evaluate the nutritional and organoleptic qualities are presented in the following Table 1.

3. Results and discussion

The heat treatment for the pasteurization of green table olives must have the aim of destroying or inhibiting, on the one hand the enzymes and on the other hand the microorganisms, the presence or the proliferation of which could alter these olives. It consists in placing the latter in the presence of a hot fluid during a certain time and then to cool them by bringing a cold fluid into contact. In these two cases, heat will be exchanged between the fluid and the product through the wall separating them, from the warmest to the coldest. This heat will also have to migrate within the product. The thermal evolution in the product results from two phenomena: a) heat migration through a wall and b) heat migration in the product either by convection following movements or displacements of matter or by conduction through exchanges between the hottest and coldest parts. Thus, in order to present the different phases of a conventional batch heat treatment and how it can be designed, controlled and validated, it is useful to propose a very usual graphical representation: recording the temperatures in the autoclave at During the processing time that are taken at the critical point of the product in the 4 baskets. It is a representation of thermal treatment scales in semi-

logarithmic coordinates, with the time t on the abscissa and the temperature at the critical point of the product at time t [16].

Table1: Analytical methods used to evaluate the nutritional and organoleptic qualities

Compounds	Methods	References
Protein Content	Method of Kjeldahl	Pomeranz et Clifton[9]
Sugar content	Method of Bertrand	Browne et Zerbán [10]
Lipid content	Method in Soxhlet	Horwitz [11]
Fatty acid	Chromatographic method Varian 5890	European Standard EN ISO 12966-2:2011[12]
Water	Steaming	Horwitz [11]
Mineral content	Atomic absorption spectrophotometry (UNICAM 929 AA "ATIUNICAM" spectrometer) on filtrates prepared from total	Method of Osborne and Voogt [13]
Phosphorus	vanadomolybdophosphoric method	Chang and Jackson [14]
Vitamin C content	Method using 2,4-dinitrophenylhydrazine	Sadasivam and Manickham [15]

The graphs (Figure 1 a, b, c and d) describe the temperature changes inside cans during the pasteurization cycle of green olives. They correspond to the temperature values inside the cans. One can very well distinguish the main stages. A phase of "Come Up Time" (CUT) which consists of a period of rise of the temperature of the enclosure approximately 25 min, to the temperature of regime ($T_r = 90^\circ\text{C}$), followed by a phase of During which the steady-state temperature is kept constant for about 20 minutes, and then a "Come Down Time" (CDT) phase consisting of cooling which lasts about 30 minutes. Thus, the heat treatment scale chosen by the company applied in autoclaving corresponds to the constant high temperature range 90°C . To which the products are subjected for about 20 min in order to end up with a reduction to 10-9 pathogen per box, or Rather a chance on a billion that a box contains a pathogen. This is the maximum risk accepted by the pasteurization standard for the marketing of the product.

This scale is chosen according to the nature of the product, its pH, the packaging, the initial number of microorganisms, contained in the product before pasteurization, the type of autoclave employed, the heating medium (water or steam) and the time taken to put the autoclave into operation [17,18,19].

Thus, the design of a pasteurization scale combines at the same time:

- The definition of the objective of thermal intensity and decontaminating efficiency to be achieved.
- The definition of the area of the product where this minimum treatment is to be applied: concept of a critical point of the product.
- The rate of penetration of heat into the product, itself influenced by a possible agitation.
- The choice of the treatment temperature in step.
- The initial temperature of the product.
- The characteristics of the material (autoclave) used.

As soon as the thermal characteristics of the product / packaging couple and the heat treatment equipment are known, the choice of the working temperature induces a scale time necessary to reach the objective of the pasteurizing value at the critical point. This scale duration can be determined by methods of predictive calculations (Ball method), by successive experiments (Bigelow or Flambert method) or by adapting an existing internal scale already validated for similar manufacturing conditions [20].

Taking into account, for example, the pH parameter, in products having a pH of less than 4.5 (between 3 and 4) such as olive preserves, the spores of the microorganisms do not develop. All fruits, including pH varies between 2 and 4.5, do not contain bacteria capable of generating spores. Unlike vegetables, meat and fish (pH 5-7.5), spores develop up to 120°C only when their pH is above 4.5. Under this barrier of 4.5, it is considered that a temperature varying between 80 and 90°C . is sufficient to sterilize and pasteurize the food of the microorganisms with the exception of yeasts and molds which develop on products between pH 2 and 10.

However, there is an antagonism between the "high" heat treatment necessary to guarantee the stability of the product and the sensory result of the products [21,22]. The control of the heat treatment is therefore a paramount parameter to guarantee the organoleptic qualities. In order to allow knowledge of the lethal effect of heat treatment, the notion of Sterilizing Value (SV) or pasteurizer (VP) was introduced as a "heat treatment intensity scale". By definition, VP is a heat treatment time, expressed in minutes, at a reference temperature (T_{ref}) which allows the destruction of an amount of target microorganisms whose thermal resistance characteristics are known.

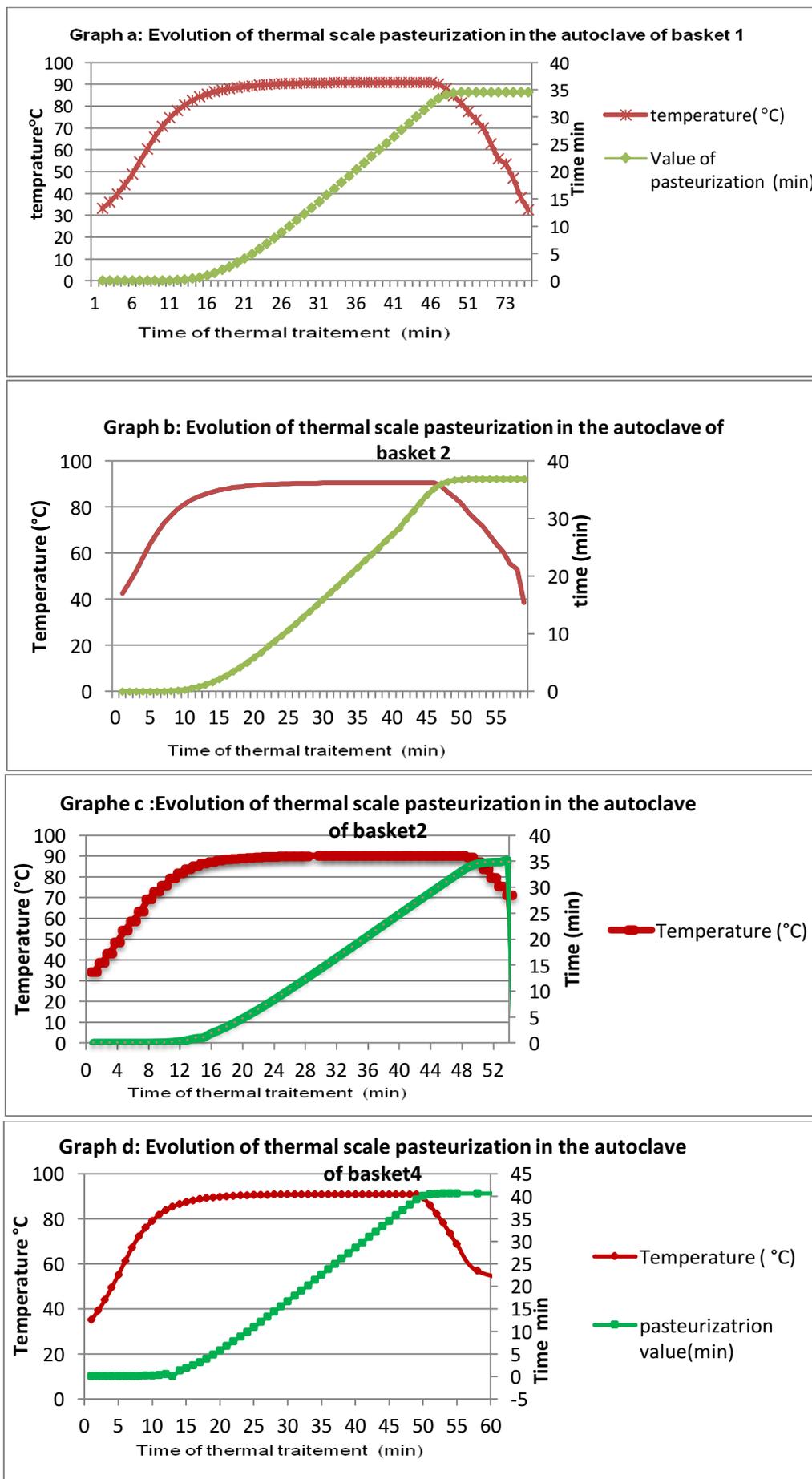


Figure 1: (Graphs a, b, c, d): Temperature recordings during the heat treatment applied for green table olives in the 4 baskets

In the literature, $F_0 = 30$ min is the pasteurizer sanitary value corresponding to the minimum heat treatment for acid preserves ($\text{pH} < 4.5$) at a temperature below 90°C . Thus, this pasteurizing value should have reached the cans in the coldest zone and corresponds to the critical point according to the HACCP plan.

Indeed, the key point in the design of a heat treatment is the knowledge and the quantification of the parameters describing the rate of heat penetration in the whole conditioned product (heating, and then cooling). In the process of appertization, the heat of the heating fluid, allowing the destruction of the microorganisms, is transmitted to the contents through the wall of the container and penetrates more or less rapidly inside the product to the slowest point at which is generally the center or also the furthest away from the wall (Figure 2).

From the mode of penetration of heat into the product, the notion of a "critical point" is also often called a "cold point": it is defined as the zone of the product that will receive during the complete thermal cycle the lowest intensity of treatment, i.e. the point of the product for which the pasteurizing value acquired is the lowest after treatment. It is of course in this zone that thermal measurements must be made for the monitoring of treatments.

The graphs (a, b, c, d) (Figure 1) represent graphically and in writing the evolution of the temperature as a function of time and the pasteurizing value calculated at each time interval t_i . The probes have been programmed to record the evolution every 60 seconds (our Δt is 1 min). Using these data, we were able to perform the calculations necessary to determine the pasteurizer value and the heat transfer characteristics across the product. Subsequently, after calculating the pasteurizer value F of the can which is at the center of each basket, a comparison is made between the values obtained in the different baskets and the reference pasteurizer value which is $F = 30$ min (Figure 2). These results allowed us to identify a VP value in all baskets greater than the reference pasteurizer VP value of 30. This shows the conformity and the validity of the scale (20 min, $T^{\circ}\text{max } 90^\circ\text{C}$) of heat treatment of green olives and the attainment of commercial sterility [23].

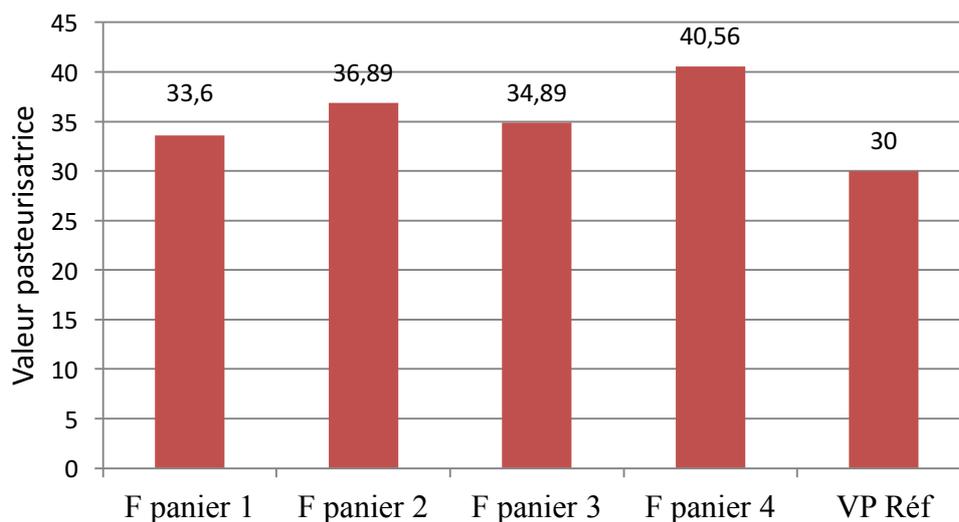


Figure 2. Graphical comparison of the pasteurizing values F of the baskets with the reference value F (Ref VP)

On the other hand, the test showed that the heat distribution throughout the autoclave is more or less homogeneous except in the basket 4 which has the zone where the pasteurizing value VP reaches 40.56. So you have to diagnose the autoclave, determine and correct the cause of this great value. In fact, heat treatments at constant temperature have disadvantages, especially for conductive products where heat is propagated step by step, and methods for optimizing these treatments have limits [24]. The scales thus calculated are often somewhat excessive and underestimate the thermal inertia of the products on cooling.

On the other hand, constant bearing scales always induce unnecessary over-firing of the conductive products [25]. These are the disadvantages of constant temperature scales, especially those calculated by the Ball method [6,8]. After heat treatment at constant temperature, these products exhibit a high heterogeneity of cooking between the center and the periphery, thus affecting the organoleptic characteristics of the product which should be preserved.

In order to ensure the validity of the pasteurization scale while maintaining the nutritional quality of the olives, organoleptic parameters of the green olives were monitored with other parameters that may reveal a reduction or a problem the nutritional quality of the olives (Table 2). These checks are carried out mainly for the research of the relation sterilization scale and organoleptic quality of the product [26-29]. Table 2 shows

that the main nutritional or organoleptic components to be tested show good nutritional, organoleptic and sanitary qualities of green olives with the pasteurization scale devised by the company.

Table 2. Analyzes of organoleptic parameters carried out on green table olives

Composition	Quantity	% AJR	Average difference cat.
Energetic value			
Energy - Calories	136 kcal	7%	-27%
Energy - kilojoules	557 kJ		-28%
Proteins	0.97 g	2%	-58%
Alcohol	0 g		-100%
Water	74.8 g		+8%
Carbohydrates:	0.54 g	0%	-92%
with sugar	0.54 g	1%	-85%
with starch	0 g		-100%
Lipids:	13.2 g	19%	-19%
of which saturated fatty acid	1.75 g		-51%
of which monounsaturated fatty acid	9.74 g		+42%
of which Polyunsaturated fatty acid	1.13 g		-77%
Minerals:			
Sodium	1680 mg	70%	+99%
equivalent in salt	4233.6 mg		
Magnesium	19 mg	5%	+4%
Phosphor	10.6 mg	2%	-79%
Potassium	39.3 mg	2%	-75%
Calcium	61 mg	8%	+31%
Manganese	0.05 mg	2%	-69%
Iron	0.96 mg	7%	+3%
Copper	0.21 mg	21%	+79%
Zinc	0.23 mg	2%	-33%
Selenium	0.9 µg	2%	-58%
Iodine	4.5 µg	3%	-66%
Vitamins:			
Vitamin A - Beta-Carotene	206 µg	26%	-9%
Vitamin A - Retinol	0 µg		-100%
Vitamin D	0 µg	0%	-100%
Vitamin E	1.99 mg	17%	-48%
Vitamin C	0 mg	0%	-100%
Vitamin B1	0.021 mg	2%	-60%
Vitamin B2	0.007 mg	0%	-94%
Vitamin B3	0.237 mg	1%	-50%
Vitamin B5	0.023 mg	0%	-87%
Vitamin B6	0.031 mg	2%	-66%
Vitamin B9	4.7 µg	2%	-68%
Vitamin B12	0 µg	0%	-100%

Conclusion

In this study, in order to determine the validity of the pasteurization scale (choice of the time /temperature torque of the heat treatment) in preserves of green table olives, it is necessary to know the temperature variations in the product over time. In practice, one follows the temperature at the coldest point of the product called "critical point" or center of the baskets located in different places. The validation of a heat treatment necessarily involves the calculation of the pasteurizing values VP. Indeed, the calculation of these VPs in the four baskets shows values higher than the reference pasteurizer value, which is a testament to the conformity and validity of the scale of green olives heat treatment applied by the company and the of commercial sterility. With the exception of basket 4, VP is excessive and can affect the organoleptic qualities of the product. Analyzes in this direction have been made and have confirmed that the nutritional and organoleptic qualities are not altered by this table of appertization while ensuring the sanitary quality of the green olives. Indeed, in order to optimize this heat treatment by taking into account these constraints and these requirements, other parameters deserve to be studied, namely the factors related to the product and to the packaging as well as the mastering of the cycle of autoclaving .

References

1. J.J. Bimbenet, A. Duquenoy, G. Trystram, Génie des procédés alimentaires, des bases aux applications, Paris: ed. Dunod., ISBN 9782100044351(2002) 553.
2. Y. Rokni, N. Ghabbour, N. Chihib, P. Thonart, A. Asehrou, *J. Mater. Environ. Sci.* 6 (6) (2015) 1740-1751
3. M.L. Ouaziz, M. Zaaraoua, A. Aboudia, A. Mouabad, A. El Antari, *J. Mater. Environ. Sci.* 7 (11) (2016) 4151-4157
4. N. A. Jarvis, C. A. O'Bryan, T. M. Dawoud, S. H. Park, S. C. Rieke, *Food Control*, 68, (2016) 280-290
5. H. Moumene, A. Hasib, C. Charraou, A. Jaouad, *Revue de Génie Industriel*, 8 (2012) 93-107
6. C.O. Ball, F.C.W. Olson, *Sterilization in food technology - Theory, Practice and Calculations*. First edition, McGraw-Hill Book Company, Inc. (1957).
7. W.D. Bigelow, *J. Infect. Diseases* 29, (1921) 528-536.
8. O. Couvert, *Thèse de doctorat. Université de Bretagne occidentale.* (2002) 180.
9. Y. Pomeranz, M.E. Clifton, *Food Analysis: Theory and Practice. Van Nostrand Reinold*, ISBN: 0442283164, *New-York, USA.* (1987)797.
10. C.A. Browne, F.W. Zerban, *Physical and Chemical Methods of Sugar Analysis*, third ed. John Wiley and Sons, (1955) 497-512.
11. W. Horwitz, (Eds.) (2002), "Official Methods of Analysis of the Association of Official Analytical Chemists", International (AOAC), vol. II, 43 Spices and Other Condiments, 43. 1. 02 Color Extractable in Spices. 17th ed. Gaithersburg, Maryland
12. Norme ISO 12966-2 : (2011), Corps gras d'origines animale et végétale-- Chromatographie en phase gazeuse des esters méthyliques d'acides gras - Partie 2: Préparation des esters méthyliques d'acides gras.
13. D. Osborne, P. Voogt, *Academic Press Inc., London Official Methods* (1978) 6.2- 6.3
14. S.C. Chang, M. L. Jackson. *Soil Sci.* 84(1957)133-144
15. S. Sadasivam, A. Manickam, *Wiely Estern Ltd., Madras* (1992).
16. F. Zuber, M. Biton, and A. Cazier, *Technique de l'Ingénieur*, (2008b) F 2032
17. M.H.S. Santos, H.N. Kalasic, A.C. Goti, M. R. Enguidanos, *Int. J. Food Microbiol.* 16 (1992) 275-281.
18. M. Lopez, M. Mazas, I. Gonzalez, A. Bernardo, Gonzalez, *J. Microbiol. Alim. Nutr.* 12, (1994) 317-322.
19. P.S. Fernandez, M.J. Ocio, F. Rodrigo, M. Rodrigo, Martinez. *Int. J. Food Microbiol.*, 32 (1996) 225-233.
20. F. Zuber, M. Biton and A. Cazier, *Bases Scientifiques pour la maîtrise des produits appertisés. Techniques de l'Ingénieur*, (2008a) F 2031.
21. A. R. Lespinard, R. R. Bambicha, R. H. Mascheroni. *Food and Bioproducts Processing*, 90 (4) (2012) 799-808
22. E. Sobczak, G. Cordier, *Information Technique du CTCPA*, 218 (2004).
23. L. Huang, *J. Food Eng.*, 79 (2007) 1166-1171.
24. T.R.A. Magee, *J. Food Eng.*, (1995) 223-232.
25. J.R. Banga, Perez-Martin, R.I. Gallardo, J.M and Casares. *J. Food Eng.*, 14 (1991) 25-51.
26. E. R. Bornhorst, J. Tang, S. S. Sablani, G. V. Barbosa-Cánovas. *LWT - Food Sc. And Technol.*, 82 (2017) 454-463
27. R. Simpson, H. Nuñez, S. Almonacid. 3: Modeling thermal processing and reactions: sterilization and pasteurization. Book chapter. *Modeling Food Processing Operations*, (2015) 67-93
28. R. Condon, C. Farrokh, K. Jordan, P. McClure, O. Cerf, *Int. J. of Food Microbiol.* 192 (2015) 20-25
29. D. Rodrigo, W. Tejedor, A. Martínez, *Encyclopedia of Food and Health*, (2016) 311-315

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