An Acad Bras Cienc (2020) 92(2): e20190072 DOI 10.1590/0001-3765202020190072
Anais da Academia Brasileira de Ciências | *Annals of the Brazilian Academy of Sciences*
Printed ISSN 0001-3765 I Online ISSN 1678-2690
www.scielo.br/aabc | www.fb.com/aabcjournal

ENGINEERING SCIENCES

Time and energy reduction on grape dehydration by applying dipping solution on freeze drying process

CICERÓN GONZÁLEZ-TOXQUI, ALVARO GONZÁLEZ-ANGELES, ROBERTO LÓPEZ-AVITIA & ISMAEL MENDOZA-MUÑOZ

Abstract: To preserve freshness and nutritional quality of fruits and vegetables is required large energy expenditure due to most storage techniques require low temperatures, making the product difficult to transport and store. Purpose: To reduce energy expenditure, dehydration processes are being improving by pretreatments and changing frozen stage. Method: Alkaline emulsion pretreatment was introduced to freeze-drying method to reduce the drying time of grapes and obtain significant energy savings. Results: Dehydration process for untreated fresh grape samples was 22 hours, using industrial freeze-drying equipment. It was obtained a high quality product with range 3 % to 7% of final humidity, without losing natural characteristics and organoleptic properties of the grapes. Conclusions: it was found by modifying standard equipment frozen method, using dry ice (CO_2) and applied coconut oil alkaline emulsion pretreatment, a 54% energy saving. Even more the dehydration process decreased the microbial load in the fruit. A product with low number of microorganisms may be consumed by patients with low defenses as cereals, nutritional bars, salads, yogurts, etc. Finally, if all enterprises involved in this field take into account these findings and improve constantly their process they will stop emitting several kg of CO2 into the atmosphere.

Key words: Freeze drying, dehydrated grape, dehydration process, energy saving.

INTRODUCTION

Nowadays, to preserve freshness and nutritional quality on fruits and vegetables are applied several methods, due to a different perishable factors as fungi, yeasts, bacteria and enzymes (De Torres et al. 2015). When it is exposed to open air and high temperatures, the decomposition process is accelerated, having a relatively short life. When this product is decaying, it is harmful to health and useless.

Most storage techniques require low temperatures, making the product difficult to transport and store, and increasing energy expenditure. On the other hand, drying methods change the appearance and physical structure of products, giving advantage in transport, since it is extremely lightweight and covers less space than a fresh product to store and send, impacting positively, handling and large scale transport of product. There are several dehydration methods such as osmotic dehydration, vacuum, ultrasound, heat-pump, microwave, freeze-drying, spry, and a combination thereof (Candia-Muñoz et al. 2015, Fernandes et al. 2011, James et al. 2014, Sagar & Suresh Kumar 2010, Samoticha et al. 2016, Souza et al. 2014).

Freeze-drying is an interesting alternative as a food preservation method (Pei et al. 2014, Vallan 2007), which allows extending the lifetime significantly, while maintaining its quality-related physical and physicochemical properties (Fabra et al. 2009). This method consists in removing water from the product by sublimation, the product is frozen and then vacuum is applied to sublime the frozen water, along with evaporation step for remaining non-frozen water (James et al. 2014). Sublimation occurs when the vapor pressure and temperature of the ice surface is below to triple point of water (Ghio et al. 2000).

Freeze-dried fruits and vegetables are considered a healthy product that can be consumed at any time; raw or cooked can be eaten, and are available throughout the year (Park et al. 2015). At the same time, this kind of food can encourage the intake of fruits and vegetables, so it aims to reduce obesity and illnesses associated with high intake of fats and carbohydrates that are currently present in population diseases (Basu et al. 2009, Dávila-Torres et al. 2015). The preparation of the product aims to satisfy the necessities of different markets. In a way, it seeks a place in the mind of the consumers, and arrive them by producing “fruit snacks” such as cereals, nutritional bars, complements salads, etc. On the other hand, the largest market segment (food processing industry), they use dried fruits as raw material in the production of yoghurt, ice cream, bakery, etc (de Lima et al. 2016).

The food research has made great progress in the conservation area, as well as in industry demand for obtaining high quality product and high life (Zhang et al. 2006). Throughout history to keep food in optimum condition for consumption, it has been and will be an outstanding issue and a challenge for technology development, process innovation and energy saving.

In this investigation, an alkaline emulsion pretreatment was introduced to freeze-drying method to reduce the drying time and achieve significant energy savings in food preservation.

MATERIALS AND METHODS

Fresh grape was obtained in the local market (vitis vinifera). 5.4 kg of whole grapes were used in the experiment. All specimens had good condition, no cuttings or damage surface, not showing low quality evidence. They were washed with water and dried at room temperature.

Moisture content in the samples was determined by a thermo balance (Ohaus MB-35 model with halogen source heating), which evaluates moisture percentage and weight (g). Sample testing factors 105 °C for 3.5 minutes. In fact, weighing was performed on same digital balance skills and two replicate samples were selected and weighed. The moisture content of all the rehydrated samples was also determined.

Two alkaline emulsion were prepared by mixing 1 liter of water, 0.05 kg of K_2CO_3 (with 98% of purity, ASC reagent, Aldrich Co.) and 0.005 kg of olive oil (pH 12); and the another 1 liter of water, 0.05 kg of K_2CO_3 and 0.005 kg of coconut oil (pH 11)(Doymaz & Pala 2002). The samples (5g) were immersed in the solution for 1 minute at room temperature.

For freeze-drying process was used the Sanshon equipment model FDG - O.5(Zhejiang sanshon machinery manufacturing CO. LTD.), with 31 cm x 54 cm chamber process area, 0.5 m^2 platform drying area and 5 liters of water capacity every 8 hours in drying process. It possesses a pump vacuum of 6 x $10.^{-1}$ mbar model TRIVAC D16B, 3-ph, 240-265/415-480 V and 60 Hz. In addition contains a vacuum chamber, equipped with three heating plates (80 °C) and three shelves where the specimen is placed, freezing chamber (-40 °C), water heating system (105 °C).

The dehydration process was performed by stages; first, the samples (1.6 cm) was frozen for 3 hours at - 35° C. Once the sample is frozen, a vacuum pressure of 6 x 10^{-1}mbar is applied. After, two temperature ramps were programmed, one for sublimation at 80 °C for 5 hours and another for drying the remnant water (14 h)at 60°C. The specimens were analyzed in a microscope under visible light using a digital camera (AxioCam, Carl Zeiss, Germany).

Frozen is the first step on freeze dried method, the vacuum chamber on freeze dried equipment is also equipped by frozen capacity, reaching -40°C in process. Additionally, the frozen phase on equipment was change for CO_2 (dry ice), reaching -78.5°C instead -40°C. Diverse soaking times were used, 3 hours, 1:30 hour, 10 minutes and 5 minutes in order to reduce the freezing time and analyze the quality impact on final product.

For bacterial analysis was used Gram staining technique. Due to this method allows distinguishing between two groups of Gram positive and Gram negative bacteria, as behave of staining. The smear slide was heat-fixed for one minute with Crystal Violet stain, washed with water, covered with Lugol solution for 1-2 minutes, and washed again with water, decolorized mixture ethylether / acetone alcohol. Drained and covered with Safranin (color contrast) for 1 to 2 minute, washed and dried (Camacho et al. 2009).

The morphology of the studied sample was observed in an optic microscope (Carl Zeiss, Germany, under visible light) and in a scanning electronic microscope JEOL JSM- IT 100.

RESULTS AND DISCUSSION

For untreated freeze-drying process samples described above, it is necessary 22 hours. It is important to highlight that to determine the drying time for the untreated sample (evaporation of remaining water at 60 °C for 14 h), several soaking times were performed to obtain final moisture range content of 3% to 7 % and suitable consistency of final product. In other words, the process reached the optimal moisture in shorter time, (8 hours) but the sample showed a soft and sticky consistency, surface sample does not show crystallizes appearance and had a honey consistency in surface, which should be crystallizes and bright appearance surface. It was up to 14 hours vacuum time in dried stage when the specimen got proper consistency and appearance.

To the pretreated sample (immersed for one minute in the alkaline solution), the same procedure was performed to find the appropriate drying time (final stage of the freeze-drying). It was found that with 6 hour obtained 7.57% final moisture and crunchy taste (fructose vitrified), obtaining a process total time of 14 h. Comparing the total times of freeze-drying process for grapes without treatment (22 h) and the treated samples(14 h), it observes a 36% time saving.

The saved time on drying process was due to alkaline emulsion impacts on waxy surface sample. It is well known that cuticle prevents plant surfaces from both becoming wet and helps to prevent plants drying out (control the moisture

On the other hand, it is important to mention that the frozen stage must be as quickly as possible due to faster the frozen of product, smaller ice crystals will be formed and therefore less damaging to the cell wall. Due to above mentioned, the freeze drying process was modified in the first stage, using dry ice to frozen the samples at different soaking times (3 hours, 1:30 hours, 10 minutes and minutes), keeping the stages of sublimation and dried at 5 hours each one.

The results show that samples (with frozen soaking time of 3 hours, 1:30 hours and 10 minutes) have good appearance, bright crunchy and final moisture of 3.02%. The 5 minutes frozen sample does not look good, low quality and dark color with 6.20% of moisture. Comparing full time between normal freeze dried process (frozen 3 h, sublimation 5 h and dried 14 h) and modified frozen phase for coconut oil (10 minutes frozen, sublimation 5 h and dried 5 h), it obtains a 54% of energy saving. For olive emulsion dehydration time was very similar to coconut oil (10 minutes frozen, sublimation 5 h and dried 6 h), having 50% of energy saving. Due to above mentioned the coconut oil is slightly better than olive oil.

The figure 1 shows grape sections obtained after being immersed in the alkaline solution and phenolphthalein, in order to determine the penetration of the alkaline solution. The red color reveals the presence of the alkaline solution, at the same time shows that does not reach the pulp, the alkaline solution impacts only in skin sample.

After freeze-drying process, the product was carefully packaged and sealed in bags for its conservation and storage, due to it has the hygroscopic property (absorbs moisture).

On the other hand, some dried samples were rehydrated and its humidity was both measured and compared with that of fresh product. In table I, it can see that humidity achieved in rehydrated samples (coconut oil alkaline solution) was 75 % as compared with initial humidity of fresh grapes.

As freeze-dried samples were assessed their organoleptic properties (taste, odor and appearance) showing optimal conditions for consumption. The flavors highlight the palate with strength when wet in the mouth, and the odor is extremely concentrated and pleasant to smell, tends to be sweet smell. It has a slightly wrinkle appearance, with crispy consistency and intense shiny color, due to grape skin membrane retains the sugar, and it is crystallized by drying process (Figure 2).

The organoleptic properties for rehydrated samples are slightly low than fresh grapes (less intense flavor and dull color) but consistency is almost same than fresh product (Figure 3).

Regarding frozen are two types, slow speed and quick speed. Freezing process play an important role in freeze-drying method. In this investigation, quick freezing was used, due in samples forms small crystals water and therefore less damage as possible in cell membrane, otherwise, slow frozen process developed big crystals water and the cell membrane can damage by these crystals. This effect can affect the sample appearance, also smell and taste on final products, as well on process parameters, which is highly important in freezing process and good quality product.

It is important to remember that any fresh product subjected to freezing and thawed, does not have the same fresh look, as there was a broken cell membranes by the crystals freezing process. The figure 4 shows as the freeze drying process helps the decrease of microbial load. To have low number of microorganisms is of particular interest for patients with low defenses.

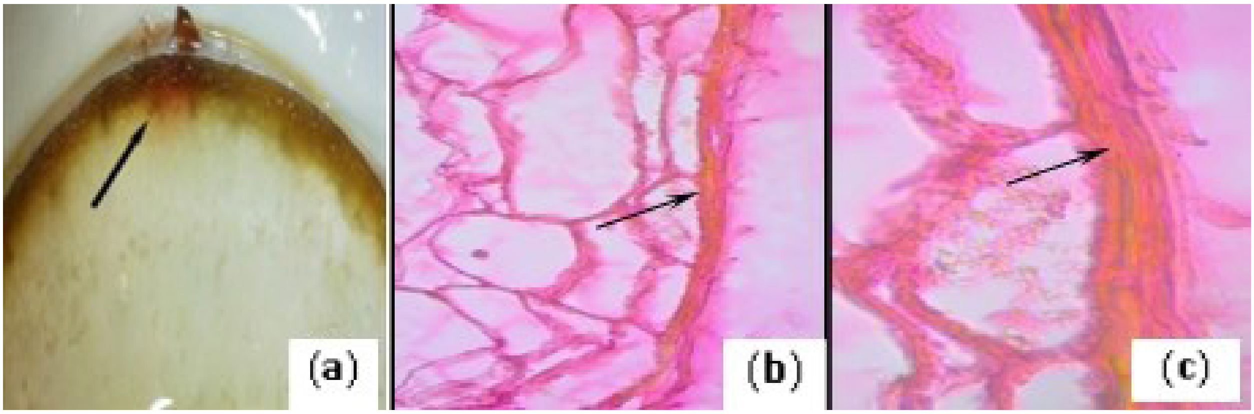

Figure 1. Grape sections after being immersed in the alkaline solution. The phenolphthalein turned bright magenta immediately on contact with the alkaline solution: (a) arrow pointing grape skin 32 x magnification microscopy, (b) arrow pointing grape skin 400 x magnification microscopy, (c) arrow pointing grape skin 1000 x magnification microscopy.

Table I. Shows moisture percent in product through different steps of dried process.

Product	Initial Moisture As raw material	Final Moisture As freeze-dried	Rehydrated
Grape	69.77%	4.58%	52.56%

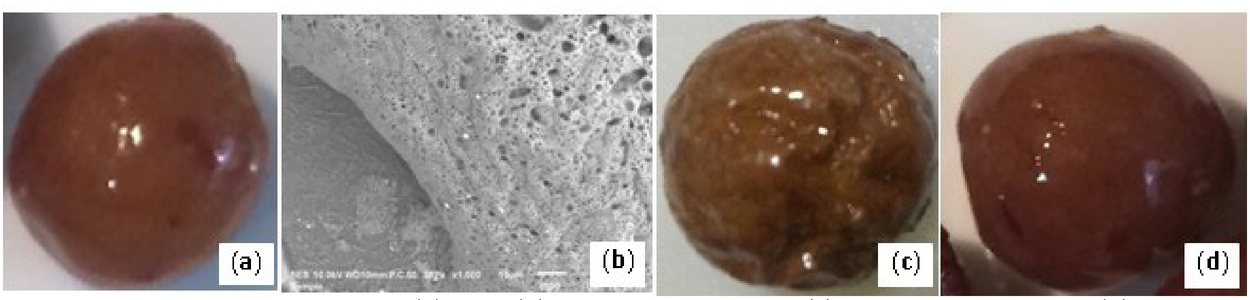

Figure 2. Grape exterior view, in stage (a) fresh, (b) as freeze-dried in SEM, (c) as freeze-driedand and (d) rehydrated.

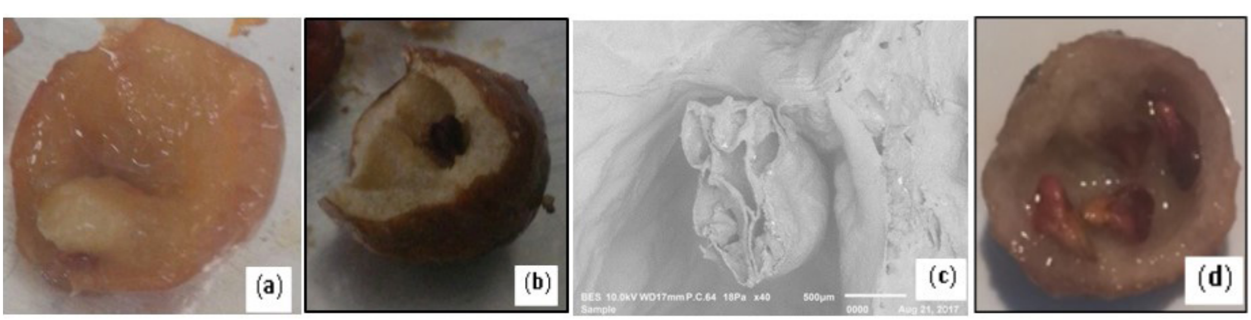

Figure 3. Grape sectional view: (a) fresh, (b)as freeze-dried, (c) as freeze-dried in SEM and (d) rehydrated.

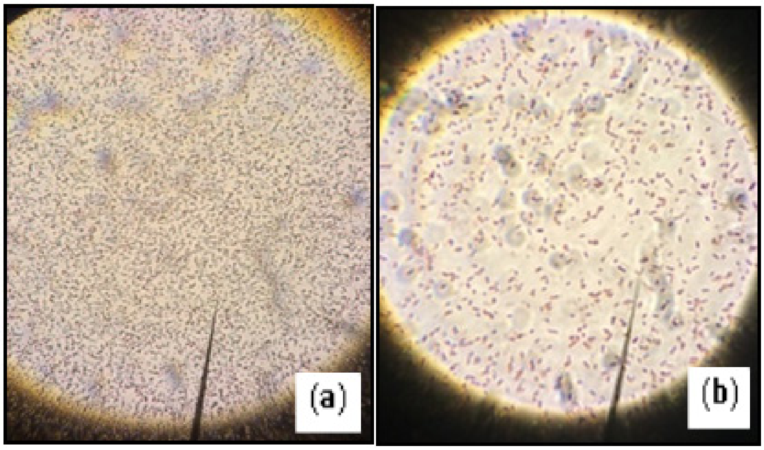

Figure 4. Microbial load on grape sample before (a) after (b) freeze dried process 40 X and 100 X respectively.

CONCLUSIONS

In freeze drying grape sample process the alkaline emulsion pretreatment generated 36% time saving. Modifying standard equipment frozen method by using dry ice (CO_2) and applied coconut oil alkaline emulsion pretreatment, shows a 54% energy saving. Coconut oil emulsion is lightly better than olive oil emulsion.

For dried samples were obtained a range 3% to 7% of final moisture. The humidity achieved for rehydrated samples was 75% as compared with initial humidity of fresh grapes. Freeze-dried product can be consumed or used on several ingestion types as dehydrated (cereals, nutritional bars, complements salads, etc), or in aqueous solutions (yogurts, shake milk, etc). After freeze-drying process, products have hygroscopic properties and high considerations on storage conditions must be applied, such as storage in cool, dry place away from sunlight, thus properties will be preserved to either rehydration or consumption. Finally, if all enterprises involved in this field take into account these findings and improve constantly their process they will stop emitting several kg of CO_2 into the atmosphere.

Acknowledgments

The authors thank the National Council for Science and Technology of Mexico, grant no 692364. Therefore, we want to acknowledge PhD Nicolás Serafín Higuera and M SC Gustavo J. Martínez Coronilla from Universidad Autónoma de Baja California.

REFERENCES

BASU A, WILKINSON M, PENUGONDA K, SIMMONS B, BETTS NM & Lyons TJ. 2009. Freeze-dried strawberry powder improves lipid profile and lipid peroxidation in women with metabolic syndrome: baseline and post intervention effects. Nutr J 8(1): 1-7.

CAMACHO A, GILES M, ORTEGÓN A, PALAO M & VELÁZQUEZ O. 2009. Método para la determinación de bacterias coliformes , coliformes fecales y *Escherichia coli* por la técnica de diluciones en tubo múltiple (Número más Probable o NMP). Tec Anal Microbio Alim 2(1): 1-17.

CANDIA-MUÑOZ N, RAMIREZ-BUNSTER M, VARGAS-HERNÁNDEZ Y & GAETE-GARRETÓN L. 2015. Ultrasonic spray drying vs high vacuum and microwaves technology for blueberries. Phys Procedia 70: 867-871.

DÁVILA-TORRES J, GONZÁLEZ-IZQUIERDO J & BARRERA-CRUZ A. 2015. Panorama de la obesidad en México. Rev Med Inst Mex Seg Soc 53(2): 240-242.

DE LIMA MM, TRIBUZI G, DE SOUZA JAR, DE SOUZA IG, LAURINDO JB & CARCIOFI BAM. 2016. Vacuum impregnation and drying of calcium-fortified pineapple snacks. LWT- Food Sci Tech 72: 501-509.

DE TORRES C, SCHUMACHER R, ALAÑÓN E, PÉREZ-COELLO S & DÍAZ-MAROTO MC. 2015. Freeze-dried grape skins by-products to enhance the quality of white wines from neutral grape varieties. Food Res Int 69: 97-105.

DOYMAZ I & PALA M. 2002. The effects of dipping pretreatments on air-drying rates of the seedless grapes. J Food Engr 52(4): 413-417.

FABRA MJ, TALENS P, MORAGA G & MARTÍNEZ-NAVARRETE N. 2009. Sorption isotherm and state diagram of grapefruit as a tool to improve product processing and stability. J Food Engr 93(1): 52-58.

FERNANDES FAN, RODRIGUES S, LAW CL & MUJUMDAR AS. 2011. Drying of Exotic Tropical Fruits: A Comprehensive Review. Food Bioprocess Tech 4(2): 163-185.

GHIO S, BARRESI AA & ROVERO G. 2000. A Comparison of Evaporative and Conventional Freezing Prior to Freeze-Drying of Fruits and Vegetables. Food Bioprod Process 78(4): 187-192.

JAMES C, PURNELL G & JAMES SJ. 2014. A Critical Review of Dehydrofreezing of Fruits and Vegetables. Food Bioprocess Tech 7(5): 1219-1234.

PARK JN, SUNG NY, BYUN EH, BYUN EB, SONG BS, KIM JH & LYU ES. 2015. Microbial analysis and survey test of gamma-irradiated freeze-dried fruits for patient's food. Radiat Phys Chem 111(1): 57-61.

PEI F, SHI Y, MARIGA AM, YANG W, TANG X & HU Q. 2014. Comparison of Freeze-Drying and Freeze-Drying Combined with Microwave Vacuum Drying Methods on Drying Kinetics and Rehydration Characteristics of Button Mushroom (Agaricus bisporus) Slices. Food Bioprocess Tech 7(6): 1629-1639.

SAGAR VR & SURESH KUMAR P. 2010. Recent advances in drying and dehydration of fruits and vegetables: A review. J Food SciTech 47(1): 15-26.

SAMOTICHA J, WOJDYŁO A & LECH K. 2016. The influence of different the drying methods on chemical composition and antioxidant activity in chokeberries. LWT - Food Sci Tech 66(1): 484-489.

SOUZA VB, DE FUJITA A, THOMAZINI M, DA SILVA ER, LUCON JF, GENOVESE MI & FAVARO-TRINDADE CS. 2014. Functional properties and stability of spray-dried pigments from Bordo grape (*Vitis labrusca*) winemaking pomace. Food Chem 164: 380-386.

VALLAN A. 2007. Measurement system for lyophilization process monitoring. 2007 Ieee Instm Meas Tech Conf 1-5: 1512-1516.

ZHANG M, TANG J, MUJUMDAR AS & WANG S. 2006. Trends in microwave-related drying of fruits and vegetables. Trends Food Sci Tech 17(10): 524-534.

How to cite

GONZÁLEZ-TOXQUI C, GONZÁLEZ-ANGELES A, LÓPEZ-AVITIA R & MENDOZA-MUÑOZ I. 2020. Time and energy reduction on grape dehydration by applying dipping solution on freeze drying process. An Acad Bras Cienc 92: e20190072. DOI 10.1590/0001-3765202020190072.

Manuscript received on January 23, 2019;
accepted for publication on October 16, 2019

CICERÓN GONZÁLEZ-TOXQUI
https://orcid.org/0000-0001-7603-4774

ALVARO GONZÁLEZ-ANGELES
https://orcid.org/0000-0002-9475-5759

ROBERTO LÓPEZ-AVITIA
https://orcid.org/0000-0003-3615-6560

ISMAEL MENDOZA-MUÑOZ
https://orcid.org/0000-0002-0810-2090

Universidad Autónoma de Baja California,
Facultad de Ingeniería, Blvd. Benito Juárez, s/n,
21280 Mexicali, Baja California, México.

Correspondence to: **Alvaro González-Angeles**
E-mail: gangelesa@gmail.com

Author contributions

C.G.T. accomplished the study. A.G.A. drafted the manuscript. R.L.A. contributed substantially to the interpretation of results. I. M. M facilitated the freeze-drying equipment. All authors have critically revised and contributed to the final version of this manuscript