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COMPARISON OF TWO ALTERNATIVES OF COMBINED DRYING TO PROCESS BLUEBERRIES (O'Neal): EVALUATION OF THE FINAL QUALITY.

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Abstract

In this work, we examined and compared two combined alternatives for the drying of blueberries (**O'Neal**). Pretreatments of osmotic dehydration (60°Brix sucrose solution, at 40°C for 6h) and hot air drying (60°C, 2.5 m/s for 90 min) were performed to reach the same water content. Pretreated blueberries were then dried by microwave at different microwave output power values: 562.5W, 622.5W and 750W. The combined drying processes were also compared with hot air drying alone (control). The effects of the processes over blueberries were studied in terms of decrease in water content, drying rate, mechanical properties (firmness and stiffness), optical properties (L*, a* and hue angle (*h*)), antioxidant capacity and rehydration capacity. The hot air-microwave drying decreased the process time and presented a high drying rate compared with the osmotic dehydration-microwave processes and the control drying. In terms of quality, the antioxidant and rehydration capacities were the most affected. The results showed that the best drying method to obtain the desired final product was the hot air-microwave drying (750W).

KEYWORDS: Blueberries, combined drying, texture, color, antioxidant capacity, rehydration capacity

1. INTRODUCTION

Blueberries (*Vaccinium corymbosum* cv. O Neal), from the Southern highbush blueberries group, are one of the highly productive varieties commercially grown in Argentina. Blueberries are characterized by being rich in antioxidants, by having attractive sensorial attributes and by being increasingly requested by consumers, since they contribute to the nutritional quality of a diet [1]. However, the high perishability and limited shelf-life of blueberries is a common problem for their production. Processing methods such as dehydration are usually applied to extend their commercial life and even allow reducing storage and transportation costs [2, 3]. More recently, researchers focus on improving drying techniques and also meeting consumer demand for high-quality dried commodities [4].

Value-added products based on blueberries could be designed by applying combined drying processes [5]. These processes include a mild pretreatment step such as osmotic dehydration, hot air drying at low temperature or solar drying, followed by microwave drying or another finishing drying method to reduce the disadvantages of using hot air drying alone and thus preserve the final product quality. The synergistic and additive effect of both drying stages allows preserving the food with a higher quality than when using only one method such as conventional drying [6].

Several authors have studied osmotic dehydration as a pretreatment and have found that it is a good alternative to preserve the attributes and nutritional value of fresh fruit [2, 5 7]. Osmotic dehydration consists in the immersion of fruit in a hypertonic sugar solution. The sugar infusion allows removing a large amount of water and introducing sugar into fruits. However, a complementary method is required to produce a stable product. Microwave drying of osmotically dehydrated products has demonstrated to improve the drying rate and maintain product quality [8, 9].

Another alternative may be the combination of hot air drying and microwave drying. Hot air drying is the most common method used to dehydrate fruit. However, the main disadvantage of this process is that it takes a long time, even at high temperatures, which degrades the quality of the dried product [10]. On the other hand, microwave drying has been of great interest in the food industry because microwave waves may penetrate through untreated or partially dehydrated samples. This type of drying causes heating in the wet parts and increases the drying rate. Several authors have proposed that the combination of microwave drying and hot air drying may be used to overcome the disadvantages associated with the application of each method alone [10, 11, 12]. The application of hot air drying followed by microwave drying can reduce the costs and process time and obtain high-quality final products.

The aim of this work was to compare two combined drying processes, between them and with a control drying. We determined both the effect of the process variables over the efficiency of the drying time and the different quality parameters of blueberries (cv. O'Neal) required to obtain a final product that can be used as a food ingredient.

2. METHODS AND MATERIALS

2.1. Blueberries

Blueberries (*Vaccinium corymbosum* cv. O'Neal) were purchased from Don Pedro establishment in San Pedro (Buenos Aires, Argentina) and stored at 0°C. Previous to the drying process, blueberries were washed and dried with tissue paper. The initial characteristics were: water content 83.44±1.01% (wb), soluble solids 13.5±0.8 (°Brix) and initial water activity 0.978±0.001.

2.2. Chemical Pretreatment

Previous to the drying process, blueberries were dipped on alkali emulsion of ethyl oleate (AEEO) for 1.5 min at 60°C, to increase the permeability of the skin. The AEEO was prepared by mixing distilled water, ethyl oleate (2% v/v) and sodium hydroxide (0.5% w/v). The concentrations applied have been reported previously [13].

2.3. Experimental Design Of The Drying Process

Combined processes were performed in the following way: an initial stage where a portion of blueberries was pretreated with osmotic dehydration (OD) and another portion with hot air drying (HAD), and a second stage in which all pretreated blueberries were exposed to microwave drying (**MWD**). All the drying processes were performed in

triplicate. The conditions of the pretreatments OD and HAD were selected by preliminary tests [14].

OD was performed in a solution of commercial sucrose of 60° Brix at 40° C for 360 min with constant agitation (100 rpm) in a shaker (brand Ferca, Argentina). The fruit/syrup relation selected was 1:20 and the sample amount used was 90 ± 0.5 g.

HAD was performed in a horizontal prototype drier (Didacta Italia) adapted in our Institute, supplied with stainless steel trays. The conditions were air temperature at 60° C and air speed 2.5m/s for 90 min. This pretreatment time allowed reaching the same water content as that of OD, to compare both combined drying methods. The control drying was performed in the same conditions but for 480 min. The sample amount used was $60\pm0.5g$.

MWD was performed with a domestic microwave oven (brand Whirlpool, model JT 359, China) with the incorporation of an autotransformer (El Toroide, Argentina), which allowed working with the magnetron set at the highest power (750W) in a continuous way (without the on/off cycles of the normal control) and at different discrete levels of power (100%, 83% and 75% of the set power). In this way, the operation conditions simulated the performance of continuous industrial equipment. Each percentage set in the autotransformer was imposed to the **microwave oven working at its maximal power to obtain:** 750W (100%), 622.5W (83%) and 562.5W (75%). The sample amount for each experiment was $24.5 \pm 0.5g$.

Blueberries were dried until reaching a water activity value < 0.6. In this way, dried blueberries were considered stable against the microbiological agents and chemical reactions. On the other hand, all processes were performed within a total operation time similar to that of a workday in the food industry (8h).

2.4. Determination Of Water Content And Water Activity

Water content (WC) was determined by drying the blueberries in an oven (Gallenkamp, UK) at 70°C until constant weight (24h), according to the AOAC method with some modifications [15]. Water content was calculated using equation (1):

$$WC = \frac{m_w}{m_t} * 100$$
 (1)

Where WC is the water content (% -wb-), m_t is total mass (g), and m_w is water mass (g). All measurements were performed in duplicate.

Water activity (aw) was determined at 25°C by the AOAC hygrometric method 978.18 [15], using a temperature-controlled AquaLab 3TE meter (Decagon Devices, Inc.). All measurements were performed in duplicate [16].

2.5. Drying Rate

The moisture ratio (MR) of samples was calculated using equation (2):

$$MR = \frac{Mt - Me}{Mo - Me} \cong \frac{Mt}{Mo}$$
(2)

where Mt is the moisture content at any time (kg water/ kg dry solid), Mo is the initial moisture content (kg water/ kg dry solid), and Me is the equilibrium moisture content of

the sample (kg water/kg dry solid). This equation can be simplified because the values of Me are relatively small, hence the error involved in the simplification is negligible [17, 18].

The *drying rate (DR)* was calculated as the derivative of MR over time, considering that MR has an exponential decay with time:

$$MR = a * e^{(-k^*t)} \tag{3}$$

$$DR = \frac{dMR}{dt} = -k * a * e^{(-k*t)}$$
(4)

DR (min⁻¹) is the drying rate, MR is the moisture ratio, t is time (min), and *a* and *k* are constants. A graph of DR vs. MR was used to determine the drying behavior.

2.6. Analysis Of Mechanical Properties

The mechanical properties of the natural fresh and dried blueberries were evaluated using a puncture test. Measurements were performed at 25°C in a Texture Analyzer (model TA-XT2, Stable Microsystems, England) at a constant speed of 0.5 mm/s using a cylindrical puncture probe of 3 mm in diameter. All determinations were performed in 15 replicates. The parameters analyzed were: Firmness (N), i.e. the maximum force to break the sample, and Stiffness (N/mm), i.e. the slope of the force-distance curve from the origin of the curve to the breaking point [19].

2.7. Analysis Of The Optical Properties

The optical properties of the natural fresh and dried blueberries were determined using a colorimeter Konica Minolta Chromameter (Model CR 400/410, Japan). The instrument was calibrated with a standard white reflector plate and the system selected was CIE L*

a* b*. This system has been suggested by other authors [20] as the best color space for quantification in food with curved surfaces.

All determinations were carried out in 15 replicates. The results were expressed as L*, a* and hue angle (h). The h was calculated using equation (5):

$$h = \arctan\left(\frac{b^*}{a^*}\right) \tag{5}$$

2.8. Analysis Of The Antioxidant Capacity

2.8.1. Reagents And Standards

ABTS2, 2'-azino-bis (3-ethylbenzothiazoline-6-sulphonic acid), Trolox (6-hydroxy-2, 5, 7, 8-tetramethylchroman-2-carboxylic acid), potassium persulphate and DPPH (2, 2–diphenyl-1-picry-hydrazyl) were purchased from Sigma-Aldrich Corporation, Buenos Aires, Argentina.

2.8.2. Determination With The DPPH Method

The DPPH radical scavenging activity test of blueberries was performed according to the method described by Shimada et al. [21] with some modifications. Measurements were performed with 1 mL of DPPH solution (0.05 g/L in 96% ethanol) added to 0.5 mL of ethanol extracts of blueberries in concentrations of 0.5, 1, 1.5 and 2 mg/mL and reacted at room temperature for 60 min. The absorbance was measured at 515 nm with a Hitachi spectrophotometer model U-1900 (England). The sample mass required to cause 50% DPPH inhibition (EC₅₀) was determined. All determinations were performed in duplicate.

2.8.3. Determination With The ABTS Method

ABTS radical cation scavenging activity was determined according to the method described by Re et al. [22]. ABTS⁺ was produced by mixing ABTS stock solution (7mM) with potassium persulfate (2.45mM). The solution was held at room temperature in darkness for 960 min. Once the radical was formed, the absorbance was adjusted to 0.7 at 734 nm by dilution with 96% ethanol. ABTS⁺ solution was prepared for each analysis. ABTS⁺ (1 mL) was added to 20 μ L of ethanol extracts of blueberries and the reaction mixture was allowed to stand at 25°C for 6 min and the absorbance at 734 nm was immediately recorded. The measure becomes valid when 20-80% of inhibition is obtained, compared to the absorbance of a blank solution prepared with 20 μ L of 96% ethanol and 1000 μ L of ABTS⁺ (without extract). The standard curve was performed using Trolox, with well-known concentrations. All determinations were performed in duplicate.

The results are expressed as the percentage of antioxidant retention and were calculated using the following equation (6):

Antioxidan t Retention (%) =
$$100 - \left(\frac{ACo - ACt}{ACo} * 100\right)$$
 (6)

where ACo is the initial antioxidant capacity and ACt is the antioxidant capacity at the end of the drying process.

2.9. Analysis Of The Rehydration Capacity

Dried blueberries were rehydrated by immersion in distilled water in a 1:10 ratio for 120 min. The rehydration time was selected based on that reported by other authors [7, 23].

The rehydration capacity can be expressed as the weight ratio between the rehydrated sample and the sample before rehydration (g) [**24**]. The rehydration capacity was calculated using the following equation (7):

$$RC = \frac{m_r}{m_s}$$
(7)

where RC is the rehydration capacity, m_s is the dried mass (g) and m_r is the rehydrated mass (g). The mechanical properties of the rehydrated blueberries were analyzed as described in section 2.6.

2.10. Statistical Analysis

The statistical analysis was performed using SYSTAT software version 12. Results are presented as the average of three determinations \pm SD (standard deviation). The results obtained were analyzed using one-way analysis of variance (ANOVA). The process variables and their interactions that presented a p<0.05 indicated significant effects over fruit. The means were compared through a Tukey analysis at 95% level of trust. Significant differences were found when p<0.05.

3. RESULTS AND DISCUSSION

3.1. Comparison Of The Combined Dryings Of Blueberries

Moisture-time diagrams showed different behaviors depending on the conditions applied (Figure 1a). The control drying was performed with HAD because **MWD** -as the only drying- generated a lower yield of undamaged blueberries (data not shown). Although blueberries were pretreated with AEEO to eliminate the wax of skin, the skin was not very porous. When the MW energy is converted into thermal energy within moist

materials, the vapor pressure increases [25]. The skin hinders the exit of the vapor inside the blueberries, leading to the rupture of the fruit structure. On the other hand, OD was not applied as control drying due to it slow efficiency compared to the hot-air drying. This behavior can be seen in Figure 1a, which shows that OD requires more process time to decrease the water content to a determined value.

Regarding the combined processes, neither OD nor HAD decreased the water content in great proportion (only 4%), but allowed keeping intact the superficial structure of the fruit during **MWD**. The cell membrane of blueberries experienced physicochemical changes that made it more flexible, a fact that facilitated the diffusion of the water vapor. The HAD pretreatment (90 min) was 75% more efficient in process time than OD (360 min) to reach the same water content (Figure 1a). On the other hand, the HAD-**MWD** processes had a lower process time (66-73%) than the control drying (Figure 1a). Similar results were obtained by Zielinska et al. [26] in the combined drying of blueberries. These authors observed that the use of microwaves throughout the entire drying process by up to 95 % in comparison with hot air drying. The OD-**MWD** processes and the control drying had similar process times because of the time required in the OD stage (Figure 1a).

The increase in the microwave power allowed decreasing the process time (Figure 1b and 1c). By working at 750W instead of at562.5W, the process time of OD-**MWD** decreased between 9 and 11%, whereas the process time of HAD-**MWD** decreased between 12 and

18%. Similar results were reported by Venkatachalapathy and Kamadenahally [27] in the combined drying of blueberries and **Stanis/awski** [28] in the combined drying of sliced carrots.

When the **microwave output power of** 562.5W was applied, blueberries pretreated with OD took more time to reach the stable condition (aw ≤ 0.6) than the blueberries pretreated with HAD (Figure 1b and c). This behavior might be justified probably because OD favors sugar crystallization in some parts of the outer layers of blueberry tissues, hindering water transfer [29].

When analyzing the kinetics of the variation in water activity (Figure 2), we found that this parameter was unaffected during the pretreatments but quickly decreased when the microwave process was applied. All dried samples reached an aw value in the range of 0.45 to 0.55.

3.2. Drying Rate

The drying rate (DR) demonstrated that the water removal from blueberries depended on the drying method (Figure 3). These results are consistent with those of Therdthai and Zhou [30] in the drying of mint leaves and with those of de Bruijn and Bórquez [31] in the combined drying of strawberries.

At the beginning of **MW** drying, the DR was higher due to the higher moisture content in the blueberries. As the drying advanced, the moisture gradient within the product decreased, hindering the transport of the moisture, and the DR decreased (Figure 3). Alibas [32] observed that as the drying progressed, the loss of moisture in the product caused a decrease in the absorption of microwave power and resulted in a fall in the drying rate. These results suggest that diffusion was the dominant physical mechanism of moisture movement [29].

The DR of the control drying was lower than that of the MWD. Also, the DR increased with the increase in **the microwave output power** (Figure 3), in agreement with that reported by other authors [10, **32**]. This behavior is due to the internal heating generated by MW. The evaporation that occurs inside blueberries creates additional partial pressure and concentration gradients of water vapor [8], which act as additional driving forces to enhance the mass flux of water and thus the effective water diffusion rate during **MWD** [31].

The combined processes were able to reduce the moisture content of blueberries. However, when OD was applied as pretreatment, this combination resulted in a lower DR in the MW stage than that of samples with HAD pretreatment. This could be attributed to the fact that OD decreases the dielectric constant and yields a reduced absorption of microwave energy by the product [33].

3.3. Mechanical Properties

The mechanical puncture tests applied to dried blueberries showed that both firmness and stiffness were significantly affected by the conditions of the treatments (Figure 4a and b).

The control drying promoted both firmness and stiffness. This mechanical behavior is probably due to the lower porosity and the formation of a surface crust as a consequence of the long time required to reach the stable condition (aw <0.6) in the blueberries [34]. Cui et al. [35] reported that the shrinkage and collapse took place during the whole hotair drying, resulting in low transport rate of water and prolonged drying time and increased tough texture.

Regarding the combined processes, the increase in the microwave power decreased the firmness and stiffness of the dried blueberries, independently of the pretreatments. Blueberries dried at 622.5W and 750W had a softer and less firm texture than those of the control drying, whereas blueberries dried at 562.5W had slightly lower mechanical responses than those of the control drying (Figure 4a and b). This is common in **MWD** due to the fast water evaporation, which yields a more open structure and lower shrinkage degree as a result of the vapor expansion within the product [31]. These characteristics may indicate that dried blueberries reached a rubbery state [34]. It is believed that if the texture of the dried blueberries is softer, the quality is better [36].

3.4. Optical Properties

The desired color properties for dried blueberries were values of L*, h and a* near 36, 300° and 0.272, respectively. These values represent a blue-purple coloration on the surface of the fruit.

Before drying, blueberries were dipped into AEEO, which led to an increase in h and a*

(Figure 5a and b). Blueberries treated with AEEO presented a more purple-reddish coloration than natural fresh blueberries (Figure 5a and b). This color change may be due to the addition of NaOH, which generates a pH change that controls the enzyme activities, involving pigment destruction, as reported for browning reactions [37, 38]. Regarding lightness, it presented no significant differences (Figure 5c). This may be due to the dark coloration of blueberries where the light beam emitted by the colorimeter might be reflected in the same way for fresh and treated fruits. Münsell [39] and Moreno and Victor [40] reported that two different colors (as red and blue) can reach same lightness values if we consider the concept as the same degree of clarity and darkness in relation to the same amount of white and black content.

When comparing the different combined processes and the control drying, the ANOVA showed that *h* and a* presented significant differences (p<0.05) between the drying methods (Figure 5a and b). Blueberries pretreated with OD presented a purple-reddish tonality (Figure 5a and b). These results suggest that OD had a protective effect on the color of the dried blueberries in the final process [41, 42]. However, the samples treated with OD-**MWD** maintained the color obtained at the end of the chemical pretreatment (undesired color property).

On the other hand, blueberries dried with HAD-**MWD** and control drying presented *h* and a* values near 300° and 0.3-0.4, respectively (bluer coloration). Similar results were obtained by Yemmireddy [3] in blueberries dried by forced air drying. This behavior may be due to the conversion of the reddish anthocyanins to colorless carbinol base and to the

fact that the remaining bluish brown copigments started to dominate the color of dried blueberries [43].

The lightness of blueberries was not significantly affected (p>0.05) by the drying conditions (Figure 5c). The difference in lightness between the natural fresh blueberries, the blueberries treated with AEEO and the blueberries treated with the different drying methods was very low, indicating that the dried blueberries retained their typical dark color after drying [3]. Besides, through a visual evaluation, we observed that the surface color of the dried blueberries subjected to the different drying methods had no important changes.

3.5. Antioxidant Capacity

As described in the Materials and Methods section, the effect of the different drying treatments on the antioxidant characteristics of blueberries was evaluated by the ABTS and DPPH assays. The results were expressed in terms of percentage of retention of the antioxidant capacity (AC) respect to fresh fruit. Results showed a similar pattern for both assays. All the samples processed presented a significant decrease in AC. The highest antioxidant retention was observed in blueberries dried by the control drying (HAD), reaching 46 and 76% retention for ABTS and DPPH, respectively, whereas the lowest antioxidant retention was observed in blueberries dried by the combined treatments OD-**MWD** 622.5W and 562.5W (Figure 6a and b). Although the control drying was a long process, the temperature was always 60°C, which preserved the antioxidant capacity of dried blueberries [44]. Garau et al. [45] reported that the most appropriate drying

temperature to preserve the AC of products would be around 60°C, which suggests that antioxidant compounds have a higher resistance to heat degradation at this temperature.

In contrast, the combined processes allowed reaching the stable condition (aw< 0.6) in less time than the control drying, but the nutraceutical properties of blueberries were significantly affected (p<0.05). This is because the temperature inside the fruit may increase above the boiling temperature of water, which generates overheating, with the consequent degradation of the antioxidant compounds [25]. The highest antioxidant retention was observed in blueberries treated by HAD-**MWD**-750W. This phenomenon was due to the fact that dried blueberries were less time exposed under these conditions, which generated less degradation of the antioxidant compounds. Similar trends were found by Ghanema et al. [46], who reported that antioxidant compounds were less affected with the increase in the microwave power.

3.6. Rehydration Capacity

The rehydration capacity of dried blueberries was significantly affected by the process conditions (Figure 7a). Dried blueberries presented low rehydration capacity (21%-56%), reaching a water content of 35-37% (-wb-). Similar results were obtained by Nsonzi and Ramaswamy [43] on the rehydration ratios of rabbiteye blueberries dried using osmo-convective dried blueberries or conventionally air-dried (control) blueberries. This behavior may be due to the fact that the skin of blueberries presents high resistance to moisture diffusion [25]. On the other hand, distortion of cells and capillaries in plant

tissue due to the thermal process may lead to textural changes, thus lowering the water absorption characteristics and affecting the rehydration capacity [47].

Microwave drying allowed a higher rehydration of blueberries (20% more) than the control drying, which means that this method generates less thermal damage on the blueberries structure. Similar results were obtained by Drouzas and Schubert [48], who reported that the lower rehydration values evidence the product shrinkage caused by severe heating and/or prolonged drying, which generates irreversible physicochemical changes. These changes seemed to be lower in the microwave finish dried blueberries.

All rehydrated blueberries presented much lower firmness and stiffness than natural fresh blueberries (Figure 7b and c). These results suggest that the loss of tissue integrity and the collapse of the structure of dried blueberries decrease the hydrophilic properties, generating modifications in the firmness and stiffness associated with the thermal damage of the fruit [49, 50].

The rehydrated blueberries obtained with the control drying presented higher firmness and stiffness than those obtained with the combined drying processes. On the other hand, blueberries dried by HAD-**MWD** and OD-**MWD** presented different firmness and stiffness. These results showed that blueberries pretreated with OD were slightly less firm and stiff than those pretreated with HAD. This behavior may be explained by the fact that the sugar uptake during OD minimized shrinkage, thus reducing the compactness of the dried samples [51]. The analysis of the rehydration capacity allowed defining the properties of dried blueberries as products. All samples presented low rehydration capacity but softer texture than natural fresh blueberries. Thus, this product may be used as a food ingredient where high rehydration is not needed, like in cereal bars or ice creams [52].

CONCLUSIONS

The determinations of different parameters of quality allowed selecting the best conditions of blueberry drying. All the drying processes reached the stable condition in dried blueberries (aw < 0.6) within 8 hours of process, being feasible for the food industry. On the other hand, the final product showed good quality characteristics which may be used as food ingredient in products that do not need high rehydration, like cereal bars or ice cream. Based on the results of the drying rate, optical properties, mechanical properties, the retention of antioxidant capacity and rehydration capacity, we conclude that the best drying process to obtain the desired final product was the hot air-microwave drying (750W).

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Figure 1. a) Kinetics of combined processes (OD-**MWD** and HAD-**MWD**) and the control drying (HAD) of blueberries in function of time; b) Kinetic curves of MW drying in blueberries pretreated with OD; c) Kinetic curves of microwave drying in blueberries pretreated with HAD





Figure 2. Kinetic curves the water activity during blueberries drying

Figure 3. Behavior of the drying rates of the combined processes (HAD-MWD and OD-



MWD) and control drying (HAD) of blueberries in function of MR

Figure 4. Mechanical responses a) Firmness and b) Stiffness of dried blueberries obtained at the end of the different combined processes (OD-MWD and HAD-MWD) and control drying (HAD). Bars with different letters indicate significant differences according to a Tuckey test (p<0.05)



Figure 5. Color parameters a) h, b) a* and c) L* of natural fresh blueberries, blueberries treated with alkali emulsion of ethyl oleate (AEEO) and at the end of the combined processes (OD-**MWD** and HAD-**MWD**) and control drying (HAD). Bars with different letters indicate significant differences according to a Tuckey test (p<0.05).



Figure 6. Effect of the different combined drying and the control drying over antioxidant capacity of dried blueberries samples a) Antioxidant Retention (%) by $1/\text{EC}_{50}$ and b) Antioxidant Retention (%) by ABTS. Bars with different letters indicate significant differences according to a Tuckey test (p<0.05) analysis.





