Microscopy analysis of dried edible fruits modified by different physical treatments

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This work shows alternative treatments for dehydrated edible fruits. Drying was carried out under vacuum bylyophilization. Their surface modification was done by electric discharges with the Corona technique and, in one case, a thermal treatment in an inert atmosphere was done. Lyophilization was applied in various fruits, vegetables and edible leafs. Particularly interesting results were found in frozen fruits: strawberry (*Fragaria vesca*); and a couple of leafs: prickly pear (*Opuntia ficus-indica*) and aloe (*Aloe vera*). Interesting results were identified in these samples with the naked eye, since strawberries maintained their structure, characteristic color and odor. Moreover, prickly pear was observed as a foamy fiber with its characteristic green color. The Aloe proved to have one of the most interesting results, since such sample were observed after lyophilization as a highly porous net comprised of whitish fibers with a foamy or sponge-like appearance. Some different treatments were applied to these kinds of samples. Aloe and prickly pear samples were carbonized in a nitrogen atmosphere with the aim of obtaining a carbon net-like electrode. Other aloe prickly pear samples were maintained only as obtained by lyophilization. Also, corona discharges were applied during a selected period of five seconds to other similar samples. All these different kinds of samples were analyzed by SEM in order to further observe their microscopic characteristics. Contact angle and surface energy measurements were conducted.

Keywords: Edible nuts; surface modification; SEM; carbonization; lyophilization

1. Objective

Observing the gross and microscopic changes caused in a number of samples of fruit and vegetables by dehydration treatment action, carbonization in an inert atmosphere as well as corona discharge.

2. Methodology

2.1 Choosing samples

The study was conducted choosing one fruit and two edible leafs, which were selected among others according to their low shrinkage in the vacuum drying process [1]. The fruit was strawberry (*Fragaria vesca*) and the two leafs were prickly pear or nopal (*Opuntia ficus-indica*) and aloe (*Aloe vera*).

2.2 Lyophilization treatment

2.2.1 Freezing

The samples were washed by physical action with an aqueous soap solution at room temperature. The samples were allowed to dry and then were treated separately according to their particular physical characteristics. Firstly, the strawberry was cut into a piece of approximately 3 cm² by selecting a portion, in such a way that the external and the internal areas of the fruit could be observed. Subsequently, it was placed on an acrylic plate and then frozen at −10 °C for 10 hours.

The prickly pear or nopal was washed and then stripped with a knife withdrawing its epidermis and thorns, which are characteristic of this kind of leaf. So, by using this procedure, the inner mucilage and fibrous structure was obtained. Subsequently, square samples measuring around 3 cm² were obtained. They were placed on an acrylic plate and then frozen at −10 °C for 10 hours.

In a correspondingly way, the aloe leaves were cut withdrawing their epidermis and thorns from their edges. So, the inner mucilage was obtained, which was almost completely transparent including most of the inner fibrous structure. Subsequently, square samples measuring around 3 cm² were obtained. They were placed on an acrylic plate and then frozen at −10 °C for 10 hours.
2.2.2 Lyophilization

The three kinds of samples were solidified by the action of the freezing of their water content. They were dried by lyophilization based on sublimation. They were lyophilized for around 24 hours up to a constant weight of each [1-3].

After these drying processes into vacuum, the three kinds of samples were observed as dried solid structures showing interesting features. The first and second cases, strawberry and prickly pear or nopal almost retained their initial exterior volumes and their characteristic red and green colors, respectively. On the other hand, aloe transparency was transformed to a rather whitish fibrous net structure [4-7]. The sponge like appearance was particularly intricate and openly spaced. Their mucilage was considered too dry around the cellulose fibers.

2.3 Sectioning samples

Once the samples were processed by lyophilization, they were cut with a sterile scalpel into smaller pieces according to the specific capabilities of each sample, the treatment processes, and the characterization analyses conducted.

The strawberry samples were observed by SEM both in the inner section as well as around seeds on the outer areas. The prickly pear or nopal samples were of two kinds: one as obtained by drying with lyophilization and a second subjected to carbonization in inert atmosphere (450 °C). The aloe samples were of three kinds: one as obtained by drying with lyophilization, a second subjected to carbonization in inert atmosphere (450 °C), and a third treated with corona discharges for 5 s. The SEM analyzed samples can be observed in Figure 1.

![Fig. 1](image.png)

*Fig. 1* The image shows pieces of treated fruits: 1a) lyophilized strawberry, 1b) lyophilized-carbonized aloe, 2b) lyophilized aloe, 2c) lyophilized-corona treated aloe, 1c) lyophilized-carbonized nopal, 2c) lyophilized nopal.

### 3. Results

#### 3.1 Overall results

As seen in Figure 1, the strawberry samples subjected to lyophilization treatment were observed practically complete and noticeably maintained their volumetric shape and color. Moreover, they intensified their external red color and penetratingly increased their odor.

Aloe samples subjected to lyophilization treatments were transformed to a characteristic lightweight structure. It is similar to a foamy or sponge like with porous cavities therein. Also, testing its structure by carbonizing it under inert atmosphere, it was successfully obtained still in a porous volumetric form, but very brittle to the touch.

Aloe samples were subjected to corona discharge treatments. These tests were conducted in order to improve their rehydration capacity. The corona discharges applied to this kind of samples resulted more suitable to brownish their white color, which was indicative of slight carbonization by thermal action of the discharges. This was attributed to the finely lamellar structure covering the filaments forming the fibrous net. These lamellar layers were associated to the mucilage, which is initially particularly abundant in this kind of samples. Also, such covering layers, originally as a transparent gel, may now disperse light causing a white appearance.

The nopal lyophilized samples resulted in green foamy structures. They were far denser than aloe dried samples, showing thick cellulose fibers. These very soft green forms changed to black lightweight charcoal appearance posterior to annealing at 450 °C. This kind of samples had a particular interest because it resembles the carbon nano foams, which may result useful for other applications, such as, carbon electrodes or activated carbon with high specific area for adsorbing organic and heavy metals pollutants.
3.2 Strawberry

As mentioned previously, strawberry samples were subject to two visualizations, the first of which took place at the inner part of the sample, while the second took place in a zoom of one of the seeds that are so characteristic outside this particular fruit. Figure 2 shows an image of the inner areas of the strawberry sample taken by SEM at 5000X. It resulted interesting to observe some cracks on its right side.

![Figure 2](image1.png) ![Figure 3](image2.png)

Fig. 2  Strawberry inner section.  Fig. 3  Strawberry seed.

As can be seen, the image show particularly flat areas at 5000X. The lyophilization is a process for extracting moisture which does not affect severely the structure of samples for this specific fruit. There was not observed individual detachments or brittle areas, neither big sized cracks nor collapse of the structure.

Figure 3 shows a seed of strawberry, which was taken at 500X. This morphology is characteristic of the strawberry seed without appreciable changes after a lyophilization process. The corona treatment showed no morphological changes that may resulted evident in a SEM image.

![Figure 4](image3.png) ![Figure 5](image4.png)

Fig. 4  Fluorescence image from Confocal Microscopy of the outer red area of a strawberry.  Fig. 5  3D fluorescence image from Confocal Microscopy of the outer red area of a strawberry.

Figure 4 shows a fluorescence image from Confocal Microscopy of the outer red area of a strawberry. The image size is 350 µm each side. Figure 5 shows a 3D representation with around 1.4 mm each side. The images were composed only by fluorescence from the sample, which had two main emissions, one centered at 578 nm and other at 648 nm. There were not observed changes associated to corona treatments other than those of disappearing areas, lacking any emission, were atypically electric arcs burned and carbonized the organic matter.
3.3 Nopal

As mentioned in the methodology, two kinds of samples were used for the specific case of nopal. The first was frozen and lyophilized until achieving a constant weight and eliminate possible high water activity (WA). Meanwhile, other kind of samples besides being lyophilized was carbonized into an inert atmosphere (nitrogen), in order to obtain a specimen of nano foam or electrode characteristics. Thus, one kind of sample was dried and the other additionally was annealed at 450 °C. However, the two kind of samples share similar physically characteristics.

As can be seen, the nopal samples were organic alike strawberry samples with lyophilisation dehydration treatments. However, the perceived outcomes are very different from those seen in the Strawberry (Figure 2) as these can be observed as a series of structured and segmented but interconnected sheets. Meanwhile, as shown in Figure 6, a SEM image at 500X, the lyophilized nopals show microscopic sheets that were sectioned by the possible physical action of dehydration, each of these films have an approximate size around 100 µm. Also, a crack of one of such sheets allowed setting its thickness around 3 µm.

3.4 Aloe

This section shows the microscopic observations of three kinds of aloe leafs; those treated by lyophilization, lyophilization-carbonization, and lyophilization-corona treatment. The microscopic physical changes are shown.
In Figure 8, the SEM image of the lyophilized aloe vera is shown with an increase of 1,000X. The tiny multiple layered conformation was attributed to the dried mucilage covering the fibers. This is a very useful picture because it shows the aloe vera fibers beneath these layers. The fracture shows the thickness of the layers, which were approximately 1 um.

Comparing directly Figures 8 and 9, lyophilized aloe vera leaves with a foam appearance, without and with annealing at 450°C with inert gas atmosphere. The carbonized sample had a very sharp image of the small flat pieces remaining after fracture of the mucilage layers breakage. Meanwhile, the lyophilized Aloe vera without annealing shows large multiple layers that cover the fibers.

Figure 9 shows a SEM image with a 1000X magnification of a carbonized aloe sample. The image is of particular interest because it shows a significant change comparing to the sample of Figure 7, carbonized nopal, because fiber particles are much more identifiable.

Figure 9 shows how the tiny layers of aloe were very obviously cracked. These fractures were of the order of 10 to 25µm. The central area of the figure shows a conglomeration of flat pieces attributed to the mucilage. So, it can be established that the process of carbonization of organic samples of lyophilized cracked the tiny layered structures of mucilage that covers the cellulose fibers. The whitish samples obviously become dark along the process, but not only caused by the carbonization itself but caused by losing the transparent mucilage dried in layers with a thickness that caused light dispersion.

Figure 10 corresponds to an SEM image with a 1000X magnification of a sample of freeze-dried Aloe vera with corona discharge treatment for 5 seconds. As mentioned, this treatment is performed in order to improve wettability and plant products rehydrate process for dehydrated edible fruits. This sample is of particular interest for this research because the fiber did not suffer any transformation as in the case of carbonized sample. It is very similar to the image corresponding to an only lyophilized sample shown in Figure 8. In Figure 10 one can also see a number of particles of about 1 micron, located in the lower right area, which can also be observed to a lower extent in Figure 9.

4. Conclusions

The purpose of this chapter was to observe the macroscopic and microscopic changes caused in samples of fruits and vegetables, such as: strawberry (Fragaria vesca), prickly pear or nopal (Opuntia ficus-indica), and aloe (Aloe vera); who were affected by the action of treatments of dehydration by freeze drying, carbonization in an inert nitrogen atmosphere, and corona discharge.

Each of the samples showed interesting and even unexpected results. First, it was observed the woodiness of the lyophilized strawberries. The dried fruit kept its volume, red color and highly increased its sweet penetrating odor.

On the one hand, it is important to note that nopal samples and the lyophilized – carbonized samples shown no significant changes. In both images, fiber breakage was observed in a similar order of size and number. So, it is assumed that carbonization treatment does not affect seriously the structure of the nopal samples. This kind of samples had a particular interest because it resemble the carbon nano foams, which may result useful for other applications, such as, carbon electrodes or activated carbon with high specific area for adsorbing organic and heavy metals pollutants.

Finally, the lyophilized - carbonized Aloe vera samples were visibly broken compared to only lyophilized sample. The former samples were physically very light and very little stiff, which shows that the action of the carbonization treatment affects severely Aloe vera samples. Moreover, it was observed that the sample with applied corona discharges showed no significant changes other than reduction of contact angles and increase of wettability. This was very interesting as it shows that Corona discharge only superficially affects the sample. These results could be used for further studies that will help research in the application of this technology to facilitate rehydration to dehydrated food products.
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