

## QUALITY CHARACTERISTICS OF DRIED ALOE

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**Abstract:** The effect of drying method on the preservation of aloe's functional substances, as well as total antioxidants and glass transition temperature ( $T_g$ ) of dried aloe were investigated. The differentiations of drying upon the polysaccharides of the gel were studied using <sup>1</sup>H NMR and FT-IR. The concentration of minerals in aloe vera gels were determined by ICP-AES before and after drying. Total antioxidants were measured using DPPH enzyme.  $T_g$ , measured for various water activities using DSC, decreased with water activity increment. Concentration of polysaccharides was affected by the drying process, while minerals concentration remained practically constant.

**Keywords:** Aloe gel; Antioxidant capacity; Drying; Inorganics; Polysaccharides

### INTRODUCTION

*Aloe vera* is a member of the *Liliacea* family and is widely used as a natural treatment and alternatively therapy for various types of diseases. The plant can be separated into two products: aloe latex, a bitter yellow exudate from the pericyclic tubules in the outer skin of the leaves and aloe gel. The gel consists primarily of water (>98%) and polysaccharides (pectin, cellulose, hemicelluloses, glucomannan, acemannan and mannose derivatives). Acemannan is considered the main functional component of *Aloe vera*. There is a controversy about the identity of the active substances in the *Aloe vera* gel and is broadly accepted that there is a synergistic action between the polysaccharides and the other components [Hamman, 2008].

The potential use of *Aloe vera* products often involves some type of processing, such as drying, resulting in powdered samples, which can be further mixed with other ingredients to form dietary, pharmaceuticals and cosmetics products. However, food products are sensitive to drying temperatures, which can induce degradation. Alternatively, although it is more expensive, vacuum drying was tested as a drying method as well as freeze-drying, as a mild technique to provide powdered samples with the least deterioration. The aim of this study was to

evaluate the effect of air, vacuum and freeze-drying in the preservation of some *Aloe*'s functional substances, such as, polysaccharides and minerals.

### MATERIALS & METHODS

Fresh *Aloe vera* leaves obtained from the Peloponnisos district in Greece, were used as the raw materials. The leaves, with length between 30 and 50 cm, corresponded to a 4-year old plant. Whole leaves were washed with distilled water and the epidermis was carefully separated from the parenchyma by a scalpel-shaped knife. The fillets were washed with distilled water to remove the exudates from their surfaces [Femenia et al., 1999], diced to 1cm<sup>3</sup> cubes and dehydrated either in a conventional air drier at 70°C (AD samples) or in a vacuum drier at 60°C (VD samples). In addition, *Aloe* filets were freeze-dried (FD sample), and used as reference samples in NMR and FT-IR analyses. This was necessary, because gel was not dissolved in D<sub>2</sub>O water for NMR analysis and it could not be treated as KBr disk in FT-IR analysis. Gel was collected from the inner part of *Aloe* leaf and was frozen at -30°C for two days. Before freeze-drying, it was treated with liquid N<sub>2</sub> for 1 h in order to lower its temperature.

<sup>1</sup>H NMR spectra at 300 MHz were recorded on a Varian Gemini 2000 spectrometer. Approx. 10 mg of dried *Aloe vera* samples were dissolved in 2 ml of

99.9% deuterium oxide (Euriso-Top, France), and transferred in Schott Economic 5 mm NMR tubes. No internal shift standard was added. Fourier Transformed Infrared (FT-IR) spectra were obtained on a Jasco 4200 spectrometer, after preparing a KBr disc containing 2 mg of FD, AD and VD samples. The single beam traversing each sample was rationed with the single beam of the corresponding background. The inorganic residues of the dried samples and the fresh fillets were determined after heating at 550°C overnight. Then, the residues were dissolved in HNO<sub>3</sub>. Minerals K, Na, Ca, Mg were determined using an atomic absorption spectrometer (Varian, AA 240 FS). Heavy metals like Pb, Cr, Ni, Mn, Fe and Cu were determined by an Inductively Coupled Plasma Atomic Emission Spectrometer (Jobin Yvon, Model Ultratrace JY 138 ). The P content was estimated using the phosphoro-vanadium-molybdenum complex at 466 nm by a portable spectrometer (HACH DR /2010). The antioxidant capacity was measured using DPPH enzyme. Vacuum dried samples were mixed with methanol and DPPH solution, and stored for 30 min in dark conditions, after stirring. The samples were then, measured at 515 nm. %DPPH is proportional to the concentration of antioxidant. The concentration of the antioxidant that causes a 50% decrease in initial concentration of DPPH is defined as EC<sub>50</sub>.

In addition, dried products were stored in a desiccator at room temperature, for three weeks and then equilibrated over saturated salts solutions (LiCl, MgCl<sub>2</sub>, Mg(NO<sub>3</sub>)<sub>2</sub>, NaCl, KNO<sub>3</sub>) of constant water activities, ranging from 0.11 to 0.94, at room temperature for other three weeks. The moisture content of each sample was determined using the oven method at 70°C under vacuum. GAB model was applied to the experimental data:

$$X = \frac{X_m \cdot C \cdot K \cdot \alpha_w}{(1 - K \cdot a_w) \cdot (1 - K \cdot a_w + C \cdot K \cdot a_w)} \quad (1)$$

## RESULTS & DISCUSSION

<sup>1</sup>H NMR has been proved an essential tool to access the quality of *Aloe vera* gel preparations, monitoring the presence of the acemannan. Acemannan is a linear polysaccharide composed by β-(1→4)-linked mannan partially acetylated in positions 2, 3 or 6. In a spectrum, these acetyl groups generate a characteristic signal (2.00 to 2.26 ppm), which can be considered as the fingerprint of *Aloe vera* [Bozzi et al., 2007]. In the <sup>1</sup>H NMR of FD sample (Fig. 1), which is used as a reference sample, a clear signal in the above mentioned ppm range is presented, which shows that during freeze-drying the features of fresh gel, concerning the acemannan, are remained. On the contrary, in AD samples, a remarkable decrease of

the signal of acemannan is observed and this decrease is more intense during the air drying. The absence of signal at 1.33 ppm is a negative indication for the formation of lactic acid, an end product of lactobacillus fermentation, showing that the air and vacuum processes do not induced enzymatic degradation into *Aloe vera* gel.

In Fig. 2, the FT-IR spectra of VD and AD samples revealed remarkable decreases of the bands of 1740 and 1250 cm<sup>-1</sup>, which correspond to the C=O and C-O-C stretches of the acetyl groups, in comparison with FD samples. This is due to a deacetylation of the polysaccharide backbone, during drying process in agreement with literature [Femenia et al., 2003].

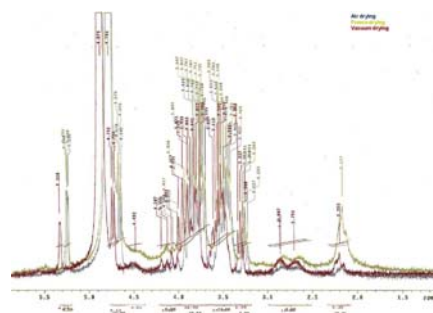


Fig. 1. <sup>1</sup>H NMR spectra of freeze dried, vacuum dried, and air dried samples

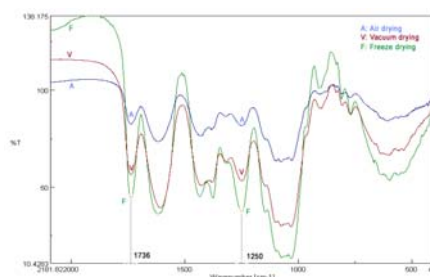


Fig. 2. FT-IR spectra of freeze dried, vacuum dried, and air dried samples

The predominant mineral elements in fresh and dried samples are K, Na, Ca and Mg and secondly Mn. Fe, Cu, Zn was in the range of a few mg/100 g dried material (d.m.), whereas the toxic metals like Pb, Cr, Cd was lower than 1 mg/100 g d.m. in all samples. The level of Ca was high in all samples; this is important for humans bone and tooth development. In combination with other ions such as, Na, K, and Mg provides an ionic balance for the vascular membrane, promoting vasorelaxation and a reduction in blood pressure [Miranda et al., 2009]. The peak values in the fresh gel for the mineral content in K, Na, Ca and Mg reached 1600, 1400, 2370 and 350, respectively. The drying processes lead to small differences into mineral content due to differences in solubility, through hardening process during drying. The P content was at low level unaffected from

drying process. In Table 1 the inorganic residues of various dried samples at 550°C are presented.

In addition, sorption isotherms obtained, follow the characteristic sigmoid profile (type II), which corresponds to multilayer adsorption of *Aloe*. As shown in Fig. 3, equilibrium moisture content increased with the increment of water activity. The knowledge of the equilibrium moisture content of *Aloe* is important for the specification of the storage conditions of the products. The estimated constants of the GAB model are presented in Table 2.

Table 1. Inorganic residues of the dried samples, as well as of fresh fillets at 550°C

Samples	Inorganic Residues (g/100 g dm)
AD	13.07
VD	12.45
FD	11.94
GEL	11.31

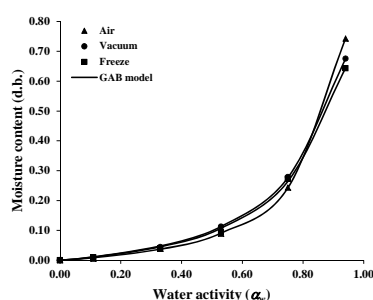


Fig. 3. Correlation of moisture content with water activity of the dried samples

Table 2. Estimated values of GAB constants

Samples	$X_m$	$C$	$K$
AD	0.170	0.435	0.894
VD	0.332	0.332	0.801
FD	0.401	0.261	0.784

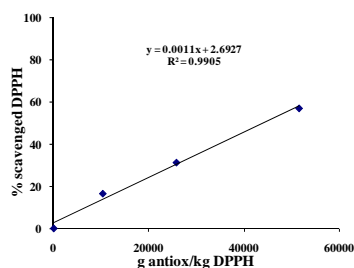


Fig. 4. Antioxidant capacity of vacuum dried *Aloe*.

Fig. 4 illustrates that vacuum dried samples gave satisfactory antioxidant capacity. According to

unpublished results, freeze-dried samples presented the best antioxidant capacity, followed by vacuum and air dried samples.

## CONCLUSIONS

Concentration of polysaccharides was affected from drying process, through deterioration of acemannan polysaccharides as the NMR and FT-IR analyses showed. On the other hand, minerals concentration remained practically constant. *Aloe* gel contains a significant number of organic components and minerals. The preservation of these substances can be achieved by drying of *Aloe* gel. Nevertheless, drying process causes partial distraction of organic molecules and degradation of the produce *Aloe* powder. The antioxidant capacity of dried samples was satisfactory. In addition, equilibrium moisture content increased with the increment of water activity.

## NOMENCLATURE

X moisture content kg water/kg dry solids

C, K constants

Greek letters

$\alpha$  water activity

Subscripts

m adsorbed monolayer

w water

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