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COMPARATIVE STUDY OF SPRAY-DRYING AND FREEZE-DRYING ON THE SOLUBLE COFFEE PROPERTIES

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ABSTRACT. The paper presents a comparative study of some physical properties, microstructure and antioxidant capacity of soluble coffee obtained at laboratory scale using spray-drying (SD) and freeze-drying (FD) as dehydration techniques. SEM was used for monitoring structures and size of the coffee powder. The results of SEM show the difference in the microstructure with the used drying technique. SD dried coffee has only spherical shape, narrow distribution, with the particle mean size of about 10 μm and smooth surface. The FD dried coffee has spherical shape with the size of about 40 μm and flaky structure, non-spherical shape, with the dimensions between 10 x 20 μm and 30 x 90 μm. EPR was used for the determination of coffee antioxidant capacity. SD coffee powder shows a higher antioxidant activity in comparison with FD dried coffee.

Keywords**:** *soluble coffee, spray-drying, freeze-drying, physical properties, microstructure, antioxidant activity.*

INTRODUCTION

Soluble coffee, also called instant coffee or coffee powder, is obtained from freshly ground-roast coffee beans by extraction with hot water at high pressure in order to extract water-soluble compounds. This soluble material is then cooled and sometimes centrifuged, concentrated by heating, and dried to reduce moisture to approximately 5%. Alternatively, steam/water and/or oil may be used to rewet the surface of the instant coffee granules, followed by drying. This process is called agglomeration [1].

Manufacturers use different techniques to improve the appearance and taste of the final product. Ground-roast coffee generally consists of

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Arabica species. Robusta coffee is often used at a high percentage or alone in blends designated for instant coffee production. Robusta seeds contain higher amounts of soluble solids, which increases yield extraction [2].

There are two basic methods available to convert the liquid coffee extract to the dried powder: spray-drying (SD) and freeze-drying (FD) or lyophilisation. By spray-drying the coffee liquid is pulverized into hot air, in spray dryer (atomizer). Through the heat the water evaporates during the downfall of coffee to the bottom of the spray dryer [3]. Freeze-drying is a gentle method used for sensitive products. The concentrated coffee liquid is first frozen and than water changed in ice crystals is removed as vapours by sublimation [4, 5].

Comparing these drying techniques, it is obvious that spray-drying is done at high temperature, affecting some characteristics of final product, but it is less costly and shorter times drying, and so, it allows larger scale economic production [6]. The freeze-drying provides a product with higher quality, overcoming the loss of flavor and aroma, but is energy intensive and expensive due to the low temperature and low-pressure operation [7]. Till now freeze-drying technology is only used at industrial scale to dry coffee, milk, spices, meats and other high-value foods.

In the present work, some physical properties and the antioxidant activity of coffee powder obtained by spray-drying (SD) and freeze-drying (FD) were determined.

Antioxidant activity is an important issue that must be followed during the technological process of producing soluble coffee. According to the literature, coffee is one of the food sources rich in antioxidants. Antioxidants are substances that through their action protect the body against oxidative stress, which unfortunately increase the risk of various diseases of which the most common are cancer, cardiovascular and neurological.

Antioxidant activity of coffee brews is related to chlorogenic, ferulic, caffeic, and *n*-coumaric acids contained in it [8]. Melanoidins (brown pigments) and phenylalanines showing strong antioxidant activity are synthesized during the roasting process when the Maillard reaction is produced [8,10-12].

RESULTS AND DISCUSSION

a. Moisture content of dehydrated materials plays an important role in the handling of soluble coffee powder. The moisture content (% wet basis) determined gravimetric by the mass loss on coffee powder shows higher moisture of freeze dried coffee (7.46 %) than spray dried coffee (4.72%). The lower moisture content of spray dried (SD) coffee, which means better preservation and stability, was expected due to the high temperature used by this technique comparative to freeze-drying process.

b. Microscopic structures, the shape, the surface morphology and the particles size of coffee powder, were examined by **Scanning Electron Microscopy** (SEM). The results of SEM show the difference in the microstructure with the used drying technique (Figures 1 and 2).

In the case of SD technique, the obtained dried particles have only spherical shape, monomodal with a mean size of about 10 μm, and smooth surface. As in other articles atomization seems to promote the formation of particles with narrow distribution and spherical shape [13].

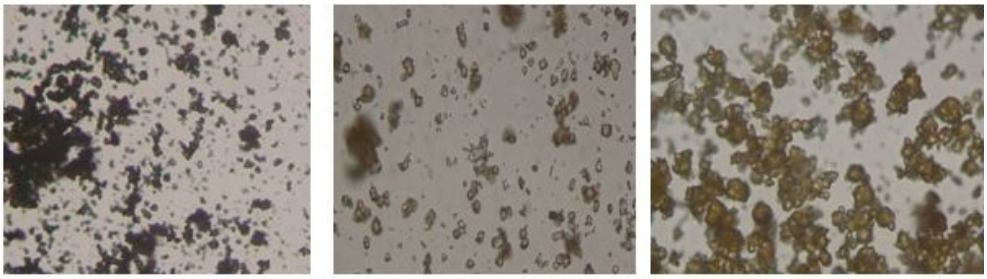
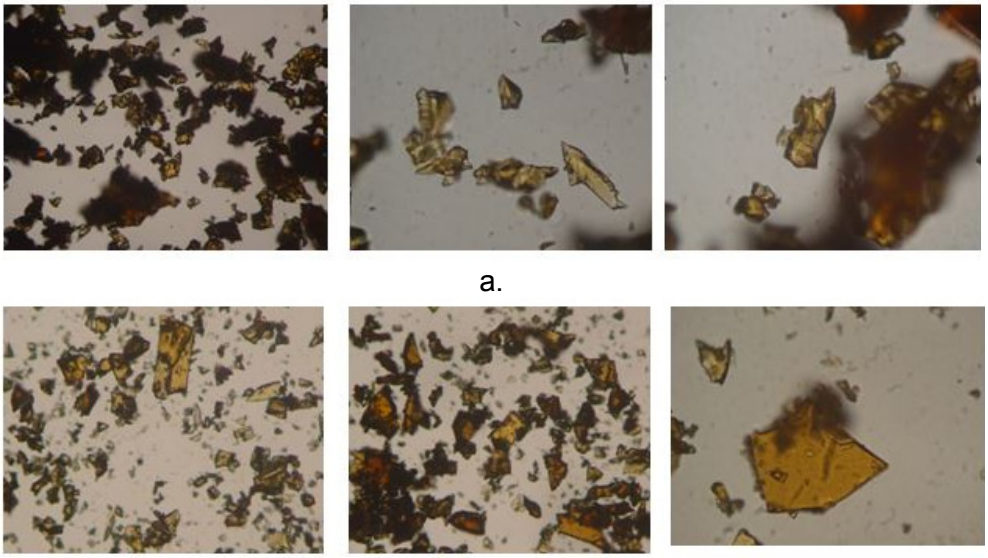


Figure 1. SEM micrographs of spray dried coffee sample



a.

b.

Figure 2. SEM micrographs of freeze dried coffee sample:
a. first experiment, b. second experiment

In the case of powder obtained by FD, the particles show sperical and flaky structure with irregular shapes: sperical shape with the size of about 40 μm and flaky structure, non-spherical shape, with the dimensions of 20 x 50 μm and 30 x 90 μm, for first experiment and spherical particles with diameter of 40 μm, and flattened particles with the dimensions 10 x 20 μm și 20 x 30 μm, for the second experiment [14].

Scanning electron micrographs of freeze dried and spray dried coffee were realized at a magnification of 4x, 10x and 40x. Due to the smoller size of SD coffee particles it is expected an increase of specific surface area and in consequence the increse of solubility comparative with the FD powder, as in other articles has described [15, 16].

c. Bulk porosity

An other property which characterizes the soluble coffee is the bulk porosity. Bulk porosity was calculated by determining the ratio of particle density (ρ_p) and bulk density (ρ_b) using next equation [17]:

$$\varepsilon_b = 1 - \frac{\rho_b}{\rho_p}$$

The particle density (ρ_p) of soluble coffee was determined by pycnometer method. The bulk density of the coffee powder obtained from both drying techniques was measured following the procedure described in literature [16, 17]. The free bulk density of SD dried coffee was 0.324 g/cm^3 comparative to FD dried coffee it was 0.338 g/cm^3. The higher values of FD powder bulk density can be due to its higher residual moisture content, and wider particle size distribution. The obtained values of porositiy soluble coffee were: for SD powder $\varepsilon_{b(SD)}$ = 0.640 and for FD powder $\varepsilon_{b(SF)}$ = 0.624. In our case, the porosity of the SD powder containing smaller particles has a higher porosity than FD dried powder.

d. Antioxidant capacity

Roasting markedly affects the composition of the coffee polyphenols through the Maillard reaction and confers to coffee its pleasant taste and aroma [18]. The coffee beverage is the dietary source of potential antioxidants, such as caffeine, phenolic compounds (mainly chlorogenic acid), hydroxycinnamic acids and Maillard reaction products [19]. The antioxidant capacity of coffee is related to the presence of both natural constituents and compounds formed during its processing [20].

The antioxidant activity of obtained coffee powders was evaluated through their ability to reduce tempol free radical by EPR.

The tempol was used as solution of 0,01% mixed with dried coffee powder obtained by SD and FD techniques.

An overview on the fall time of the soluble coffee antioxidant activity obtained under laboratory conditions is shown in Figure 3: coffee_1 is SD dried coffee, coffee_2 the FD dried coffee (first experiment) and coffee_3 the FD dried coffee (second experiment). The antioxidant activity decreases after 30 minutes to half from the initial activity followed by a slower decline after that. It can be seen for all three samples that the antioxidant activity decreases at a rate of about 60-70% in the first 60 minutes. After that the decrease is slower, reaching zero value in about 400 minutes from the start of the experiment.

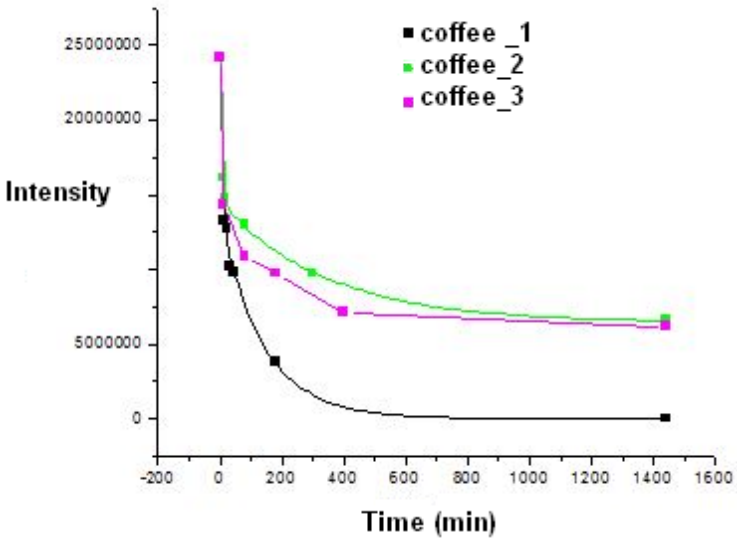


Figure 3. Antioxidant activity vs. time for: coffee_1 - SD dried (black), coffee_2 - FD dried sample 1 (green) , and coffee_3 - FD dried sample 2 (pink)

The results have shown that the FD dried coffee obtained by us has a lower antioxidant activity than SD dried coffee. The lower antioxidant activity of freeze dried coffee can be explained considering the duration of the drying method which is larger for freeze-drying (hours) comparative with the spray-drying (seconds). Similar conclusion was obtained when the antioxidant activity was analyzed as total phenolic content [21].

CONCLUSIONS

The physical properties and the microstructure of soluble coffee were affected by drying methods.

By spray-draying technique it was obtained a product with smooth and spherical shape and large number of smaller particles of about 10 μm diameter.

SD coffee powder has shown smaller free bulk density, higher porosity and higher total antioxidant activity in comparison with FD coffee powder, for the laboratory conditions mentioned in the presented study.

EPR analysis could be a useful method of coffee antioxidant activity evaluation during the drying.

On the basis of physical characteristics (residual moisture, particle size, microscopic structure, bulk porosity) and antioxidant activity we consider spray-drying a good technique which can be used to obtain soluble coffee.

EXPERIMENTAL SECTION

Materials

Coffee solution/liquid was prepared by adding 30 grinded coffee from Firma Tchibo Exclusiv (purchased from local market) to each 100 mL of distillated water. The obtained liquid was heated until it had foamed twice, allowed to settle and than decanted/filtrated. The concentrated solution was used in order to obtain soluble coffee through spray-drying and freeze-drying.

Spray-drying (SD)

SD was carried out in a single stage in the laboratory dryer (Atomizer Mini Spray Dryer BUCHI B-290, Suisse) with co-current drying configuration. Peristaltic pump was used to deliver the feed coffee solution to the atomizer. Ambient air with the flow rate 410±5 L/h was electrical heated. The inlet air temperature was 145 °C. The outlet air temperature was maintained at 100 °C by the adjusted feed flow rate. Product was collected from the outlet chamber and stored in a desiccator at the ambient temperature.

Freeze-drying (FD)

The concentrated coffee solution, with layers of 7 mm placed in Petri dishes, was frozen for 1 hour at -80 0C in the freezer. Than the frozen samples were transferred inside the freeze dryer (ALPHA 1-2 LD_{Plus}, MARTIN CHRIST

Gefriertrocknungsanlagen GmbH, Germany) for a period of 7 hours under the pressure of 0.1 mbar (experiment 1) and 0.045 mbar (experiment 2) and a shelf temperature starting from 20 °C. The condenser temperature was kept to -50 ^{0}C. The obtained freeze dried coffee powder was stored in a desiccator at the ambient temperature.

Moisture content

The moisture content (% wet basis) was analyzed based on the gravimetric determination of the mass loss on drying. 1 g of the coffee sample was placed on the dish and heated at 95 °C for 4 h in the hot air oven. The analysis was performed in duplicates, and the mean value was calculated.

Scanning Electron Microscopy (SEM)

SEM was used to examine the shape, the surface morphology and the particles size of coffee powder samples. The equipment used in our reserch was *Nikon Eclipse E200* from Nikon GmbH Niederlassung Wien, Austria. The used resolution was 40x Binocular.

EPR spectra were recorded with a Bruker EMX spectrometer (Germany), operating in the X-band (9.1GHz – 9.6 GHz) equipped with a computer acquisition system.

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