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APPLICATIONS OF SPRAY-DRYING IN MICROENCAPSULATION OF HESPERIDIN DELIVERED FROM CITRUS PEEL

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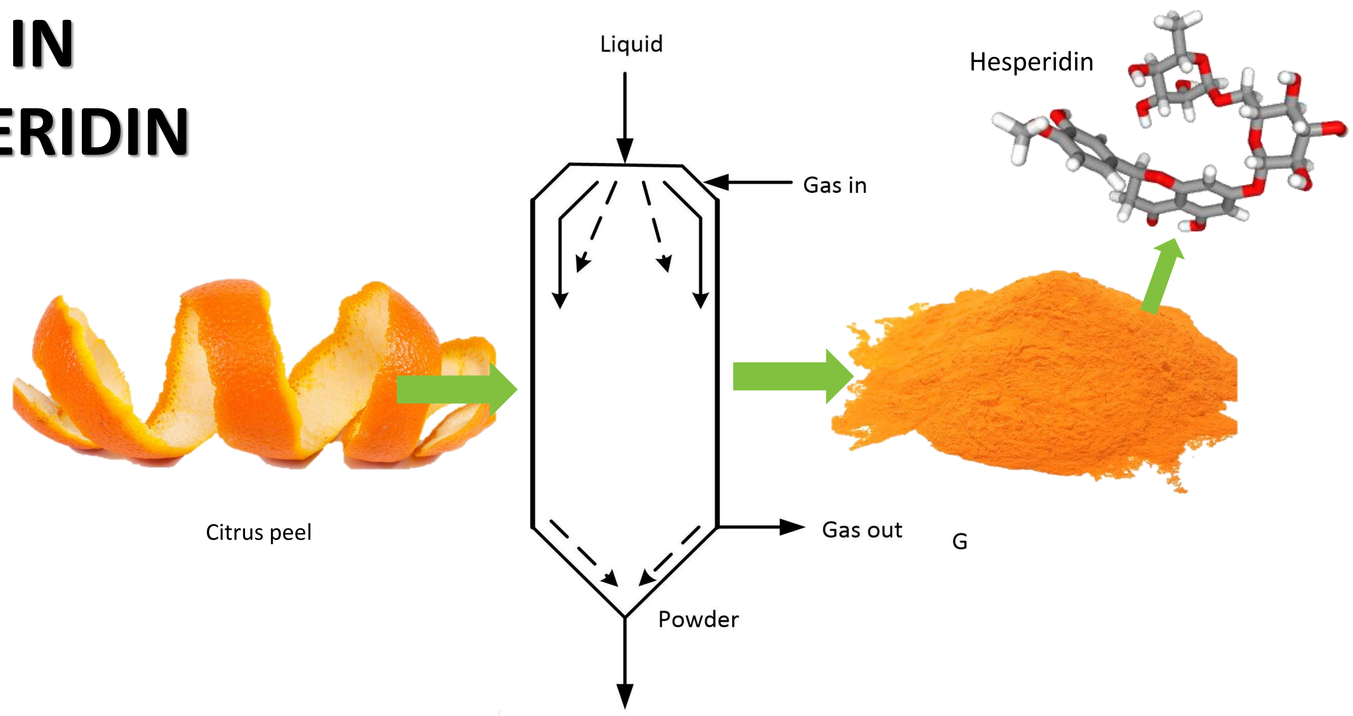

INTRODUCTION

Citrus fruits are one of the most important crops with worldwide production, while citrus by-products represent a problem regarding their disposal due to the environmental risk. The citrus peel contains hesperidin, a flavonoid with a wide range of biological activities. Recent scientific literature discusses the extraordinary versatility of hesperidin, which is reflected in its antioxidant, anti-inflammatory, cardio-protective and antidiabetic properties. However, hesperidin is quite unstable and therefore should be encapsulated to protect its bioactivity from the effects of environmental conditions. Among the other popular microencapsulation techniques, spray drying allows rapid evaporation of water and maintains a relatively low temperature within the particles. The aim of this study was to investigate the possibility of applying spray drying technique for the encapsulation of hesperidin from mandarin peel (*Kuno* variety), formed as by-products during the growth and fresh fruit processing.

METHODS

Samples (whole citrus fruits, satsuma mandarin, Citrus unishu, medium late variety Kuno) were obtained from family farm OPG Pačić. Citrus fruits were grown and harvested in the Metković, Neretva Valley, Croatia in the season 2021/2022. After harvesting, the peel was removed and stored at -80 °C. Before extraction peel was dried, grounded at a laboratory mill and sieved. Citrus peel extracts were produced by ultrasonic-assisted extraction with 70% ethanol as a solvent. Carriers (maltodextrin and Arabic gum) were added to feed in the amount of 100% compared to the dry matter of the extract. The feed flow rate was adjusted to 4 ml/min, the airflow rate was 283 L/h and the temperature of drying was 120 °C. Microcapsules were separated using a high-performance cyclone and collected in the collecting chamber, weighted and stored until further analysis. Determination of hesperidin was performed using high-performance liquid chromatography, and microcapsules were characterized using Powder X-Ray diffraction analysis, Fourier-transform infrared spectroscopy and thermogravimetric analysis.
Encapsulation efficiency was calculated using equation:

$$EE=(THC-SHC)/THC$$

where THC is total hesperidin content and SHC is surface hesperidin content.

RESULTS

Table 1 Encapsulation efficiency of hesperidin microcapsules produced using spray drying

No	Samples	Total hesperidin content [μg ml^{-1}]	Surface hesperidin content [μg ml^{-1}]	Encapsulation efficiency (EE) [%]
1	CPE+MD+SD	461.025	261.332	43.32
2	CPE+GA+SD	433.760	400.940	7.56

CPE – citrus peel extract
MD - maltodextrin, GA - gum Arabic, SD - spray drying

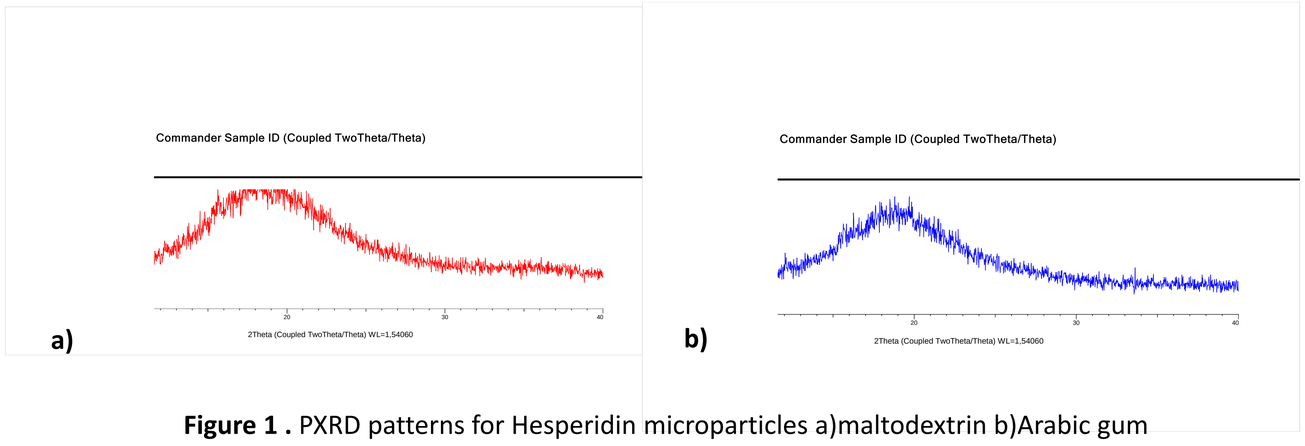

Figure 1 . PXRD patterns for Hesperidin microparticles a)maltodextrin b)Arabic gum

Figure 1 presents PXRD diffractograms of hesperidin microparticles. Few patterns were observed in hesperidin microparticles which showed few peaks with different peak intensities. These findings provide evidence that hesperidin microparticles were lost its crystalline structure during encapsulation processes.

The FTIR spectra of hesperidin as pure compound, hesperidin microparticles were recorded in the range from 400 to 4000 cm^{-1} and compared in **Figure 2.** In hesperidin microcapsules, the peaks existent in frequencies between 2900 cm^{-1} to 3500 cm^{-1} were predominantly found pertaining to hydrogen bonds (O-H stretch), carboxylic acids and residual water. The band around 1604 was assigned to the carbonyl (C=O) stretching in microcapsules with maltodextrin.

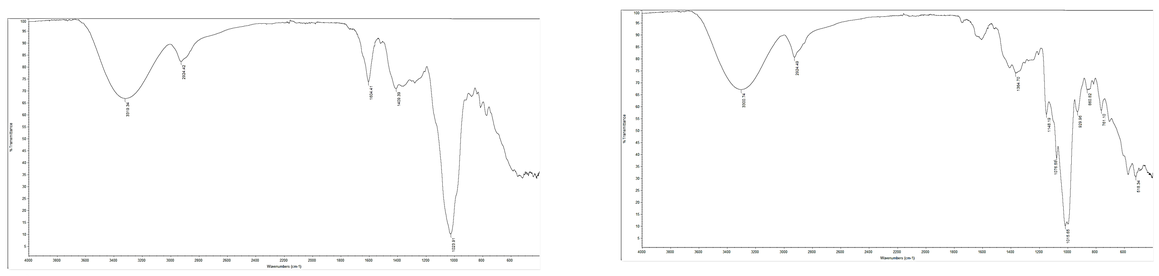

Figure 2. FTIR spectra of Hesperidin microparticles a)maltodextrin b) Arabic gum

TGA data suggest that the thermal degradation of the hesperidin microparticles is a complex process, which occurs in several stages as evidenced by the presence of several peaks in the TGA curve in **Figures 3.** This is likely a consequence of citrus peel complex chemical composition, which is characterized by the presence of several macrocomponents (i.e., pectin, cellulose, hemicelluloses, and lignin) and minor constituents (e.g., proteins, fats, phenolic compounds, etc.) in varying proportions. Hesperidin microcapsules with maltodextrin were thermally stable up to the temperature of about 125 °C while and decomposed in three-stage, while microcapsules with gum Arabic were thermally stable up to the temperature of approximately 115 °C and decomposing in two stages.

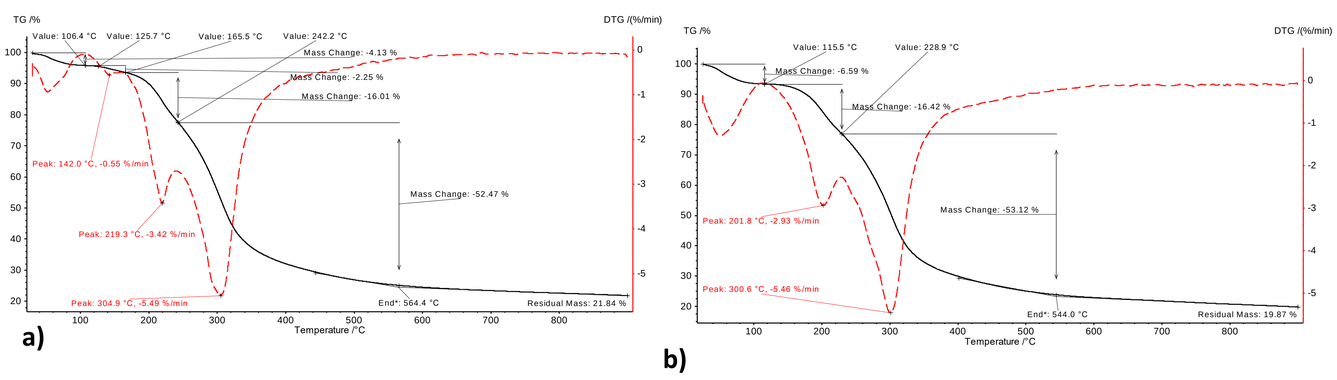

Figure 3. TGA diagrams for Hesperidin microparticles a)maltodextrin b) Arabic gum

CONCLUSION

While dealing with pure flavonoid-hesperidin is more convenient from an analytical standpoint, non-purified phenolic extracts are more commercially viable, saving time, cutting costs, and delivering the largest yield of polyphenols without waste. Citrus peel showed as a possible alternative to commercial hesperidin sources, while spray drying showed as a reliable and effective tool for its encapsulation. The amorphous form of bioactive compounds, such as hesperidin represents the most energetic solid state, which provides the greatest advantage in terms of solubility and bioavailability. The hesperidin retention in the microcapsules was 461.03 and 433.76 mg/g for microcapsules encapsulated with maltodextrin and gum Arabic, respectively. However, higher encapsulation efficiency (difference between surface and total hesperidin content) and highest thermal stability was achieved when maltodextrin was used as an encapsulating agent.

Acknowledgments:

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