

MICROENCAPSULATION OF ELSHOLTZIACILIATAHERB ETHANOLIC EXTRACT BY SPRAY-DRYING: EFFECT OF DIFFERENT ENCAPSULATING AGENTS FOR SPRAY-DRIED MICROCAPSULESQUALITY AND ENCAPSULATION EFFICIENCY OF ROSMARINIC ACID, CHLOROGENIC ACID, AND APIGENIN

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I. INTRODUCTION

Spray-drying is the most popular encapsulation method used for stabilization and protection of biologically active compounds from various environmental conditions, such as oxidation, moisture, pH, and temperature. The aim of this work was to study the effects of different wall materials on encapsulation efficiency (EE) of rosmarinic acid (RA), chlorogenic acid (CA), and apigenin (AP) from *Elsholtziaciliata* herb.

II. MATERIALS AND METHODS

Dried *Elsholtziaciliata* herb („Zolynu namai", Vilnius, Lithuania). Wall material compounds: sodiumcaseinate (SCAS), skim milk (SKIM), maltodextrin (MD), gumarabic (GUM), beta-cyclodextrin (BCYC) were purchased from Sigma-Aldrich, Germany, resistant-maltodextrin (RMD) (Promitor 85™) was purchased from Bang&Bonsomer, Vilnius, Lithuania. All chemical reagents were analytical grade. Powdered material of dried *E. Ciliata* was extracted with 70% (v/v) ethanol in a flask by ultrasound-assisted extraction performed in an ultrasound bath at 25 °C for 30 min. Ethanolic extract and wall material solutions (10-30%) were mixed together. The prepared liquid feeds were spray dried in a Buchi B-291 Mini Spray-Dryer. Spray-drying conditions: temperature was 160 °C, outlet temperature- 80-90 °C, spray flow feed rate- 30 mL/min, air pressure- 6 bar, aspirator- 100%. RA, CA, and AP amounts were obtained using HPLC.

III. RESULTS

Study shows that the RA EE (%) of spray-dried powders varied from 17.69±0.09 to 93.33±0.62% and was the highest when wall material contained 30% RMD and the lowest when it contained 10% RMD (Figure 1). The EE of RA increases with increasing

concentration (10 > 20 > 30%) of wall material in liquid feed solution for spray drying: SKIM 45.98, 67.71, 83.33 %, GUM 24.89, 62.47, 96.36 %, RMD 17.69, 40.56 and 93.33 %, respectively). However, the EE of RA decreases using SCAS (81.33 > 63.48 > 52.71 %) and MD (62.75 > 61.25 > 35.15 %) as coating materials at the same range of concentrations (10, 20 and 30 %). The CA EE of spray-dried powders varied from 4.07±0.52 to 94.46±0.4% and was the highest when wall material contained SCAS (30 %) and the lowest when GUM (30 %) was used (Figure 1). The EE of CA decreases with increasing concentration (10 > 20 > 30%) of wall material in liquid feed solution for spray drying: MD 32.02, 26.81, 11.32 %, GUM 4.93, 4.19 and 4.07%, respectively. The AP EE of spray-dried powders varied from 30.13±0.69 to 99.31±0.64 % and was the highest when wall material contained SKIM (30 %) and the lowest when RMD (10 %) was used (Figure 5). The EE of AP increases with increasing concentration (10 > 20 > 30%) of wall material in liquid feed solution for spray drying: MD 65.08, 90.38, 97.16 %, RMD 12.66, 7.51 and 98.2 %, respectively.

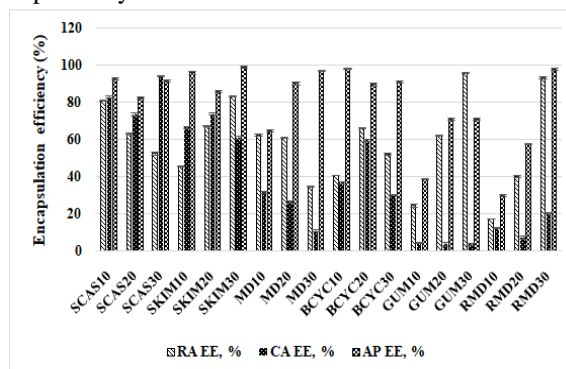


Figure 1. Encapsulation efficiency (%) of RA (rosmarinic acid), CA (chlorogenic acid) and AP (apigenin) obtained in spray-dried powders with different wall materials. SCAS (sodiumcaseinate), SKIM (skim milk), MD (maltodextrin), BCYC (beta-cyclodextrin), GUM (gumarabic), RMD (resistant-maltodextrin). Numbers 10, 20, and 30 means concentration (%) of wall material in liquid feed solution composition for spray-drying.

The microcapsules' morphology was observed by using SEM (Figure 2). Analyzing the micrographs, the most common morphology of micro capsules was semi-spherical. GUM and BETA micro particles showed more folded and dented particles. Diameters of GUM microparticles varied from about 748 nm to 3.88 μm , BETA – from 599 nm to 345 μm (Figure 2). SKIM microparticles were semi-spherical and there were particles that looked compressed. Diameters of these particles varied from about 562 nm to 4.59 μm and more. MD micro particles showed more semi-spherical particles with smooth and wrinkled surfaces in diameters range from about 761 nm to 370 μm and more. In Diameters of SCA microparticles varied from about 1.06 nm to 2.81 μm and most of the particles were deeply wrinkled (Figures 11-12). In addition, the results on Figure 11 show that RMD particles presented a different morphology when compared to particles spray-dried with other wall materials. Diameters of RMD microparticles varied from about 800 nm to 5.62 μm and more (Figure 12). The majority of RMD microparticles had a semi-spherical appearance with smooth and less wrinkled surfaces which was important to confer a better encapsulation of the *E. ciliata* extract. This may be due to the excellent film forming property by the RMD. The water evaporation rates observed in the spray-drying process is most likely the reason of

morphological irregularities that have appeared on the surface of the microparticles.

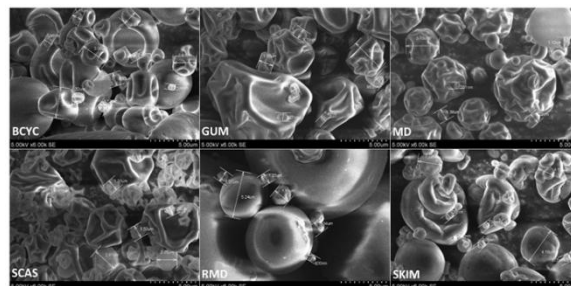


Figure 2. SEM images of micro capsules using 6000x magnifications with measured diameters: GUM (gumarabic), SKIM (skim milk), BCYC (beta-cyclodextrin), MD (maltodextrin), SCAS (sodiumcaseinate), RMD (resistant-maltodextrin). Images were prepared using micro particles which were spray-dried using 20 % of wall materials.

IV. CONCLUSION

According to obtained results, encapsulating materials are suitable for phenolic compounds encapsulation. RMD, SCAS, and SKIM are mostly suitable for RA, CA, and AP encapsulation. Considering these results it is necessary to examine two or more wall materials to obtain the highest EE values of biologically active compounds and microparticles with perfect shape.

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