# OPTIMIZATION OF THE SPRAY DRYING PROCESS OF GRAPE POMACE EXTRACT

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## AIM

The aim of this study was to find optimal spray drying conditions in order to achieve maximum encapsulation efficiency and protection of phenolic compounds from liquid grape pomace extract when 1 % sodium alginate was used as coating material.

## EXPERIMENTAL SETUP

### SAMPLE

- grape pomace Cabernet Sauvignon variety (Erdut winery, Croatia, harvest 2018)
- dry matter content: 92.9 %
- particle size: 1 mm

### EXTRACT PREPARATION

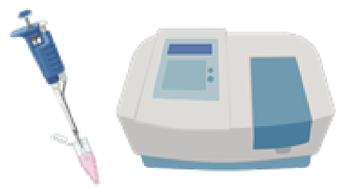
- solvent (50 % ethanol) - sample ratio: 40 mL/g
- extraction by shaking water bath ($T$ = 80 °C, $n$ = 200 rpm, $t$ = 120 min)
- evaporation of ethanol from liquid extract by rotary evaporator ($T$ = 50 °C, $p$ = 48 mbar)

### ENCAPSULATION BY SPRAY DRYING

- 1 % sodium alginate was used as coating material
- Buchi Mini Spray Dryer B-290 was used for spray drying
- experiments were performed according to Box-Behnken design (Response surface methodology – RSM) which was set up using Design Expert software 12; Stat-Ease Inc., USA
- 15 drying experiments were carried out under different process conditions (3 independent variables at 3 levels) as follows:

| Independent variables (Factors) | Variable levels | | |
|---|---|---|---|
| $X_1$ – Air inlet temperature ($T$, °C) | 160 | 180 | 200 |
| $X_2$ – Feed flow rate ($F$, mL/min) | 4 | 6 | 8 |
| $X_3$ – Volume of added extract ($V_{EXT}$, mL) | 50 | 100 | 150 |

### POWDER ANALYSIS



- determination of total phenolic compounds content (TPC) and surface phenolic compounds content (SPC) by spectrophotometric methods at $\lambda$ = 765 nm
- encapsulation efficiency were calculated according to *Eq. 1*:

$$\textbf{\textit{EE}, \%} = \frac{c\,(\mathrm{TPC}) - c\,(\mathrm{SPC})}{c\,(\mathrm{TPC})} \cdot 100 \qquad (\textbf{\textit{Eq. 1}})$$

- experimental data were fitted with 2nd order polynomial model (*Eq. 2*):

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i \cdot X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{\substack{i=1 \\ i<j}}^{k-1} \sum_{j=2}^{k} \beta_{ij} X_i X_j \qquad (\textbf{\textit{Eq. 2}})$$

where $Y$ is responses (*EE*, % and TPC, mg/$g_{db}$), $\beta_0$, $\beta_j$, $\beta_{ij}$, $\beta_{jj}$ are the regression coefficients for intercept, linear, quadratic and interaction terms, respectively; $X_i$ and $X_j$ are the independent variables (temperature, feed flow rate, extract volume); $k$ is the number of variables.

## RESULTS

***Table 1.*** Regression coefficients of significant terms ($p$-values < 0.05), coefficient of determination ($R^2$) of the predicted models for the response variable

| Term | *EE* (%) | *TPC* (mg/$g_{db}$) |
|---|---|---|
| **Model** | **Q** | **Q** |
| **$\beta_0$ – intercept** | 78.03 | 19.24 |
| $\beta_1$ – temperature | - | - |
| $\beta_2$ - feed flow rate | - | - |
| **$\beta_3$ – extract volume** | - | 11.03 |
| $\beta_1 \beta_2$ | - | - |
| **$\beta_1 \beta_3$** | -10.92 | - |
| $\beta_2 \beta_3$ | - | - |
| $\beta_1^2$ | - | - |
| **$\beta_2^2$** | - | -2.97 |
| $\beta_3^2$ | - | - |
| **$R^2$** | 0.92 | 0.99 |

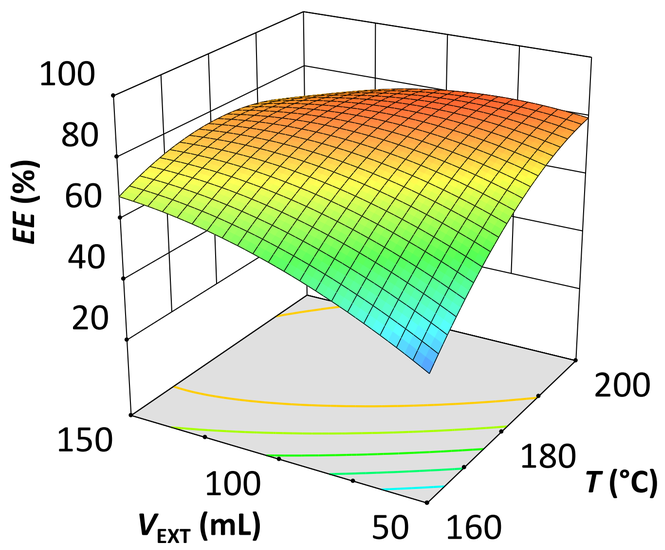



***Figure 1***. 3D graphics surface of encapsulation efficiency (*EE, %*) at fixed feed flow rate (20 mL/min) as a function of air inlet temperature ($T$, °C) and volume of added extract ($V_{EXT}$, mL)

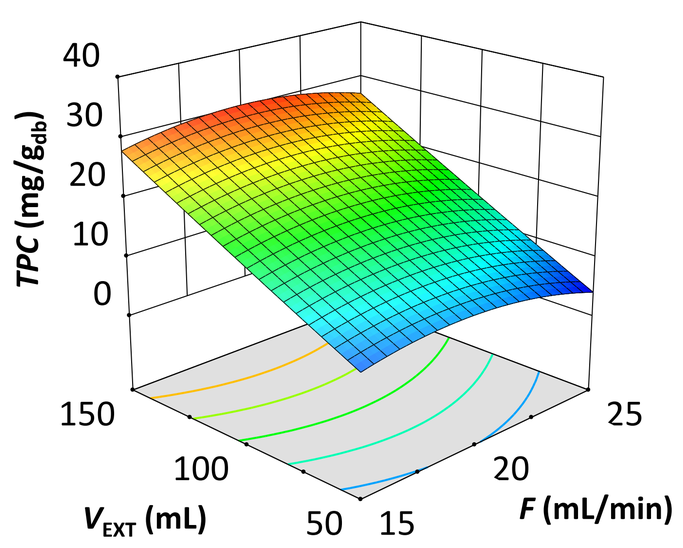



***Figure 2***. 3D graphics surface of total phenolic content in powder (*TPC*) at fixed air inlet temperature (160 °C) as a function of feed flow rate ($F$, mL/min) and volume of added extract ($V_{EXT,}$ mL)

## CONCLUSIONS

- The *EE* were up to 85.59 % and the highest *EE* was obtained when 200 °C, a feed flow rate of 15 mL/min and the addition of 100 mL of extract were used in the drying process (***Figure 1***).
- The *TPC* of the powder extract ranged from 4.18 to 31.11 mg/$g_{db}$ and the highest *TPC* was obtained when 200 °C, a flow rate of 20 mL/min, and the addition of 150 mL of extract in the drying process were used (***Figure 2***).
- The quadratic (Q) model (***Eq. 2***) showed good agreement with the experimental data of *EE* and *TPC* which was confirmed by a high value of the coefficient of determination ($R^2 \geq 0.92$).
- RSM analysis showed that the interaction between the air inlet temperature and the amount of extract added had the greatest effect on *EE*, which was confirmed by the statistically significant interaction term of ***Eq. 2***. *TPC* content was strongly influenced by the feed flow rate and the amount of extract added (***Table 1***).
- With respect to the set criterion of maximum yield of TPC and achieving maximum efficiency, optimal conditions for spray drying were determined such as an air inlet temperature of 180 °C, a feed flow rate of 6 mL/min, and a volume of 150 mL of grape pomace extract. Under optimal conditions, the predicted value for the yield of *TPC* was 30.50 mg/$g_{db}$ and *EE* was 77.16 %.

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