

Valorization of Hibiscus Flower (*Hibiscus sabdariffa* L.) Anthocyanins to Produce Sustainable Spray-Dried Ingredients

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Abstract: The recent increase in sustainability awareness has triggered the industry to establish novel, eco-friendly sources of plant-based ingredients. In the present study, hibiscus flowers (*Hibiscus sabdariffa* L.) were investigated as a sustainable source of anthocyanins for use in spray-dried ingredients with antioxidant capacity. To this end, the extraction and spray-drying microencapsulation of hibiscus flowers anthocyanins were optimized and the final products were evaluated for their oxidative stability index. Initially, the anthocyanins extraction was optimized through a 2² central composite design considering the ethanol concentration (44–56% v/v) in the extraction solution and extraction time (1.6–6.4 h) as independent factors. The optimum extraction

conditions (8 hours, 41.6% v/v ethanol concentration) were used to produce anthocyanin-rich extracts which were microencapsulated by spray drying using a 22 central composite design with the carrier addition rate (1–3% w/v) and inlet temperature (160–192 °C) as factors. Maximum values of solids recovery (60.8%) and anthocyanin retention (96.0%) were reached when 3.2% w/v of starch–alginate carrier blend and a 170.7°C inlet temperature were used. Finally, when hibiscus microcapsules were added to soybean oil, higher oxidative stability was achieved compared to the control. Overall, we demonstrate an industrially friendly and scalable approach that takes advantage of abundant hibiscus flowers as a viable source of anthocyanins for multiple applications.

Keywords: sustainable ingredients; revalorization; novel products; circular economy; bioactivity.

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Tables

Table S1. Analysis of variance (ANOVA) of regression model from full factorial 2² central composite experimental design for the optimization of ethanol concentration (% v/v) and time (h) for the extraction of anthocyanins from HFF.

Factor	df	SS	MS	F-value	p-value
Model	5	1,474E+06	2,949E+05	29,96	0,0001***
A: EtOH concentration (%)	1	4,716E+05	4,716E+05	47,92	0,0002***
B: Time (h)	1	8,831E+05	8,831E+05	89,73	< 0.0001***
AB	1	8649,00	8649,00	0,8788	0,3797
A ²	1	81244,29	81244,29	8,26	0,0239*
B ²	1	13924,47	13924,47	1,41	0,2730
Residual	7	68891,31	9841,62		
Lack of Fit	3	26094,11	8698,04	0,8130	0,5497
Pure Error	4	42797,20	10699,30		
Cor Total	12	1,543E+06			

Statistically significant factors are indicated as follows: * $p < 0.05$, ** $p < 0.01$, *** $p < 0.001$. Legend: HFF – hibiscus-flower flour; EtOH - ethanol; df – degrees of freedom; SS – sum of squares; MS – mean square.

Table S2. Analysis of variance (ANOVA) of regression model from full factorial 2² central composite experimental design for the optimization of carrier ratio (% w/v) and inlet temperature (°C) for solids recovery from the spray drying of anthocyanin-rich extracts from HFF.

Factor	df	SS	MS	F-value	p-value
Model	2	283,90	141,95	5,60	0,0302*
A: Carrier ratio (% w/v)	1	3,60	3,60	0,1420	0,7161
B: Temperature (°C)	1	280,30	280,30	11,05	0,0105*
Residual	8	202,87	25,36		
Lack of Fit	6	191,82	31,97	5,79	0,1546
Pure Error	2	11,05	5,52		
Cor Total	10	486,77			

* indicate statistically significant factors ($p < 0.05$). Legend: HFF – hibiscus-flower flour; df – degrees of freedom; SS – sum of squares; MS – mean square.

Table S3. Analysis of variance (ANOVA) of regression model from full factorial 2² central composite experimental design for the optimization of carrier ratio (% w/v) and inlet temperature (°C) for ANC retention from the spray drying of anthocyanin-rich extracts from HFF.

Factor	df	SS	MS	F-value	p-value
Model	5	108,53	21,71	90,94	< 0.0001*
A: Carrier ratio (% w/v)	1	93,55	93,55	391,94	< 0.0001*
B: Temperature (°C)	1	0,7428	0,7428	3,11	0,1380
AB	1	0,0000	0,0000	0,0000	1.0000
A ²	1	14,13	14,13	59,19	0,0006*
B ²	1	0,5876	0,5876	2,46	0,1775
Residual	5	1,19	0,2387		
Lack of Fit	3	0,8468	0,2823	1,63	0,4023
Pure Error	2	0,3467	0,1733		
Cor Total	10	109,73			

* indicate statistically significant factors ($p < 0.05$). Legend: HFF – hibiscus-flower flour; ANC – anthocyanins; df – degrees of freedom; SS – sum of squares; MS – mean square.

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