Optimization of wall material mixtures for anthocyanins microencapsulation by spray-dry

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INTRODUCTION AND OBJETIVE

Microencapsulation by spray-dry increases the shelf-life of heat sensitive bioactives such as anthocyanins. This technique with suitable wall material transforms bioactives into a stable dry powder form. Mixtures of two or more wall materials are frequently employed to improve the properties of microcapsules (Davidov-Pardo 2011). In the search of new materials for microencapsulation of anthocyanins, the present work deals with the use of Design Expert software to optimize a mixture of four wall materials components (maltodextrin, protein, succinylated starch, acetylated starch) in order to increase the microencapsulation efficiency (ME) and decrease the higroscopicity (H).

MATERIALS AND METHODS

Maize starch and maltodextrin DE 10 (Corn Products, donated by CPI, Mex.); soy protein (Diprosoy, Mex.); acetic anhydride and n-octenyl succinic (Sigma–Aldrich, U.S.A.) were used for modifying maize starches. An anthocyanins extract from purple maize grain (*Zea mays* L.), grown in Tlaxcala, Mexico; acetylated (AS) and succinylated (SS) maize starches from 0.021 and 0.064 degree of substitution, respectively, were prepared as describes Murua-Pagola (2009).

Preparation of microcapsules

Microcapsules were prepared according to the method described by Murua-Pagola (2009), with 170/90°C inlet/outlet air temperature, respectively. A dispersion of 20 g (d.b.) of wall material/100 mL was prepared of water with an anthocyanins content of 1 mg/g.

Encapsulation efficiency and higrocopicity

The microencapsulation efficiency (ME) of anthocyanins was calculated according Robert (2010). Hygroscopicity (H) was expressed as g of adsorbed moisture in a container with KCl saturated solution (84.26% RH) per 100g dry solids (g/100 g).

The IV-optimal design of experimental technique

The option IV-Optimal of the software Design-Expert 8 was employed for designing the experiments in order to study the effect of mixtures on ME and H. Table 1 shows a total number of 20 runs designed by software and their corresponding experimental responses obtained in the laboratory. The boundary restrictions of mixture components were selected based on literature (Fang 2012) and these were employed in the software. Experimental design data were analyzed in order to validate the fitness of the model and its significance and ANOVA (analysis of variance) was performed. The quality of fit of the polynomial equation was expressed with the coefficient of determination (R^2).

The numerical optimization gives the maximum and minimum level of response, Y_1(ME) and Y_2(H) respectively, within the range of factors. In order to optimize the composition we choose the factor goal "within the range" and the response goals "maximize" and "minimize" for the responses Y_1 and Y_2, respectively.

Table 1: The 20 sets of runing conditions determined by the model and their response values.

Run	Components				Responses	
	A	B	C	D	Y_1(EE)	Y_2(H)
1	49.5	49.5	0	1	94.59	17.46
2	36.5	36.5	25	2	98.53	22.50
3	74	0	25	1	97.04	19.55
4	0	75	25	0	94.80	20.07
5	0	98	0	2	97.04	16.95
6	100	0	0	0	94.16	16.66
7	48	0	50	2	98.41	23.17
8	98	0	0	2	96.52	18.26
9	0	73	25	2	95.96	19.67
10	74	0	25	1	94.35	19.54
11	24	24	50	2	98.53	23.12
12	49.5	49.5	0	1	94.35	17.51
13	0	100	0	0	94.45	15.15
14	74	0	25	1	94.59	19.53
15	0	49	50	1	96.52	21.05
16	0	49	50	1	96.52	21.03
17	49.5	49.5	0	1	97.04	17.51
18	50	0	50	0	93.99	21.77
19	25	25	50	0	94.37	18.26
20	37.5	37.5	25	0	94.80	20.07

A: succinylated starch; B: Acetylated starch; C: maltodextrin; D: protein.

RESULTS AND DISCUSSION

Fitting the equation and ANOVA

The effect of different mixtures of wall materials on ME and H are shown in Table 1. Using the Design Expert software, several models were fitted to the results, where ME follows a linear model while H follows and special cubic.

Table 2: ANOVE of encapsulation efficiency (Y_1) and higroscopicity (Y_2).

Source	Ss	df	Ms	F	P > F
Y_1(ME)	ANOVA for linear model				
Linear	31.41	3	10.47	10.85	0.0004
Residual	15.45	16	0.97		
Lack of fit	6.58	11	0.60	0.34	0.9380
Pure error	8.86	5	1.77		
Y_2(H)	ANOVA for special cubic model				
Model	80.49	13	6.19	14978.9	0.0001
Linear	61.19	3	20.40	49344.5	0.0001
AB	2.89	1	2.89	6980.6	0.0001
AC	11.19	1	11.19	27080.8	0.0001
AD	1.41	1	1.41	3418.1	0.0001
BC	13.19	1	13.19	31922.4	0.0001
BD	1.41	1	1.41	3410.0	0.0001
CD	0.41	1	0.41	986.2	0.0001
ABC	0.04	1	0.04	91.9	0.0002
ABD	7.79	1	7.79	18850.9	0.0001
ACD	7.17	1	7.17	17353.8	0.0001
BCD	7.76	1	7.76	18765.1	0.0001
Pure error	0.00	5	0.00		

Ss: sum of squares; Ms: mean square; F values represens the influence of the variable on the response; p-values are the probability of the error. A: succinylated starch; B: Acetylated starch; C: maltodextrin; D: protein.

The final equations obtained in terms of the real components are the following:

ME = 93.65*SS + 94.07*AS + 95.72*MD + 247.36*P

H = 16.66*SS + 15.15*AS -15.98* MD + 8541.25*P+17.84*SS *AS + 85.71*SS*MD - 616.92*SS*P + 67.74*AS*MD - 8608.26*AS*P - 4917.58*MD*P + 7.33*SS*AS*MD + 2436.79*SS*AS*P - 7570.05*SS*MD*P - 5565.66*AS*MD*P

F-values obtained for the responses Y_1 and Y_2 were 10.85 and 14978.9, respectively, which are significant for the models. The p-values (probability of error value) 0.0004 (Y_1) and 0.0001 (Y_2) confirm their relevance in the model. The linear model for response Y_1 yields 0.670 for the coefficient of determination (R^2); and 0.609 for the Adjusted-R^2; the value R^2 for the response Y_2, was: 0.999. The values indicate a strong correlation between the observed and the predicted values.

Optimization

The predicted optimal formulation of the mixture is shown in Table 3. In order to evaluate the accuracy of our models, the ME and H were measured experimentally under the optimum condition. The experimental values of ME (97.6%) and H (16.7 g/100g) are in agreement with the predicted values of ME (97.15%) and H (16.31 g/100g).

Table 3:Constraints and results of optimization

Name	Goal	Experimental		Optimize condition
		Lower	Upper	
A	in range	0	100	77.36
B	in range	0	100	0
C	in range	0	50	20.63
D	in range	0	2	2
Y_1(ME)	minimize	93.99	98.53	97.15
Y_2(H)	maximize	15.15	22.50	16.31

A: succinylated starch; B: Acetylated starch; C: maltodextrin; D: protein.

CONCLUSIONS

The ANOVA table demonstrates that both models and their parameters are significant. The models were numerically optimized and the optimum composition of the mixture of wall materials at the maximum microencapsulation efficiency and the minimum higroscopicity were determined. The optimum composition, consisting of 77.36% SS, 0% AA, 20.63% MD, and 2% P, produced a sample with an ME equal to 97.15% and an H equal to16.31 g/100g. The experimental values at the optimum condition were very close to that predicted by the model. Therefore, this optimum mixture can be used as an alternative optimization tool and as a method for optimizing wall materials in microencapsulation of anthocyanins.

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