MODELLING NONI (*MORINDA CITRIFOLIA* L.) PULP SPRAY DRYING USING A CROSSED MIXTURE-PROCESS DESIGN AND FEED OPTIMIZATION

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ABSTRACT
The effects of noni pulp:maltodextrin (MD) ratio and dry matter (DM) of the feed to the spray drier on different bioactive substances and drying quality attributes of the dried product were evaluated by means of a crossed mixture-process design and the response surface methodology was applied. The mixture components in the feed, noni pulp and MD, were in the 80:20 to 90:10 range. The process factor was the feed's DM, at three levels, 10, 15 and 20 g/100g. The experiments took place in a Niro pilot-scale spray dryer. The lowest powder moisture attained by the model, 3.97 %, corresponded to a feed with 15 % DM and 80:20 pulp:MD ratio. When this ratio is increased, reconstituted product browning also increases with respect to the feed. Maximum ascorbic acid and total phenol were 467 mg/100g and 866 mg gallic acid /100g respectively, corresponding to the 90:10 pulp:MD ratio. The feed's DM only had a significant effect on powder moisture.

Key words: Noni pulp, antioxidants, spray drying, crossed mixture-process design.

1. INTRODUCTION

The noni tree (*Morinda citrifolia* L.) belonging to the Rubeaceae family, is native of Polynesia, India and Southeast Asia, but it has spread to other regions, such as Australia, Latin America and the Caribbean (Cribb and Cribb 1975; Elkins 1998; Chan et al. 2006). The noni fruit has been used as food and, for its therapeutic properties, in traditional medicine for over 2000 years (Earle 2001; Chan et al. 2006). Clinical and laboratory research has been carried out lately to determine its possible effectiveness (Dixon et al. 1999; McClatchey 2002). In the last few years the consumption of products of this fruit increased notably in the United States, Japan and Europe. The FAO/WHO combined report (2008) on the food standards programme estimated a noni market of 400 million USD in 2001, while the estimate in 2006 had increased to 2000 million USD, which made it the fastest growing market in the global health products sector. Noni juice has been accepted in the European Union as a novel food and the fruit was evaluated as to safety and approved for human consumption (European Commission 2003; West et al. 2006).

Accordingly, the design and characterization of spray dried noni products, to be used by the food industry as functional ingredients in a range of health products and as novel nutritional supplements is considered interesting. As the high antioxidant capacity of this fruit is given mainly by the presence of phenols and ascorbic acid (Márquez et al. 2008), it is important to evaluate the influence of the characteristics of the spray dryer feed on the content and retention of these bioactive substances in the end product, a dry powder. On the other hand, it is well known that adhesiveness is the main characteristic limiting the usefulness of spray drying of sugar-rich products, such as fruit juices and pulps, because of low yields and operational problems in drying. The problem of adhesiveness in such dehydrated products has been related to its low glass transition temperature (Tg), a specific property of amorphous materials, which not only present difficulties during drying but are also susceptible to collapse during storage. Therefore, a common approach to drying these products has been to add high molecular weight substances, such as starches and maltodextrin (MD), to increase Tg, up to 75% of these solids being sometimes admitted in the dry matter (Bhandari et al. 1993; Roos et al. 1996; Bhandari, et al. 1997a; Bhandari, et al. 1997b; Bhandari & Howes 1999).

It is claimed (Cornell 1990), that the typical strategy for the experimental design of industrial processes has been to optimize the variables separately, using a mixture design for the formulas and a factorial design and response surface methods for the process. However, the interactions among compositional and process variables cannot be revealed by this simplistic approach; hence the importance of using a crossed design combining mixture components with process factors.

The objective of this study was to evaluate the influence of the feed's noni pulp: MD ratio and dry matter on the content of bioactive substances and drying quality attributes of spray dried noni pulp, by means of a D-optimal crossed mixture-process design as well as to optimize the feed's composition.

2. MATERIALS AND METHODS

2.1. Raw material

Ripe noni fruits (their ripeness degree characterized as "soft white") were reduced to a pulp in a pilot scale Shuttevaerweg-60 pulpier-strainer. This pulping procedure (with 80 % yield) eliminates only the seed, so integral use to the fruit is achieved. Some physical-chemical characteristics of the noni pulp thus obtained are: dry matter, 10.03 ± 0.44 %; soluble solids, 9.25 ± 0.55 ºBrix; total dietetic fibre 1.9 %, pectin 0.96 %, pH, 4.06 ± 0.01 and acidity, 0.55 ± 0.01 g/100g malic acid, the major acid component (Márquez et al. 2008; Pino et al. 2009). Maltodextrin used (IMSA Mexico) had a dextrose equivalent (DE) of 9-12% and 5% moisture.

2.2. Modelling

The experiments were carried out using a D-optimal crossed mixture-process design of experiments (Cornell 1990; Montgomery 1991; Anderson and Whitcomb 1996, 1998; Núñez 2000), which combines mixture components with process factors. In this type of design the mixture components are dependent of each other but independent of the process factors. The mixture components were the ratio of noni pulp (P) to maltodextrin (MD) in the formula fed to the dryer, in the 80:20 to 90:10 ranges. For this range the solid pulp: MD ratio varies between 30:70 and 50:50. The process factor studied was the feed's dry matter (DM), at three levels, 10, 15 and 20 g/100g of liquid formula. The design matrix encompassed a total of 11 experiments and 14 experimental runs, with replicas in the experiment with feed consisting of 85% pulp:15% MD and DM of 15 g/100g liquid formula.

It is important to point out that the addition of water to the different P:MD rate of the experimental matrix, constitutes the only way to achieve an essential condition of this kind of design, that the mixture and process variables are independent of each other, it means that feed DM does not depend on or is not determined by P:MD rate, in such a way that for a same P:MD proportion is possible, by adding water, to obtained different feed DM to the dryer.

2.3. Spray drying

The noni pulp was manually mixed with maltodextrin and water according to the formula for each experiment, homogenized in a colloidal mill and spray dried. The experimental runs were made in a pilot-scale Niro Atomizer Minor (Denmark) spray drier, fitted with a centrifugal disk (2×10^4 min^{-1}) under the same operating conditions: feed temperature, 30ºC; inlet air temperature, 150 °C and outlet air temperature, 75-80 °C. Feed flow provided by a positive displacement pump (maximum capacity, 40 L/h) was regulated in order to assure the desired outlet temperature. In each experience 20 kg of liquid formula was dried.

2.4. Chemical – physical and sensory analysis

The following analyses were carried out in noni pulp and in the experimental powder samples: humidity (AOAC 1995); ascorbic acid (NC ISO 6557: 01); non enzymatic browning (Hendel 1950) and total phenols (Kang and Saltveit 2002). Ascorbic acid and total phenol retention was calculated from the content (mg/100 g) of these compounds in the feed's dry matter and in the powdered product. The sensory evaluation of dehydrated samples colour of each experiment was carried out from the browning point of view with trained judges, by means of a categorical test of 5 points, where punctuation 5 corresponded to the pearl typical colour of the noni pulp and punctuation 1 corresponded to a much browned colour (NC-ISO 4121, 2005).

2.5. Statistical processing

The response variables of the design were humidity, browning of the reconstituted powder; increase in the reconstituted powder browning in regard to the feed; content and retention of ascorbic acid and total phenols. The response surface methodology was used to analyze the results (Montgomery 1991), and the noni pulp:maltodextrin ratios and the DM of the drier feed were optimized by the numeric optimization method, setting restrictions to the response variables by means of the DX version 6 programme.

3. RESULTS AND DISCUSSION

For the analysis of results it was kept in mind that the interpretation of the models is carried out by way of the significant terms in the coded models. The models where the process variable studied (the feed's dry matter) do not appear or has no significant influence can be interpreted properly as mixture models. The quadratic and cubic terms in a mixture model are considered relationships of non linear mixtures or combinations, not interactions, because their components cannot vary independently of each other (Núñez, 2000). The mixture models do not have an intercept. Estimates of the response variables by means of the models are performed by the DX programme with all its terms, both significant and not significant.
In the tables 1-4 appear the results of the coded models obtained for the assessed response variables. The quality indicators considered for each model were: significance of the analysis of variance of the regression ($p < 0.05$); non significance of the lack of fit test ($p > 0.05$); values of $R^2 > 0.8$; significance of the coefficients of each term of the models ($p < 0.05$) and standardized residues following a normal distribution with average zero and s = 1 without atypical observations. All the indicators behaved satisfactorily and therefore the models had the quality required for their analysis.

3.1. Analysis for moisture model of dehydrated noni pulp

From the coded crossed model for the moisture of the spray dry noni pulp (Table1), was obtained that it decreases when MD increases with respect to the pulp in the feed and that the feed's DM has no significant influence independently, but is manifest in an interaction with the proportion of MD in the mixture, so that the smallest value of powder moisture estimated by means of the model (3.97 %) is obtained with a feed's DM of 15% and an pulp : MD ratio of 80:20 (Fig. 1 and 2).

Although in general, in the spray dried products a larger feed's DM causes smaller moisture values in the final dry product, these results prove, in first place, that not all the pulps have the same behaviour and a high feed viscosity, due to the increase of the DM, from certain limits, may unfavourably influences on the drying. In second place, this is an example of the importance of the designs applied to spray drying, which take into account the interaction between process variables and mixture components to obtain minimal values of moisture in the dehydrated products.

Table 1: Results for the moisture (g/100 g of powder) model of dehydrated noni pulp

Term of model	Estimate coefficient	Significance
Intercept	8.86	
B	-4.89**	0.001
X	-1.02	0.065
X^2	-0.88	0.281
BX	0.85	0.276
BX^2	4.60**	0.006
Kind of model	Crossed lineal mixture x quadratic process	
Significance of variance analysis	0.0043**	
R^2	0.845	

B: Proportion of maltodextrine respect to noni pulp in the feed
X: DM of the feed
$0 \leq A \leq 1$ $0 \leq B \leq 1$ $A+B=1$ $-1 \leq X \leq 1$
***; **; * Coefficient and model significance to p<0.001; p<0.01; p<0.05, respectively

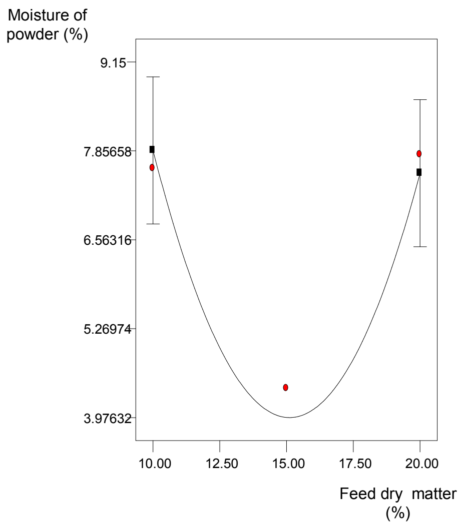

Fig. 1: Influence of feed dry matter on moisture of dehydrated noni pulp to P:MD rate 80:20 in the feed.

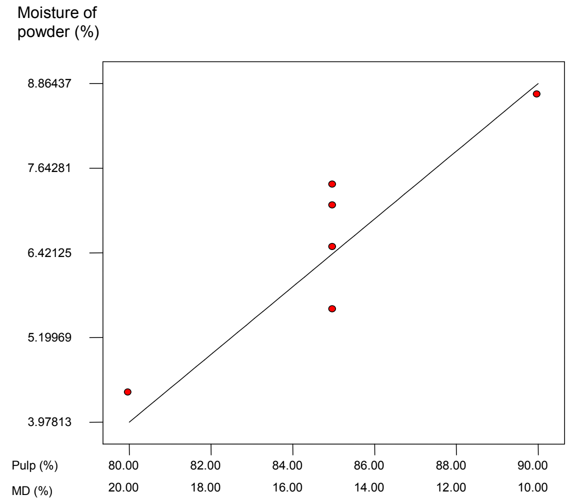

Fig. 2 .Inflluence of P:MD rate of the feed on the moisture of dehydrated noni pulp to a feed DM of 15 %

3.2. Analysis for browning models of dehydrated noni pulp

No browning model was found for the dehydrated noni pulp, but models were found for browning of the reconstituted powder and for the increase in browning of the reconstituted powder with regard to the feed. Browning of the reconstituted powder constitutes a quality indicator for possible applications of the powder, i.e., in reconstituted drinks, while the increase in browning (Table 2) allows the evaluation of the influence of the feed's composition on dried noni pulp browning. In both response variables the feed's DM showed no influence, the MD:pulp ratio did; hence, its interpretation is properly that of cubic mixture models.

Table 2: Results for the model of increase in browning of the reconstituted noni pulp powder respect to the feed.

Term of model	Estimate coefficient	Significance
A	0.064**	0.0038
B	0.031**	0.0038
AB	-0.13*	0.015
AB(A-B)	0.92***	0.0003
Kind of model	Cubic mixture	
Significance of variance analysis	0.0002***	
R^2	0.856	

A: Proportion of noni pulp respect to maltodextrine in the feed
B: Proportion of maltodextrine respect to noni pulp in the feed
$0 \leq A \leq 1$ $0 \leq B \leq 1$ $A+B=1$
***; **; * Coefficient and model significance to p<0.001; p<0.01; p<0.05, respectively

As the maltodextrin of feed mixture increased, browning of the reconstituted powder decreased as well as too its browning with regard to the feed, probably due to a bleaching effect of the MD, that attenuates pulp darkening produced by reducing sugars during drying. In fact, in some experiments negative lightly values were obtained in the increase of browning of powdered samples as compared with the fed formulae, which points out that instead of darkening a little clearing of the pulp takes place during drying.

The results of the sensory evaluation of powder colour were consistent with the analytic results of reconstituted pulp browning, as it was smaller and the punctuation higher in samples with smaller pulp to MD ratio. Nevertheless, none of the powdered samples from the experiments was rejected due to sensorial evaluation of colour.

3.3. Analysis of models for ascorbic acid and total phenols of dehydrated noni pulp

From the nutritional and healthy point of view it is important to evaluate the influence of spray drying conditions on the ascorbic acid content of the noni pulp, given its high concentration in this nutrients (150.94± 11.74 mg/100 g of powder, according to Márquez et al. 2008), similar to that of guava . From the table 3 can be observed for its interpretation that, as expected, a smaller MD:pulp ratio in the feed implies a larger proportion of pulp in the mixture and, therefore, of ascorbic acid in the dried product, so the maximum value estimated by the model was 467 mg/100 g for 90 % pulp and 10% MD.

Table 3: Results of ascorbic acid model (mg/100 g of dehydrated noni pulp)

Term of model	Estimate coefficient	Significance
Intercept	466.94	
B	1930.38*	0.022
X	0.28	0.995
BX	1056.91	0.4043
B^2	-6111.17*	0.0128
B^2X	-1928.61	0.5839
B^3	4035.30*	0.0123
B^3X	743.51	0.7448
Kind of model	Crossed Cubic mixture x lineal process	
Significance of variance analysis	0.0213*	
R^2	0.876	

B: Proportion of maltodextrine respect to noni pulp in the feed

X: DM of the feed

$0 \leq A \leq 1$ $0 \leq B \leq 1$ $A+B=1$ $-1 \leq X \leq 1$

***; **; * Coefficient and model significance to $p<0.001$; $p<0.01$; $p<0.05$, respectively

Although model obtained was crossed: cubic mixture - linear process, as the feed's DM did not have a significant influence on the powder's ascorbic acid content, its interpretation is made like a cubic mixture model.

The noni pulp is also rich in other bioactive substances, as total phenols (181.30 ± 15.84 mg of gallic acid/100 g of powder), (Márquez et al. 2008) and its content in the dried product was favoured with the increase of the pulp:MD ratio in the feed (Table 4), the same performance observed for ascorbic acid, so the highest phenol content of the powder according to the model (866 mg gallic acid/100 g) was obtained with a 90:10 pulp:MD ratio in the feed, and the lowest (552 mg gallic acid/100 g) with a 80:20 ratio (Fig. 3), regardless of the feed's DM, which had no influence on this response variable and a quadratic mixture model was obtained .

Table 4: Results of total phenols model (mg gallic acid/100 g of dehydrated noni pulp)

Term of model	Estimate coefficient	Significance
A	865.98***	0.0001
B	552.58***	0.0001
AB	-319.23*	0.0272
Kind of model	Quadratic mixture	
Significance of variance analysis	0,0001***	
R^2	0,845	

A: Proportion of noni pulp respect to maltodextrine in the feed

B: Proportion of maltodextrine respect to noni pulp in the feed

$0 \leq A \leq 1$ $0 \leq B \leq 1$ $A+B=1$

***; **; * Coefficient and model significance to $p<0.001$; $p<0.01$; $p<0.05$, respectively

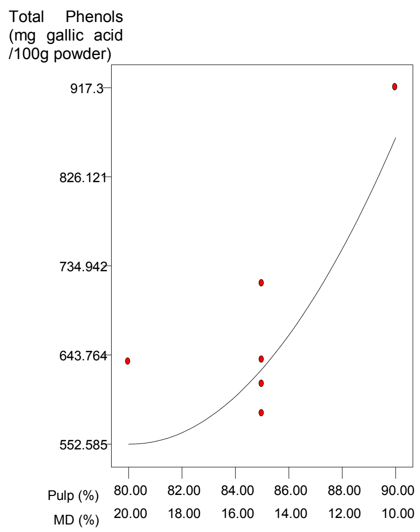

Fig. 3. Influence of P:MD ratio of feed on dehydrated noni pulp total phenols.

The maximum values of both ascorbic acid and total phenols corresponded to the higher purity powdered samples of the experimental design (45.3 – 46.5 g noni solids/100 g of the powdered sample) and the minimum values of these antioxidants corresponded well with the lowest purity samples (26.3-28.7 g noni solids/100 g of the powdered sample). Powder purity mainly depends on noni solids in the DM of the powder (determined by the pulp:MD ratio of the feed) and, in a smaller measure, on the dry powder's DM.

Feed variables did not influence on ascorbic acid and total phenol retention during drying, so that models were not found for them, at least in the range studied, though the average retention values of the experiments were good, 92.26 ± 7.78 % and 98.08 ± 2.70 %, respectively. For both antioxidants the range of experimental retention values varied in the 73 to 100 % range.

3.4. Optimization of the feed

The restrictions imposed on the response variables for dryer feed optimization were: maximization of the ascorbic acid and total phenol contents and minimization of moisture, as well as of the increase in browning of the reconstituted powder respect to the feed. Table 5 shows the two optimum conditions of the feed and the table 6 shows the values of the response variables estimated by the models for these conditions. The first condition is considered the most convenient overly (Núñez, 2000) that is, the one that better satisfies the restrictions of all the response variables.

A new industry, the so-called "well-being or health industry" manufactures products that have a great demand in the market at the moment. The spray dried noni pulp can have multiple uses as functional ingredient for this industry, in the design of new products and nutritional and/or therapeutic supplements, e.g. capsules or pills, and in powdered or liquid food formulas. The dehydrated pulp, by itself, could also constitute a product for preparing reconstituted drinks; in this case, considering the unpleasant noni taste, the addition of some flavouring substances should be contemplated.

Table 5. Optimal feed conditions for spray drying of noni pulp.

Noni pulp (%)	Maltodextrine (%)	Dry matter (%)
84.9	15.1	17.9
80.2	19.8	13.1

Table 6: Estimates of response variables by the models for the optimal conditions of feed

Response variables	1st optimal condition	2nd optimal condition
Powder moisture (%)	6.52	4.67
Increase in reconstituted powder browning respect to feed	0.009	0.011
Ascorbic acid (mg/100g powder)	474.14	330.12
Total phenols (mg gallic acid/100g powder)	625.94	552.60

4. CONCLUSIONS

- It is possible to design powdered, functional and novel noni products with this methodology, using a crossed mixture-process experimental design to optimize the proportion of components of the mixture fed to the drier and the spray drying conditions simultaneously. This can be accomplished by means of the response surface methodology and the application of restrictions to different quality indicators of the dehydrated product.
- When the MD to noni pulp ratio in the spray drier feed is increased from 10 to 20 %, powder moisture decreases. The minimum humidity value in the powder (3.97 %) obtained by the model corresponded to a feed mixture with 15% DM and 80: 20 pulp:MD ratio.
- The maximum values of ascorbic acid and total phenols were 467 mg/100g and 866 mg gallic acid/100g, respectively, and they corresponded to the highest pulp:MD ratio in the design, 90:10, regardless of the feed's DM, which did not significantly influence any of these variables. Models were not obtained for retention of these antioxidants, but they were high (73 to 100%)
- Minimizing or maximizing the response variables, according to the case, two optimum feed conditions were obtained: 84.9:15.1 pulp:MD ratio with 17.9% DM and a 80.2:19.8 pulp: MD ratio with 13.1% DM.

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