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## Data Article

## Data on spray-drying processing to optimize the yield of materials sensitive to heat and moisture content

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## ABSTRACT

Full dataset used to evaluate the spray-drying process parameters on the preparation of a micronized powder made of maltodextrin (MDX) is herein reported. The process parameters (namely, feed flow rate (FFR); inlet temperature ( $T_{in}$ ); nozzle pressure ( $P_N$ ); nozzle diameter ( $D_N$ ) and difference of pressure between cyclone and chamber ( $\Delta P$ )) were screened through a Central Composite Design ( $2^{5-1}$ ;  $2^*5$ ;  $n_C=2$ ) using the following responses: product yield, powder size and size dispersity (span) and the outlet temperature of the exhausted air ( $T_{out}$ ). Data indicate that, in the considered range, only the product yield and the powder median diameter were influenced by the process. The product yield progressively increased on increasing inlet temperature and decreasing the amount or the size of droplets to be dried. The powder median diameter was positively influenced only by the nozzle diameter. This data presented in this article completes a wider work related on "Maltodextrins as drying auxiliary agent for the preparation of easily resuspendable nanoparticles" (Magri et al., 2019).

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Subject area	Pharmacology, Toxicology and Pharmaceutical Science
More specific subject area	Pharmaceutical Science
Type of data	Tables
How data was acquired	Maltodextrin microparticles were prepared by using a Format 4 M8 (ProCepT, B) spray-drier. The size and its distribution were analysed by the single particle optical sensing (SPOS) technique, using an Accusizer 770 (PSS Inc. USA).
Data format	Raw and analyzed data
Experimental factors	Yield, size and size distribution of dried powders; temperature of exhausted air.
Experimental features	The effect of spray-drying process parameters on yield and particle size were determined by Central Composite Design ( $2^{5-1}$ ; $2^*5$ ; $n_c=2$ ) using a 5% maltodextrin DE6 solution.
Data source location	Milan, Italy
Data accessibility	Data are reported in this article
Related research article	G. Magri, S. Franzè, U.M. Musazzi, F. Selmin, F. Cilurzo, Maltodextrins as drying auxiliary agent for the preparation of easily resuspendable nanoparticles. <i>J. Drug Del. Sci. Tech.</i> 50(2019) 181–187. <a href="https://doi.org/10.1016/j.jddst.2019.01.019">https://doi.org/10.1016/j.jddst.2019.01.019</a> [1]

### Value of the data

- The optimized parameters could be used to minimize the thermal stresses which can compromise the physico-chemical properties of sensitive materials.
- The proposed spray-drying conditions could be also exploited to spray-dry solutions containing nanoparticles made of different polymers.
- The optimized conditions can be a reference in the setting of the parameters of a spray-drying process.
- The proposed approach based on the Design of Experiments can be tailored in consideration of the feed composition or the characteristics of the sensitive material being encapsulated.

## 1. Data

The adopted Design of Experiments allowed for screening of the process parameters influencing the yield, size and size distribution (expressed as span) of dried powders other than temperature of exhausted air. Table 1 summarizes the matrix of the experiments and the responses of the 28 runs.

Eq. (1) ( $p=0.014$ ,  $R^2=0.94$ ) and Eq. (2) ( $p=0.0346$ ,  $R^2=0.9$ ) describe the impact of factors on the yield and the median diameter ( $d_{50}$ ), respectively.

$$\begin{aligned} \text{Yield} = & 35.17 - 6.90 \left( \frac{\text{FFR} - 7.5}{2.5} \right) - 4.80 \left( \frac{D_N - 0.8}{0.4} \right) + 4.03 \left( \frac{T_{in} - 155}{25} \right) \\ & + 3.81 \left( \frac{P_N - 1.5}{0.5} \cdot \frac{D_N - 0.8}{0.4} \right) + 3.22 \left( \frac{\text{FFR} - 7.5}{2.5} \cdot \frac{T_{in} - 155}{25} \right) - 3.81 \left( \frac{D_N - 0.8}{0.4} \cdot \frac{\Delta P - 50}{20} \right) \end{aligned} \quad (1)$$

$$\begin{aligned} d_{50} = & 4.95 - 0.59 \left( \frac{\text{FFR} - 7.5}{2.5} \right) + 0.66 \left( \frac{D_N - 0.8}{0.4} \right) - 0.54 \left( \frac{\text{FFR} - 7.5}{2.5} \cdot \frac{D_N - 0.8}{0.4} \right) \\ & - 2.26 \left( \frac{\Delta P - 50}{20} \cdot \frac{\Delta P - 50}{20} \right) \end{aligned} \quad (2)$$

Eq. (1) points out that the yield was negatively influenced by the feed flow rate (FFR) ( $p<0.001$ ) and nozzle diameter ( $D_N$ ) ( $p=0.006$ ); whereas inlet temperature ( $T_{in}$ ) had a positive effect ( $p=0.015$ ). Moreover, the interaction between nozzle pressure ( $P_N$ ) and  $D_N$  ( $p=0.024$ ), FFR and  $T_{in}$  ( $p=0.046$ ), and  $D_N$  and  $\Delta P$  ( $p=0.024$ ) were also significant. In another words, the product yield progressively increases

**Table 1**

Experimental design matrix. The design matrix shows the input parameters set for spray drying [feed flow rate (FFR), inlet temperature ( $T_{in}$ ), nozzle pressure ( $P_N$ ), diameter ( $D_N$ ) and difference of pressure between cyclone and chamber ( $\Delta P$ )] and the output parameters that were experimentally determined [the median diameter ( $d_{50}$ ), powder yield, span, outlet temperature ( $T_{out}$ )].

Exp. no.	Pattern	FFR (mL/min)	$P_N$ (atm)	$D_N$ (mm)	$T_{in}$ (°C)	$\Delta P$ (atm)	$d_{50}$ ( $\mu\text{m}$ )	Yield%	Span	$T_{out}$ (°C)
1	----+	5.0	2.0	0.4	180	30	3.78	31.50	2.9	56.5
2	----+	10.0	1.0	0.4	180	30	3.99	30.75	3.1	49.6
3	++++	5.0	2.0	1.2	180	70	4.15	33.25	3.1	43.8
4	----	5.0	2.0	1.2	130	30	4.69	35.00	3.4	39.7
5	-0000	5.0	1.5	0.8	155	50	6.15	34.00	4.4	57.0
6	----+	5.0	1.0	1.2	130	70	8.05	11.50	5.4	34.0
7	----	10.0	1.0	1.2	130	30	3.30	9.50	2.8	39.6
8	0	7.5	1.5	0.8	155	50	4.69	33.75	1.8	45.7
9	0000+	7.5	1.5	0.8	155	70	2.05	37.00	2.1	39.1
10	----+	5.0	1.0	0.4	180	70	2.74	40.25	1.9	51.2
11	----	10.0	2.0	0.4	130	30	2.21	3.50	1.5	47.2
12	00+00	7.5	1.5	1.2	155	50	5.52	19.25	3.4	38.0
13	++++	10.0	2.0	0.4	180	70	3.22	28.50	2.3	37.2
14	----	5.0	1.0	1.2	180	30	5.83	26.75	6.0	57.9
15	0	7.5	1.5	0.8	155	50	4.21	36.50	3.4	51.9
16	0+000	7.5	2.0	0.8	155	50	5.23	35.50	4.2	52.7
17	++++	10.0	1.0	1.2	180	70	3.22	9.25	2.0	37.0
18	000-0	7.5	1.5	0.8	130	50	4.96	30.75	3.6	40.1
19	0000-	7.5	1.5	0.8	155	30	3.58	36.25	2.3	49.0
20	----+	10.0	1.0	0.4	130	70	3.22	14.75	2.3	33.8
21	----	5.0	1.0	0.4	130	30	3.99	35.00	3.1	49.5
22	000+0	7.5	1.5	0.8	180	50	6.85	33.25	4.7	54.2
23	----+	10.0	2.0	1.2	130	70	2.74	6.75	1.9	35.5
24	0-000	7.5	1.0	0.8	155	50	4.92	30.00	3.8	46.8
25	00-00	7.5	1.5	0.4	155	50	4.45	39.25	3.5	51.0
26	++++-	10.0	2.0	1.2	180	30	4.45	26.75	3.6	56.5
27	+0000	10.0	1.5	0.8	155	50	4.96	34.25	3.6	47.0
28	----+	5.0	2.0	0.4	130	70	2.52	41.00	1.6	37.8

on increasing  $T_{in}$  and decreasing the amount or the size of droplets to be dried. Similarly, FFR is the most critical parameter influencing the span and the outlet temperature ( $T_{out}$ ) ( $p < 0.05$ ). In addition,  $D_N$  improved significantly the span ( $p = 0.033$ ), whereas, as expected,  $T_{in}$  influenced positively ( $p = 0.001$ )  $T_{out}$ .

According to Eq. (2), the microparticles median diameter ( $d_{50}$ ) was positively influenced only by the  $D_N$  ( $p = 0.0094$ ). Moreover, FFR ( $p = 0.0156$ ), the interaction between FFR and  $D_N$  ( $p = 0.0288$ ) and the quadratic term of  $\Delta P$  ( $p = 0.0028$ ) had negative impact on  $d_{50}$ .

## 2. Experimental design, materials and methods

### 2.1. Design of experiment for spray-drying optimization

A Central Composite Design ( $2^{5-1}$ ;  $2^*5$ ;  $n_c=2$ ) was used to optimize the spray-drying conditions in the attempt to dry poly(lactide-co-glycolide) (PLGA) nanoparticles (NP), using maltodextrins (MDX) as novel drying auxiliary agent. Since the weight ratio of NP/MDX was 1/20 [1], it was decided to find the appropriate process input parameter ranges without using NP. Thereby, it was assumed that such a low NP weight would not significantly influence the output parameters.

MDX with a dextrose equivalent (DE) of 6 were kindly gifted by Roquette (F). Ultra-pure water prepared using the MilliQ® system was used to prepare MDX solution. A 5% w/v solution of MDX DE6 was spray-dried using a spray-drier Format 4 M8 (ProCepT, B) equipped by a two-fluid nozzle operating in a co-current manner, namely the sprayed product and the drying air flow are in the same direction. The inlet and outlet temperatures were recorded using PT-100 temperature probes.

**Table 2**

Levels and values of the process factors [feed flow rate (FFR); inlet temperature ( $T_{in}$ ), nozzle pressure ( $P_N$ ) and diameter ( $D_N$ ) and difference of pressure between cyclone and chamber ( $\Delta P$ )] considered in the Central Composite Design.

Level	FFR (mL/min)	$P_N$ (atm)	$D_N$ (mm)	$T_{in}$ ( $^{\circ}$ C)	$\Delta P$ (atm)
-1	5.0	1.0	0.4	130	30
0	7.5	1.5	0.8	155	50
+1	10.0	2.0	1.2	180	70

The factors of the design of experiment included inlet temperature ( $T_{in}$ ), feed flow rate (FFR), nozzle diameter ( $D_N$ ), nozzle pressure ( $P_N$ ) and the difference of pressure between drying chamber and cyclone ( $\Delta P$ ). Before the application of the design, a number of preliminary trials was conducted to determine an experimental space at which the process resulted in a dried powder. The levels of each factor determined by this procedure are reported Table 2. In particular, three levels were taken into account: level -1 was the lowest, level +1 was the highest and level 0 was the central one identified as the mean between the level -1 and +1.

During the optimization of a multivariable process, the responses desirability is combined to produce a product of desired characteristics. In the case of this dataset, the product yield, the span and the outlet temperature were considered the main dependent variables. The desirability goals were defined as following: the process yield should be maximized to avoid waste of material; the span should be minimized to have a narrow monomodal particle size distribution; the outlet temperature should be lowered below 40  $^{\circ}$ C as a product temperature lower than the glass transition of PLGA would avoid the formation of nanoparticles aggregates [1].

The statistical evaluation of the experimental data was carried out by one-way analysis of variance (ANOVA) and lack-of-fit analysis using a commercially available statistical software package (JMP Pro VERSION 13, SAS Institute, USA).

## 2.2. Size distribution of spray-dried MDX

The size distribution of dried powder was analysed by the single particle optical sensing (SPOS) technique, using an Accusizer 770 (PSS Inc. USA). In brief, spray-dried MDX were dispersed in anhydrous methanol and sonicated in a water bath sonicator to assure the complete dispersion of the particles. Afterwards, an aliquot was used for the analysis. The number undersize cumulative distribution data were expressed as dispersion of the size distribution (Span, Eq. (3)).

$$Span = \frac{d_{90} - d_{10}}{d_{50}} \quad (3)$$

where  $d_{10}$ ,  $d_{50}$  and  $d_{90}$  represent the diameters at 10, 50 and 90% of the size number distribution, respectively.

## Transparency document

Transparency document associated with this article can be found in the online version at <https://doi.org/10.1016/j.dib.2019.103792>.

## References

- [1] G. Magri, S. Franzè, U.M. Musazzi, F. Selmin, F. Cilurzo, Maltodextrins as drying auxiliary agent for the preparation of easily resuspendable nanoparticles, *J. Drug Deliv. Sci. Technol.* 50 (2019) 181–187. <https://doi.org/10.1016/j.jddst.2019.01.019>.