

## Instrumentation of a roll compactor and the evaluation of the parameter settings by neural networks

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### Abstract

A Fitzpatrick L83 Chilsonator was instrumented in order to understand and to optimize the roll compaction process using drum-dried waxy maize starch, a plastic deforming material as a model compound. The interrelation of the four adjustable roll compactor parameter settings namely the velocity of the rolls (RS), the speed of the horizontal (HS) and of the vertical screw (VS), and the air pressure ( $P_{\text{air}}$ ) influenced the compact and the granule quality. The granule quality was defined by the friability and particle size distribution. As a second order polynomial was not successful to model the behaviour of the friability in function of the four roll compactor parameters, a Multilayer Feed-Forward neural network (MLF) was used. It was shown that the MLF network models the friability more accurately than a second order polynomial. The HS and the  $P_{\text{air}}$  mostly influenced granule quality and should be kept at a high level. The VS had no significant influence on compact quality. © 1997 Elsevier Science B.V.

**Keywords:** Roll compaction; Dry granulation; Drum-dried waxy maize starch; Neural network; Friability

### 1. Introduction

For many years, the dry granulation process by which powders are slugged on a rotary press or are compacted by a roll compactor, is used for the

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agglomeration of materials sensitive to moisture or heat. Roll compaction is used for the densification of powders for encapsulation, for the formulation of granules for direct compression and the production of directly compressible excipients (Parrott, 1981). The merits of roll compaction to slugging are the continuous production of large quantities of materials at low cost, a better control of the compaction pressure and dwell time and the minimal need for powder lubrication (Falzone et al., 1992). A main disadvantage of roll compaction is the production of a high amount of fines. This problem can be solved by techniques as product recycling (Sheskey et al., 1994) or a vacuum deaeration system (Miller, 1994). Funakoshi et al. (1977) used concavo-convex rimmed shape rollers to obtain less dust production. In this investigation a Fitzpatrick L83 Chilsonator was instrumented in order to better understand the compaction process and to optimise the compaction of drum-dried waxy maize starch used as a model compound. The influence of the four several adjustable compactor parameters namely the velocity of the rolls (RS), the speed of the horizontal (HS) and the vertical (VS) screw and the air pressure ( $P_{\text{air}}$ ) and the interrelation of these parameter settings on the compact and granule quality was studied. A polynomial of second order (QM) and a multilayer feed-forward (MLF) neural network were compared in order to model the results of the compaction process.

## 2. Materials and methods

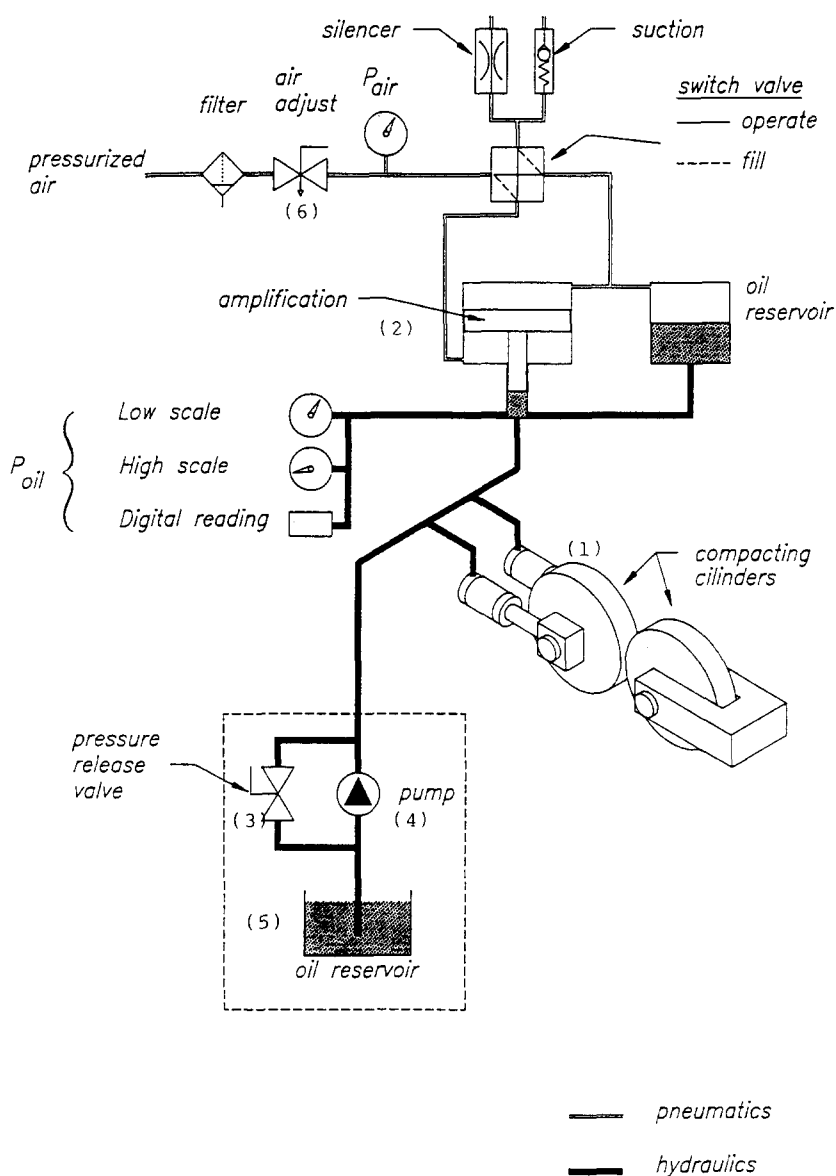
### 2.1. Description and instrumentation of the roll compactor

A Fitzpatrick L83 Chilsonator (The Fitzpatrick Company, Elmhurst, USA) was used. The Chilsonator consisted of two counter rotating rolls, having smooth surfaces. The bearing block of one roll was fixed to the frame. The other roll could move horizontally and was connected to two hydraulic jacks. An air-hydraulic booster system pressurized the hydraulic fluid in the cylinders, resulting in a force on the movable roll. Using this booster system the air pressure ( $P_{\text{air}}$ ) is

converted to a 25-times higher oil pressure ( $P_{\text{oil}}$ ). When the roll moved during compaction, the hydraulic fluid was forced back into the booster resulting in an increase of the pressure in the air part of the booster system. A built in regulator prevented  $P_{\text{air}}$  from exceeding the initial selected  $P_{\text{air}}$  by venting air whenever the air pressure tended to increase. On the other hand, the air pressure could not decrease as the air part of the booster was connected to the pneumatic mains, supplying air whenever necessary.

The powder was brought to the rolls through a feeding system consisting of two screws. A vertical screw, having a deaerating and a predensification function transported the powder to the nip of the rolls. This vertical screw was fed by a horizontal screw fixed in the powder reservoir. When the torque on the vertical screw exceeded its maximum value due to an excess of powder, the motor of the horizontal screw stopped automatically until overfeeding disappeared. Next the motor restarted automatically. This mechanism influenced the feeding and as a consequence the forces acting between the rolls, the horizontal displacement of the movable roll and the compact quality. Hence, the stop of the horizontal feed screw motor was avoided during the experiments.

The four adjustable compactor parameters were the air pressure ( $P_{\text{air}}$ ), the velocity of the rolls (RS) and the velocity of the horizontal (HS) and the vertical (VS) screw. Roll speed ranged from 3 to 13 revs./min, HS from 0 to 60 revs./min and VS from 100 to 1000 revs./min. The  $P_{\text{air}}$  could be adjusted from 0–700 kPa, resulting in a maximum hydraulic pressure and a compaction force of 2500 and 17 500 kPa, respectively. The gap between the rolls and the resulting thickness of the compacts were depending on the setting of these parameters. To measure the instantaneous force between the compacting rolls, a piezo-electric pressure sensor (Greisinger electronic, Regenstauf, Germany) was installed in the hydraulic system. The instantaneous gap between the rolls was measured by means of a displacement transducer (LVDT, Solartron Metrology, Mulheim, Germany), installed on the block containing the movable roll.



A repeatable fixed start situation, which was necessary in order to obtain a reproducible and standardised compaction process, was characterised by the initial distance between the two compaction rolls and by the initial force acting on this rolls. In order to be able to adjust a reproducible start situation, an oil reservoir with a pump system was added to the hydraulic system. For a

chosen air pressure ( $P_{\text{air}}$ ), oil was pumped into the hydraulic system until its pressure reached the maximum value ( $25 \times P_{\text{air}}$ ). This technique excluded the elasticity of the hydraulic part of the system during the built up of the compaction force. It also minimized the initial distance between the two rolls. Fig. 1 shows a schematic representation of the hydraulic system after the

modifications. Every compaction cycle started as follow: the hydraulic system was discharged by opening the pressure release valve (3) and by the simultaneous installation of the air pressure ( $P_{\text{air}}$ ) by an air adjust (6). Oil was flowing into the oil reservoir (5) and the plunger (2) was pressed downwards. Next the pressure release valve (3) was closed and the oil was pumped from the reservoir into the hydraulic system by the pump system (4) until its pressure reached  $25 \times P_{\text{air}}$ . This forced the movable roll (1) to a distance minimal to the fixed roll. Connectors for the simultaneous registration of the velocity of the rolls (RS), the vertical and horizontal screw (VS and HS), the oil pressure ( $P_{\text{oil}}$ ) and the roll distance (D) were installed in order to allow a datalogger (AD conversion) to acquire all measured data.

## 2.2. Granule manufacturing

Drum-dried waxy maize starch (DDWM) (Eridania Béghin-Say Cerestar, Vilvoorde, Belgium), was compacted at various combinations of the four compactor parameter settings: air-pressure ( $P_{\text{air}}$ ), speed of the rolls (RS), speed of the vertical screw (VS) and of the horizontal screw (HS). In order to determine the HS setting limits (shut off of the compactor) in function of VS for different RS and  $P_{\text{air}}$  values, a first experiment consisted of defining the upper and lower limit of the horizontal screw speed for a VS of, respectively, 100, 250, 500 and 1000 revs./min at three RS values (3, 8, 13 revs./min). These limit defining tests occurred at a pressure of 100 and 300 kPa, respectively.

After roll compaction, 400 g compact was milled during 6 min using a Frewitt granulator MG624 (Frewitt, Fribourg, Switzerland) equipped with a 1-mm square sieve and operating at a rotor speed of 130 revs./min. The distance between rotor and sieve was kept minimal.

The quality of the obtained granules was evaluated by sieve analysis and by friability. The sieve analysis was performed on 150 g granules, which were sieved on a sieve-shaker (Retsch, Haan, Germany) for 10 min at an amplitude of 2 mm. The sieves used were 90, 180, 250, 500,

710 and 1000  $\mu\text{m}$ . The amount of granules remaining on each sieve was determined. The friability of the granules was determined by subjecting 10 g of the 250–500  $\mu\text{m}$  fraction together with 200 glass-beads (average diameter of 4 mm) to falling shocks for 10 min in a friabilator (Erweka, Frankfurt am Main, Germany) set at a speed of 25 revs./min. After 10 min, the glass-beads were removed. All remaining material was placed on a 250- $\mu\text{m}$  screen and fixed on a sieve-shaker (Retsch, Haan, Germany) for 2 min at an amplitude of 2 mm. The material remaining on the 250  $\mu\text{m}$  screen was weighed and the percent friability was calculated (Remon and Schwartz, 1987). The reproducibility of the dry granulation process during 6 consecutive days was examined by compacting drum-dried waxy maize starch at two different parameter setting combinations resulting in a good and a poor compact quality. The good quality was obtained by using  $P_{\text{air}} = 300$  kPa, VS = 1000 revs./min, RS = 7 revs./min and HS = 10 revs./min. For the poor quality the combination  $P_{\text{air}} = 100$  kPa, VS = 1000 revs./min, RS = 7 revs./min and HS = 10 revs./min was used. The milling process and the analyses were performed following the procedure described previously.

## 2.3. Statistical analysis

Initial experiments for modelling by a quadratic model (QM) were carried out according to a central composite design. The QM was described by the following (Eq. (1)):

$$Y = b_0 + \sum_{i=1}^n b_i x_i + \sum_{i=1}^n \sum_{j=1}^n b_{ij} x_i x_j + \sum_{i=1}^n b_{ii} x_i^2 \quad (j > i; i, j = 1, \dots, n) \quad (1)$$

where  $Y$  represents the response, e.g. friability,  $b$  the regression coefficients,  $X$  the factors, e.g. the compactor parameters and  $n$  the number of factors ( $n = 4$ ). As a quadratic model seemed to be inadequate additional experiments were performed to investigate the reason of this model failure. In a second approach a neural network (NN) was then used for modelling. The total number of experiments was 80.

### 3. Results and discussion

#### 3.1. Instrumentation of the roll compactor

Essentially, the air-hydraulic booster system of the roll compactor had to provide a constant chosen compaction force between the rolls. However, due to the finite response time of the air-hydraulic booster regulator, small short-time fluctuations of the compaction force were possible. The gap between the two rolls was influenced by feeding rate (horizontal and vertical screw velocity), roll velocity, compaction force and material characteristics of the powder. Due to the interaction of the compaction force and the resulting distance between the rolls during compaction, a simultaneous determination of both parameters was necessary for a better understanding of the compaction process and for an explanation of the differences in compact quality. Installing the oil reservoir with pump system provided the repeatable fixed start situation, necessary to obtain a reproducible and standardised compaction process.

#### 3.2. Granule manufacturing and statistical analysis

The different HS parameter setting limitations (minimal and maximal HS speed) of DDWM were determined for different VS speeds and three different roll speeds (3, 8, 13 revs./min) at two different air pressures (100–300 kPa). The widest range for HS was always obtained at a maximal VS value (1000 revs./min). The increase of the HS range at a VS of 1000 revs./min was more pronounced when the roll speed increased. The results seemed not influenced by a different air pressure adjustment.

The between-days reproducibility of the whole process was evaluated by the friability of the granules and the sieve fraction yield 250–1000  $\mu\text{m}$ . When compacting at a  $P_{\text{air}}$  of 300 kPa the resulting friability was 51.7% with a S.D. of 4.33% and a CV% of 8.45. The sieve fraction 250–1000  $\mu\text{m}$  was 63.6% with a S.D. of 1.8% and a CV% of 2.8. If a  $P_{\text{air}}$  of 100 kPa was used, the resulting friability was 94% with a S.D. of 1.6% and a CV%

of 1.7 and the sieve fraction yield was 30.2% with a S.D. of 1% and a CV% of 3.31.

Table 1 shows all the parameter setting combinations used. The granule quality defined in this investigation by friability and the sieve fraction 250–1000  $\mu\text{m}$  showed a linear relationship described by the following equation  $y = ax + b$  with  $a = -0.636$ ,  $b = 95.31$  and  $r^2 = 0.85$ . Because of this linear relationship between friability and sieve fraction only the influence of the compactor parameters on the friability data were further used and modelled. From Table 1 one can also see that a varying VS did not influence the friability at different HS, RS and  $P_{\text{air}}$  values. Fig. 2 shows the friability (F) values as function of RS and HS. A fraction line could be drawn were the friability changes abruptly. The friability showed a typical sigmoid relationship; the response varied between two limits. Starting from the lower limit, the response first slowly increased, followed by a more rapid increase until it reached nearly the upper limit and it leveled off with an asymptotic increase to the upper limit. When the friability is studied in function of the HS or RS alone, a similar sigmoidal change of the friability is obtained. The effect was less important for the RS than for the HS. The decrease of the friability F due to an increasing  $P_{\text{air}}$  is more pronounced for the lower air pressures. The sigmoid relationship explains why a QM was not adequate. A sigmoid relationship cannot be modelled by a QM. In this case there are several possibilities for data analysis: one can transform the sigmoid relationship to a quadratic one or a straight-line using, e.g. the logistic transformation or one can apply non-linear modelling. It should be understood that non-linear is used here in the statistical sense, i.e. non-linear in the coefficients. In that sense, a polynomial curved relationship is linear. Non-linear regression can be applied when a physical model is available. When this is not the case (no model is known), one can try to apply neural networks. As we have no physical model available, a neural network approach was tried out. Neural Networks (NN) are specialised computer modelling systems based upon simulation of the structure and function of the brain. It performs typically human tasks such as memorising objects,

Table 1

Roll compactor parameter settings with the resulting friability (%) and sieve fraction 250–1000  $\mu\text{m}$  (%) for DDWM

P <sub>air</sub> (kPa)	RS (revs./min)	VS (revs./min)	HS (revs./min)	Friability (%)	Fraction (%)
300	13	100	11	63.5	49.6
		250		72.2	49.9
		500		67.0	45.8
		750		71.4	50.0
		1000		70.4	51.3
300	13	100	29	39.2	68.2
		250		42.7	67.6
		500		41.1	66.6
		750		43.4	68.0
		1000		41.6	69.5
300	7.6	100	9	44.7	67.3
		250		46.0	66.2
		500		48.3	64.3
		750		47.1	60.1
		1000		47.5	67.3
300	3	100	2	65.6	52.6
		250		51.8	60.9
		500		51.6	62.7
		750		48.1	65.2
		1000		44.7	66.1
300	3	100	8	30.4	73.7
		250		29.7	76.0
		500		26.5	75.9
		750		29.7	72.9
		1000		28.0	75.4
200	3	1000	5	47.7	68.2
300				34.4	73.3
350				31.6	72.7
400				28.9	75.8
450				24.3	75.2
500				24.5	76.3
200	3	1000	8	45.4	69.6
300				35.7	75.7
400				31.0	76.3
500				26.8	78.7
200	13	1000	10	88.2	41.4
300				79.4	49.5
400				73.9	58.0
500				66.9	61.6
600				60.6	63.2
200	13	1000	30	79.1	51.9
300				57.5	64.0
400	13	1000	30	44.4	70.5
500				37.1	76.5
600				29.6	76.1
150	7.6	35	9	55.1	67.8
200				46.1	70.5
225				36.1	77.9
300				36.8	78.4
350				32.1	78.7
400				34.2	76.3
500				30.8	80.1

Table 1 (continued)

P <sub>air</sub> (kPa)	RS (revs./min)	VS (revs./min)	HS (revs./min)	Friability (%)	Fraction (%)
300	4	1000	9	42.1	65.7
	5			41.7	66.9
	7			44.3	69.9
	9			58.4	62.6
	11			70.3	50.2
	13			84.5	36.6
300	8	1000	20	42.9	66.8
	9			43.7	69.1
	11			43.2	69.7
	13			43.0	69.7
300	3	50	9	39.7	67.4
	5			38.6	70.7
	7			33.1	83.2
	9			61.7	55.8
	11			70.4	50.7
	13			89.4	25.3
300	13	1000	10	51.0	49.1
			15	42.3	68.3
			20	40.3	66.3
			25	39.6	66.0
			30	39.3	66.3
			5	81.7	45.7
300	7	1000	7	72.5	56.7
			9	44.7	68.3
			12	42.4	70.7
			15	39.7	68.6
			2.5	54.7	57.0
			5	36.1	69.2
300	3	1000	7	36.2	68.0
			8	34.6	67.9

recognising patterns, generalising, estimating parameters and making decisions. Interest in neural network computing has grown rapidly since 1986 in response to the work of Rumelhart and McClelland (1986) and Lippmann (1987). Very few applications of NN in pharmaceutical technology have been published. They have been used for modelling direct compression tableting studies (Türkoglu et al., 1995), for modelling a fluidized bed granulation process (Murtoniemi et al., 1994). NN are also used in pharmaceutical product development (Hussein et al., 1991). The most commonly used network is the multilayer feed-forward network (Wythoff, 1993) with backpropagation learning algorithm and supervised learning. Neural networks are used to model experimental data, using a certain

number of variables. No *a priori* knowledge of the model is needed. Instead of processing successive individual items of data as in regression, the NN processes entire patterns. The flexibility for handling data stems from the specific architecture of the network (Fig. 3) which is composed of processing units called nodes, arranged in layers. Nodes whose output is constant and equal to 1 are called bias. The input layer handles the input to the NN, in this case the four compactor parameters while the output layer produces output, here the estimated friability. Each node of the input layer is connected with the nodes of the hidden layer and these nodes are connected with the nodes of the output layer. A weight factor is associated with each particular interconnection. The input to any

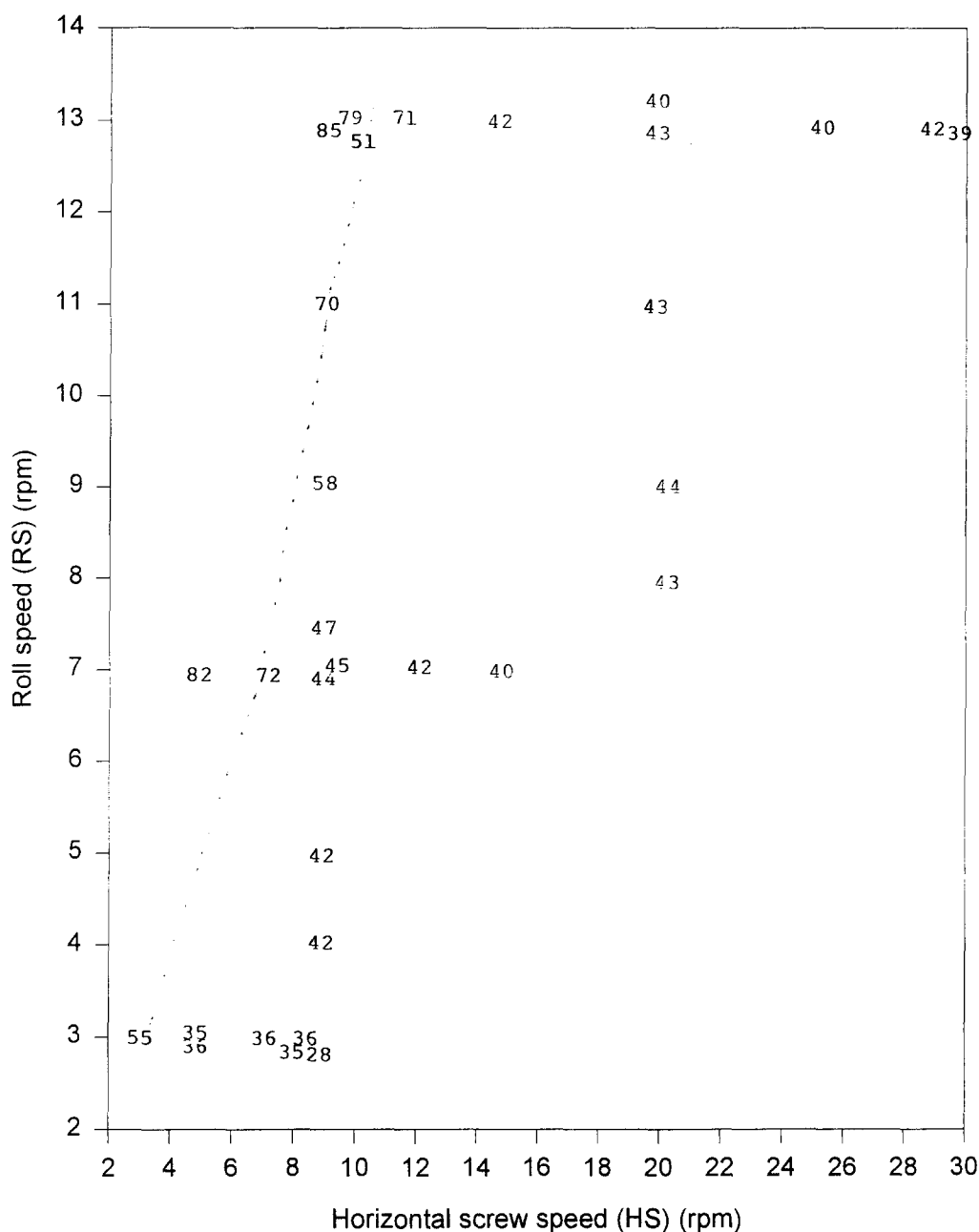


Fig. 2. Friability values in function of roll speed (RS) and horizontal screw speed (HS) with  $P_{\text{air}} = 300$  kPa and  $VS = 1000$  revs./min.

node is the weighted sum of the preceding layer of nodes, including a constant bias term. Nodal output is transformed using a sigmoid transfer function (TF). The inputs to the net are scaled

from 0.1 to 0.9 to ensure that they are in the non-linear part of the sigmoid function. This also prevents domination effects that could result from single large input values.



The initial data set is divided into a larger training set to model the friability and into a smaller test set to validate the model. There are several methods to select both sets. The most important point is that these sets have to be representative. To carry out a representative selection, the selection based on the algorithm of Kennard and Stone (1969) was chosen.

The functioning of a network is highly dependent on the way the signals are propagated through it. This signal propagation is determined by the weights of node-to-node connections. As the weight setting is not known before, the weights are initially given a random value between  $-0.1$  and  $0.1$ . The process of updating the weights in order to minimise the error between experimental and predicted values is called training. An optimal weight set is achieved by means of supervised learning. During training examples consisting of input/output pattern pairs are forced iteratively upon the initially untrained network. Each time an input pattern is presented, the output pattern given by the network is compared to the known output pattern, and the difference is used to adjust the weights in small steps by adding a correction term. The presentation of pattern forms of the training set continues until the network gives an optimally correct answer

(prediction) for each input pattern of the training set. The training is performed applying the backpropagation learning algorithm, based on the  $\delta$ -rule (Rumelhart et al., 1986). The backpropagation learning algorithm tries to locate the minimum error in the weights space, by including a gradient descent approach. The learning rate is an important network parameter, strongly determining the progress of the training procedure. If the rate is too small, the convergence of the weight set to an optimum is very slow and the network might get stuck in a local optimum. If the learning rate is too high, the system might oscillate. To damp possible oscillations, often a momentum term is invoked. The training is an iterative procedure where the maximum number of iterations (max epochs) is used as a stopping criterion. For each iteration the root mean square of errors (RMS) is calculated by Eq. (2):

$$RMS = \sqrt{\frac{\sum (y_p - y_e)^2}{m}} \quad (2)$$

with  $y_e$ , experimental values of the response;  $y_p$ , predicted values;  $m$ , number of experiments of the training set.

During training the RMS of the training set can approach zero by increasing the number of nodes in the hidden layer and training indefinitely the net. This leads to net overfitting and a model that is not robust. Therefore the model obtained after training the net with the training set is evaluated using the root mean square error of prediction (RMSEP) for the test set. The equation used to calculate the RMSEP is similar to Eq. (2) having as only difference the  $m$  value. For the RMSEP the  $m$  value is equal to the number of experiments of the test set. The net with the best predictive ability is the one for which the RMSEP achieves a minimum. At this point the net is considered trained and the weights can be used to reconstruct and predict the response in the whole range of variables.

Optimisation of the number of nodes in the hidden layer and the number of hidden layers is normally done by trial and error. No general approach is available because the problems are data-set dependent. However, there are some em-

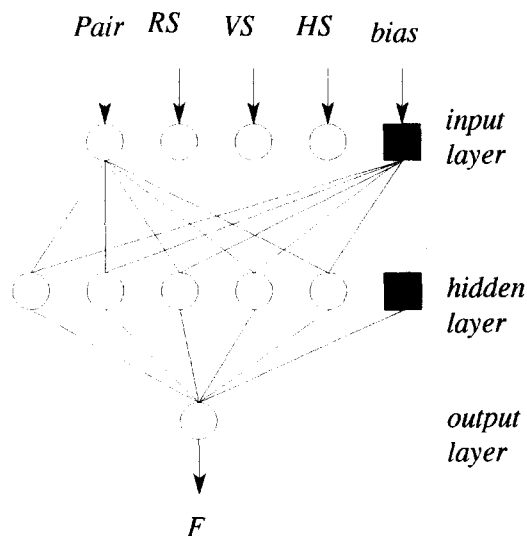


Fig. 3. Architecture of the neural network used in this investigation.

Table 2

Results of the application of neural networks (NN) and a quadratic model (QM) expressed in friability (F) and relative deviation (RD%) for the friability experiments of the test set

Experiment	Experimental F (%)	Predicted F (NN, %)	RD (%)	Predicted F (QM, %)	RD (%)
1	60.7	72.7	16.6	73.8	17.8
2	41.6	49.0	15.1	51.5	19.2
3	32.8	30.9	6.2	30.0	9.3
4	44.7	41.1	8.8	48.9	8.6
5	35.3	35.9	1.7	40.9	13.8
6	45.4	40.4	12.4	37.9	19.9
7	31.0	28.4	9.2	22.8	35.8
8	79.4	79.5	0.2	76.8	3.4
9	42.1	39.8	6.2	32.5	29.4
10	41.7	39.5	5.6	38.1	9.3
11	44.5	43.0	3.4	49.2	9.5
12	58.4	60.6	3.6	59.8	2.3
13	70.3	77.4	9.2	70.0	0.4
14	43.7	43.5	0.5	35.4	23.5
15	43.2	40.7	6.2	45.6	5.3
16	38.6	36.3	6.4	37.7	2.5
17	33.1	38.6	14.2	46.5	28.8
18	70.4	72.8	3.3	63.0	11.7
19	72.5	69.4	4.5	55.9	29.7
20	54.7	46.5	17.5	51.2	6.8
ARD%			7.5		14.2

ARD% stands for average relative deviation in %.

pirical rules described in the literature such as the number of nodes should be such that the number of weights is equal or less than the total number of inputs (Borggaard and Thodberg, 1992). Apart from this, the examples in the literature show that in calibration and probably also in modelling problems such as the one here, one hidden layer is sufficient to model the data. To optimise the number of nodes in the hidden layer, one should start from one and increase the number until acceptable results are reached. It is easy to understand that the larger the number of nodes or hidden layers, the easier it is to model the data. However, increasing the number of nodes or the number of hidden layers means increasing the complexity of the model, leading to overfitting and a non stable model.

The data set contained 80 experiments and 60 were selected with the algorithm of Kennard and Stone (1969) to be the training set. The 20 other experiments were used to test the performance of the QM and the NN model. The net used in this

data set had four inputs (the 4 variable parameters), five nodes in the hidden layer where a sigmoidal transfer function was applied, and one output, the friability (F) with a linear transfer function. The reason for using a sigmoid transfer function in the hidden layer was to be able to model the non-linearities present in the data. The results of the prediction of the F, obtained for the test set with the NN and with a QM are presented in Table 2 and expressed in relative deviations (RD), in %. The average relative deviation (ARD%) calculated for NN is half the one calculated for QM. This clearly shows that, as expected, the NN models the friability better than the QM. In order to see the effect of the non-linearity some response surfaces of F were plotted keeping at each time, two of the four variables constant. The response surface F obtained when setting  $P_{\text{air}}$  at 300 kPa, RS at 13 revs./min and varying the VS and HS is shown in Fig. 4. This figure confirms the sigmoidal relationship. It is also possible to verify that in order to obtain a

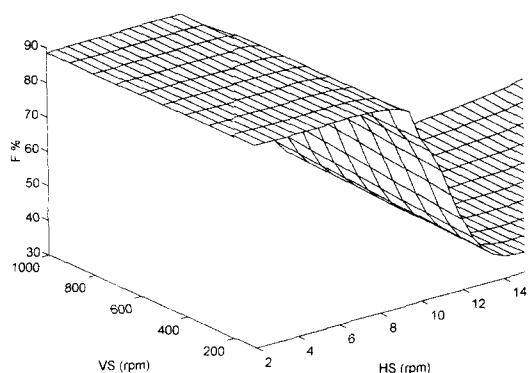


Fig. 4. Friability (F) in function of vertical screw speed (VS) and horizontal screw speed (HS) with RS = 13 revs./min and  $P_{\text{air}} = 300$  kPa.

low friability, HS should be kept high, in the total VS range. The same kind of non-linear behaviour was obtained in Fig. 5 showing the friability F in function of the RS and the HS with VS = 1000 revs./min and  $P_{\text{air}} = 300$  kPa. In order to have granules with a low friability, the ratio RS/HS should be kept around 0.5. Fig. 6 shows F in function of HS and  $P_{\text{air}}$  when setting RS to 13 revs./min and VS to 1000 revs./min. From this figure one might conclude that a low friability can be obtained maintaining  $P_{\text{air}}$  and HS at high levels.  $P_{\text{air}}$  and VS did not affect the F, having its low values when VS is high, in the complete  $P_{\text{air}}$  range. Looking to F in function of RS and VS the lowest F is also obtained at high VS values. When studying the F in function of  $P_{\text{air}}$  and RS it was shown that the best granules were obtained when

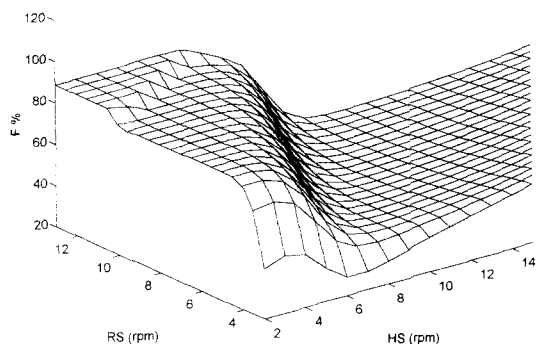


Fig. 5. Friability (F) in function of roll speed (RS) and horizontal screw speed (HS) with  $P_{\text{air}} = 300$  kPa and VS = 1000 revs./min

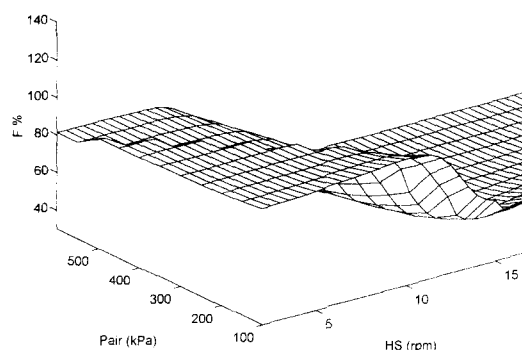


Fig. 6. Friability (F) in function of air pressure ( $P_{\text{air}}$ ) and horizontal screw speed (HS) with VS = 1000 revs./min and RS = 13 revs./min.

$P_{\text{air}}$  and RS were set to high values. The plots VS vs. HS, VS vs. RS, VS vs.  $P_{\text{air}}$ , RS vs.  $P_{\text{air}}$ , HS vs.  $P_{\text{air}}$  and HS vs. RS while keeping the other two parameters constant were put on each other. The region showing acceptable friability was selected, putting all the information together the region which is considered optimal was defined. An experiment was carried out in that region. The experimental conditions selected were  $P_{\text{air}} = 300$  kPa, VS = 500 revs./min, HS = 19 revs./min and RS = 8 revs./min. According to the model, the predicted F is 40%. The experiment led to a F of 37.6%, i.e. 6% relative deviation (RD%) between experimental and predicted values. This is in agreement with the results presented in Table 2 that predicted a mean deviation of 7.5%.

An increase in HS also resulted in an increase of the displacement of the movable roll and thus in a thicker compact leading to granules of lower F. The F as a function of the roll displacement for several HS values is shown in Fig. 7. This figure clearly demonstrated the decrease of the F value when the displacement dramatically increased. Looking at the friability versus displacement when the roll speed was changed the friability had the lowest values if a roll displacement was observed. By changing the compression force ( $P_{\text{air}}$ ), an opposite effect was seen as an increase of the compression force resulted in a decrease of the roll displacement and a decrease of the friability. While keeping the VS value constant, the HS determined the amount of material between the

rolls and controlled the roll gap. If the gap started to change remarkably due to an increase of the HS, better DDWM compacts were obtained. Roll speed seemed to determine the dwell time of the material in the compacting area. Dwell time is known to have a significant influence on type and extent of binding of plastic deforming materials. Falzone et al. (1992) showed a similar dwell time dependent behaviour of Avicel. This product showed more brittle fragmentation if dwell time was too short.

#### 4. Conclusions

The dry granulation process was found to be reproducible. In the modelling approaches a set of data describing the behaviour of the friability in function of the four roll compactor parameters was studied. The behaviour of the variables evaluated by the friability when compacting DDWM could not be described by the quadratic model as well as it could be done by the neural network.

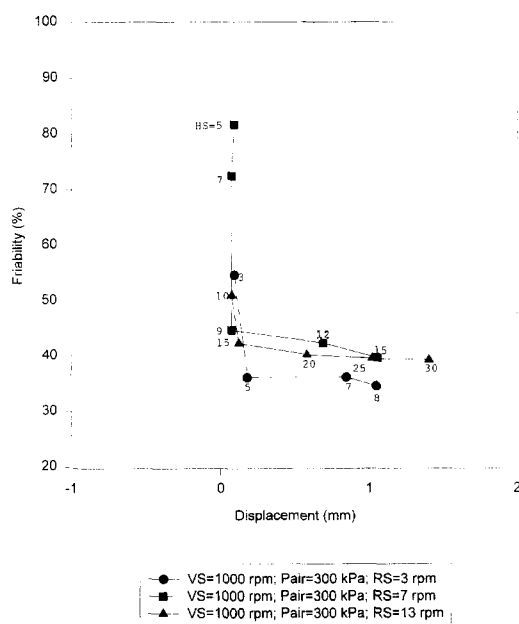


Fig. 7. Friability (%) vs. displacement (mm) if the horizontal screw speed HS (revs./min) is changed.

The horizontal screw speed (HS) and as a smaller extent the air pressure ( $P_{air}$ ) influenced the granule quality. Both parameters should be kept at a high level in order to obtain optimal granule quality. The higher the roll speed (RS) and the vertical screw speed (VS), the larger the region of work of the horizontal screw speed (HS) in the whole  $P_{air}$  range. For compacting DDWM a good granule quality was obtained if the ratio RS:HS was kept around 0.5. The VS had no significant influence on the friability in the range studied.

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