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Optimization of grinding parameters in the production of colorant paste

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ABSTRACT

The tinting strength of a final product together with powerdraw of the mill and grinding time are optimized by adjusting the grinding conditions. The following three-step optimization procedure is described in the paper: in order to determine the most significant grinding variables, the first set of experiments was designed and carried out using the Plackett-Burman method. In the second step, a set of experiments with the selected variables was designed using the Box-Behnken method. The models of tinting strength and grinding power were then identified. In the third step, multicriteria optimization was performed. The paper presents and discusses the optimization results. In particular, the sets of Pareto-optimal solutions are provided for different levels of specific energy. The optimization results of tinting strength and grinding time clearly demonstrate that a low rotor speed combined with a low specific energy can sometimes provide better tinting strength compared with the high rotor speed strategy.

Keywords: Colorant paste, experimental design, tinting strength optimization, pigment grinding

1. Introduction

The use of colorant pastes for paint production has become a widespread practice since the 1970s. Colorants have high color strength and they are normally made of one pigment and a matrix that is suitable for paint production. The same colorant pastes can be used for tinting different kinds of paint in production plants or paint shops. In order to achieve this, both the pastes and basic paints should, however, have precisely determined color strengths.

Pigments are the colorant component that is the most important for the properties and the quality of the final product. Some of the basic properties of pigments are hiding power, gloss, color, density, particle size, reactivity, oil absorption value, lightening power, light resistance, and tinting strength [1]. The tinting strength of a colored pigment is its ability to absorb the incident light and to confer color to the medium in which it is embedded. Tinting strength is an indicator of the yield of a coloring material: an improvement in tinting strength means that a smaller amount of pigment paste is needed to achieve the same tinting strength that the normal product possesses [2], which means savings in raw material costs.

Effective pigment dispersion is necessary in order to obtain optimum tinting strength, cleanliness of shade, and a good gloss in the final product. The fineness of grind and/or the development of color determine the equipment used in dispersion. High speed dispersers (HSDs) are used mainly for pre-mixing the initial pigment powder with liquid [3]. The pigment particle size is reduced in HSDs to < 250 μm. Bead grinding is a

secondary dispersing method for pigments that is widely used in the paint industry. In this process, the pigment particle size can be reduced to <10 μm. The process variables affecting the grinding process in stirred-bead mills are the following: agitator speed / rotor speed [4, 5], feed rate / pumping rate [6, 7], bead size [5, 6], bead charge (in percentage of mill volume) [5], bead density [5, 6], temperature[4], blades design[4], mill chamber shape, and residence time [8, 9]. Important variables in the bead grinding process also include: product density, specific energy, input power, product viscosity and operating methods [10, 11]. Thus, the properties of the final product can be widely varied with different milling conditions.

The primary objective of the experiments presented in this study is to optimize the bead milling process of colorant paste in terms of color strength, specific energy used, and grinding time by changing the grinding variables. The variables studied are bead charge, bead size, rotor speed, viscosity, and feed rate. The desired response of the optimized variable combination is as high a tinting strength as possible with a minimum of specific energy consumption. The secondary objective is to minimize the time needed to reach the specific energy target. This paper is structured as follows: the materials and methods are presented in Section 2, and the experimental design and its three steps are described in Section 3. The first set of experiments is designed and carried out using the Plackett-Burman method [12], the aim being to screen the most significant grinding variables. In the second step, these variables are used to produce a quadratic model of tinting strength based on the Box-Behnken experimental design method [13]. In the third step,

multicriteria optimization is performed. The results are presented and discussed in Section 4.

2. Materials and methods

2.1 Materials

The colorants normally consist of one pigment and different types of additive such as wetting and dispersing agents, biocides, humectants, defoamers, and a solvent. The composition of a typical pigment preparation is: pigment (20-65%), wetting and dispersing agent (3-15%), biocide (0.2%), humectant(0–20%), defoamer (0.1-1.0%), and solvent (remainder) [1,14]. In this study, three different pigment pastes were examined: iron (III) oxide red, cu-phthalocyanine blue, and quinophthalone yellow. The pigment pastes are produced at almost daily intervals at the tinting paste factory under research.

The paste pigment iron (III) oxide red PR 101 was studied first. This inorganic pigment has high viscosity when it is predispersed. Thus only a small amount of water is added, and the pigment requires a long wetting time. The particle shape is spherical, and the predominant particle size is 0.17 μm. The pigment density is 5.0 g/ml.

The blue paste consists of a pigment, whose color index is PB 15:3. The chemical type of this pigment is β Cu-phthalocyanine blue, a green-shaded blue. Copper phthalocyanine blue is the copper (II) complex of tetra aza tetra benzoporphine.

The yellow pigment preparation used in the experiments was Quinophthalone Yellow 138. The chemical formula of the PY 138 is: $C_{26}H_{14}N_2O_4 \cdot 0{,}5(C_7H_8)$.

2.2 Experimental equipment

In the first stage of the colorant paste manufacturing process, the pigments, certain additives and solvents are combined and mixed properly in a high-speed dissolver unit. After the wetting period, the colorant pastes are further dispersed in a high-speed, vacuum dissolver. The best colorant paste dissolution is reached when the ground paste is as thick as possible and the attrition speed creates a proper vortex, which is also required for good dispersion. Colorants are also affected adversely by high temperatures. A practical reason for the temperature limits is that in most cases the viscosity is lower at elevated temperatures. This means that during processing the particle concentration can be kept high providing a good flow of material to be processed. As a result, a better final outcome is achieved in most cases. Moreover, at very high temperatures, the loss of evaporating solvents starts to effect with higher viscosities and worse wetting / stabilization. Finally, sometimes additives are used that decompose at higher temperatures. Normally, the temperature limit for colorants is around 40 - 70 °C, and higher temperatures usually result in a reduction of product quality. To prevent overheating, the mixture is cooled with cooling jackets.

After bead grinding, the rest of the additives and extenders are added and the colorant paste is de-aired. The quality of the paste is controlled and the adjustments are made if

necessary. If the optical and rheological properties of the colorant paste pass quality control, the product is ready to be canned.

In this study, the experimental equipment consisted of a mixing tank in which the pigment paste was mixed with a small dissolver and cooled with the help of cooling water flowing through the cooling jacket. Pigment paste was pumped through a bead mill, where it was ground and then pumped back to the mixing tank. The experiment devices, and measurements are described in Figure 1.

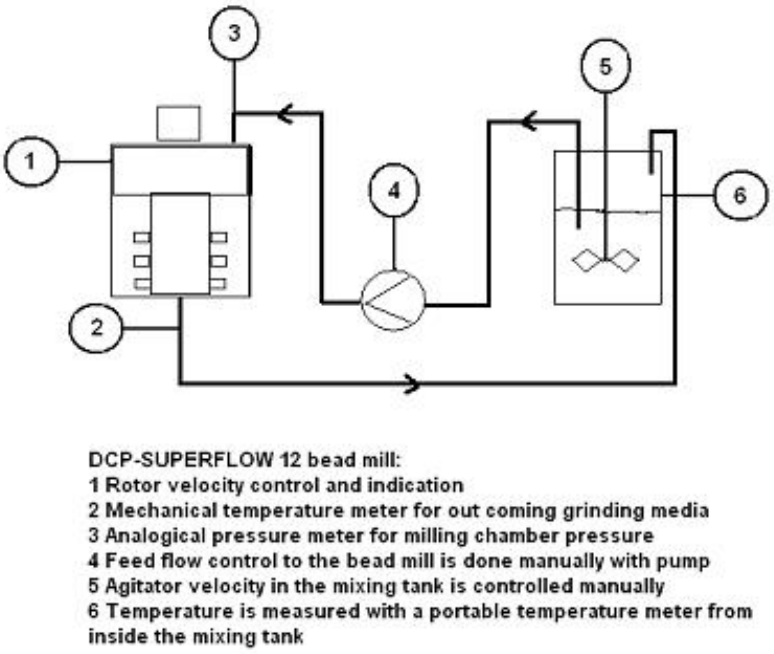

Figure 1. Experimental devices and measurements

The bead mill used in the experiments is a DCP-SUPERFLOW® 12 high-performance agitated media mill. The rotor and stator combination provides two mill chambers connected internally in series. The rotor baffles ensure effective centrifugal separation and the recirculation of the grinding medium. The pegs in the active area of the stator and

the outer side of the rotor provide intensive turbulence in the grinding medium in the mill chamber. The cylindrical protective discharge screen above the inner stator keeps all of the grinding medium inside the mill chamber. The diameter of the grinding medium ranges from 0.2 to 1.5 mm.

The experiments were carried out with Zirmil.2 ceramic beads. The beads are produced from yttrium-doped zirconium powder containing 93% ZrO2 and 5% Y2O3, which provides high grinding efficiency and high wear resistance. The bead sizes used in the experiments were 0.8, 1.0, and 1.5 mm. The physical properties of the Zirmil.2 ceramic beads are shown in Table 1.

Table 1. The physical properties of the Zirmil.2 ceramic beads.

Specific gravity	6 g/cm^3
Bulk density	3.7 kg/l
Hardness Vickers	1200 HV1

The tinting strength measurements were performed with a Konica Minolta CM-3600d spectrophotometer. The color measuring software used was Largo Match 2000 version 1.1.112. The measurements were performed as follows: Colorant was added to the tint base AA. The mixture was then shaken for 2 minutes in a paint shaker. Depending on the pigment in question, a 150 or 300 µm applicator was used to make drawdowns on black and white cards having the bases standard. The panels were allowed to dry for at least 2 hours at 23 °C or until the panels were dry in a 45 °C oven according to the instructions of the research laboratory at CPS Color.

The viscosity was measured with a Brookfield KU-1+ viscometer. The viscosity is displayed in Krebs units (KU) or in the associated gram value (gm). The spindle was of the paddle type and was driven at 200 rpm by a constant speed motor. The reaction torque of the spindle rotating at 200 rpm was converted to viscosity measured in KU. The measuring range was from 40 KU to 141 KU, at weights from 32 to 1.099 g. The accuracy of the viscometer is ±1% of the full scale range.

2.3 Experimental procedure

The colorant pastes investigated in the experiments were taken from normal production immediately before bead grinding. The analysis using the Plackett & Burman method were carried out with PR 101 colorant paste. The production of this iron (III) oxide pigmented colorant paste is challenging because the pigments are not easily dispersed and the pigment particles need a wetting time of almost 24 hours before dispersion is possible. A typical feature of this paste is that it is also normally extremely viscous before being ground in a bead mill.

PR 101 colorant paste (300 kg) was taken from a 5000 kg dispersed pigment preparation. Smaller 10 L batches with a mass of 15 kg each were prepared from this paste for grinding. The pigment paste was then diluted with water to give a final viscosity of 105 KU/ 23 °C or 120 KU / 23 °C.

The batch weight was measured in order to calculate the mass of the pigment preparation to be ground and in order to determine the required grinding energy. Three to four

samples were taken. The first sample was taken when the specific energy consumption was 80 % of the required specific energy indicated by the receipt. Samples were then taken at specific energies of 100 % and 120 %. When the viscosity was also an experimental variable, the first sample was taken immediately after adjusting the viscosity.

2.4. The three-step experimental design and data evaluation

The objective of the experiments was to optimize the bead grinding process of colorant paste with respect to color strength, specific energy consumption, and grinding time.

Step1: Determining the most significant bead grinding variables

Performing Two-level experiments is a common way of excluding insignificant variables from further consideration which makes it possible to reduce the number of experiments needed to identify the model. In order to obtain reliable results, the effect of the changes of the independent variables must be higher than the inaccuracy of the experiments, in other words, the difference between the levels must not be too small. On the other hand, selecting the largest possible difference between levels may increase the effect of the higher-order interactions, which can also distort the results of the test. In the current study, the differences of the levels are selected to be approximately 50% of the total range of the variables.

In this case, the responses were tinting strength and average powerdraw of the bead mill. The standard Plackett & Burman experimental design was used with 5 factors (tinting

paste viscosity, bead charge, grinding bead size, bead mill rotor velocity, and recycle flow rate of the ground tinting paste, see Table 2) and 12 experiments.

Table 2. The screening variables and their values.

		(-)	(+)	
A	**Viscosity**	105	120	KU
B	**Bead charge**	80	85	Volume %
C	**Bead size**	0.8	1.0	Mm
D	**Rotor speed**	1070	1200	Rpm
E	**Feed rate from max %**	90	100	%

The values $C_k, k = 1,...,11$ corresponding to every column of the experimental design matrix are first computed:

$$C_k = \bar{Y}_k^+ - \bar{Y}_k^-,$$

$$\bar{Y}_k^+ = \frac{1}{6}\left(Y_{k,1}^+ + Y_{k,2}^+ + Y_{k,3}^+ + Y_{k,4}^+ + Y_{k,5}^+ + Y_{k,6}^+\right),$$

$$\bar{Y}_k^- = \frac{1}{6}\left(Y_{k,1}^- + Y_{k,2}^- + Y_{k,3}^- + Y_{k,4}^- + Y_{k,5}^- + Y_{k,6}^-\right),$$

where *k=1,…, 11* is a column number, $Y_{k,1}^+,...,Y_{k,6}^+$ and $Y_{k,1}^-,...,Y_{k,6}^-$ are the measurements selected according to the positive and negative signs in the column *k*. While the first five effects C_k describe the factors which are studied, the rest of $C_k, k = 6,...,11$ are used to assess the variance of the effect estimations according to [15]:

$$s_{res}^2 = \frac{1}{6}\left(C_6^2 + C_7^2 + C_8^2 + C_9^2 + C_{10}^2 + C_{11}^2\right). \quad (1)$$

The statistics t_k, $k = 1,…,5$ were computed in the standard way:

$$t_k = |C_k| / s_{res}, \quad (2)$$

where the numerator is assumed to be a normally distributed random variable and the distribution of the squared denominator is assumed to be chi-square distribution with 6 degrees of freedom. The numerator and the denominator are considered to be independent and a t-test [16] was applied to evaluate the significance of the factors. The significant variables are chosen for further experiments when the following condition was fulfilled:

$$t_k > t_{1-\alpha/2}[\nu]. \quad (3)$$

The reliability of the tests could be further improved and the significance of the interactions of two variables could also be checked if sufficiently large number of experiments was used. The experiments are however costly in industrial environment and thus the number of available experiments was strictly limited.

Step2: Identification of tinting strength and grinding power models

The Box – Behnken experiment design was chosen for investigating the dependence of the variables studied (tinting strength and grinding power) on the independent variables (the variables determined to be significant using the Plackett & Burman method) for three different tinting pastes. A total of 15 experiments for three variables with three specific energy levels were needed in the three-level factorial Box-Behnken experimental design. The coefficients of the full quadratic models were identified using the least squares method.

Step 3: Multicriteria optimization

Full quadratic models of both tinting strength and grinding power were used in order to maximize the tinting strength for each power level. The optimization problems were formulated as follows:

$$\max\{TS(x_1, x_2, x_3)\}, \quad (4)$$

$$GP(x_1, x_2, x_3) \geq z,$$

$$a_1 \leq x_1 \leq b_1,$$

$$a_2 \leq x_2 \leq b_2,$$

$$a_3 \leq x_3 \leq b_3,$$

where TS and GP are the constructed quadratic models of tinting strength and grinding power; x_1, x_2, x_3 are the grinding conditions and a_1, a_2, a_3 and b_1, b_2, b_3 are the lower and upper limits for the corresponding grinding conditions. In order to obtain a set of Pareto-optimal solutions, the tinting strength was optimized while the power level was fixed at certain levels. Since grinding time can be expressed as a ratio of specific energy and grinding power, the results of the optimization (4) can be represented in terms of tinting strength and grinding time.

3. Results and discussion

3.1 Determination of the significance of the grinding variables

The effect on color strength was evaluated with a confidence level of 20%, and the effect on the grinding power was tested with a confidence level of 5%. In both cases, a two-side t-test was used. The values of the t-statistics together with the critical values are presented in Table 3and Table 4.

Table 3. The estimated effect and the significance of the variables on color strength.

Variable	**t-statistic**			t_{critic}	**Significance (80% / 100% / 120%)**
	80% energy	100% energy	120% energy		
Viscosity	0.64	0.82	1.71	1.47	no sign./no sign./sign.
Bead charge	1.61	1.38	0.19	1.47	sign./almost sign./no sign.
Bead size	0.34	1.46	0.12	1.47	no sign./sign./no sign.
Rotor speed	0.21	0.99	0.60	1.47	no sign./no sign./no sign.
Pump	1.16	0.07	0.09	1.47	no sign./no sign./no sign.

Table 4. The estimated effect and the significance of the variables on grinding time/used energy.

Variable	**t-statistic**	t_{critic}	**Significance (95%)**
Viscosity	0.02	2.57	not significant
Bead charge	3.30	2.57	Significant
Bead size	2.15	2.57	almost significant
Rotor speed	9.43	2.57	Significant
Pump	0.53	2.57	not significant

The bead charge significantly affected the tinting strength at two energy levels (80% and 100% of the used energy) used, and it also significantly affected the grinding time. The bead size was found to be a significant variable for tinting strength at energy level of 100% and it also affected the grinding time. Since the effect of the rotor speed on the grinding time was obviously very strong (t-value of 9.4), it was decided that this variable should be analysed in more detail. The pump is an insignificant variable in all four experiments. Although viscosity was a significant variable for tinting strength at the 120% energy level, the t-values at the other two energy levels were not high (0.6 and 0.8). It can therefore be concluded that viscosity does not play an important role in the grinding process. Bead charge, bead size, and the rotor speed appeared to be the most important variables. As a result, these variables were chosen for more detailed analysis.

3.2 Quadratic models of tinting strength and grinding power

The three most significant variables, which were chosen for further analysis using the Box– Behnken method, are given in Table 5. In all the experiments, the maximum feed rate was used and the viscosities for different tinting pastes were kept constant.

Table 5. The Box-Behnken variables and the levels of the variables.

Variable	**Symbol**	**Coded variable level**		
		Low	**Centre**	**High**
		–1	**0**	**+1**
Bead charge BC, (V-%)	X_1	900 ml (75%)	960 ml (80%)	1020 ml (85%)
Bead size BS, (mm)	X_2	0.8	1.0	1.5
Rotor speed RS, (rpm)	X_3	940	1070	1200

Three different pastes were tested. The first tests were performed with PR 101 paste, in which the particles to be ground are iron (III) oxide red pigment. The second was a blue tinting paste (PB 15:3) in which the pigment to be ground was β-phthalocyanine blue pigment, and the third paste was PY 138. In addition to measuring tinting strength and grinding power, particle size was measured in the experiments and particle size models were constructed in the same way for all three pastes.

3.2.1 Analysis of the accuracy of the experimental results

In order to estimate the measurement accuracy and the experimental accuracy, five measurements were carried out for each paste and on each of the three batches ground under the same conditions. The measurements obtained for the PB 15:3 paste are presented in Table 6. Samples 1.1, 2.1, and 3.1 used 80% of the specific energy; samples 1.2, 2.2, and 3.2 used 100% of the specific energy; and samples 1.3, 2.3 and 3.3 used

120% of the specific energy. Since the standard deviations given at the bottom of the table are very small, the measurement errors can be neglected. To estimate the variability of the results of trials ground under the same conditions, the standard deviations of the means of the trial measurements were assessed for every level of specific energy and the results are summarized in Table 7 for all three pastes. The errors of colour strength models are expected to be of the similar magnitude as the experimental accuracies are.

Table 6. Measurements of tinting strength and standard deviations for the PB 15:3 paste.

PB 15:3	**1.1**	**1.2**	**1.3**	**2.1**	**2.2**	**2.3**	**3.1**	**3.2**	**3.3**
1	1.37	1.29	1.81	1.79	1.39	1.68	1.07	1.18	2.33
2	1.24	1.33	1.55	1.79	1.42	1.88	1.12	1.33	2.2
3	1.23	1.34	1.49	1.71	1.5	1.83	1.15	1.33	2.34
4	1.41	1.33	1.52	1.83	1.58	1.92	1.16	1.29	2.24
5	1.08	1.33	1.7	1.68	1.46	1.7	1.03	1.12	2.39
Stdev	0.13	0.02	0.14	0.06	0.07	0.11	0.06	0.10	0.08

Table 7. Standard deviations of the average of the tinting strength measurements for trials ground under the same conditions.

	PR 101	**PB 15:3**	**PY 138**
Measurement	0.10	0.08	0.19
Trial	0.60	0.27	0.75

3.2.2 Quadratic models of the tinting strength

In this section, the results of the experiments and the tinting strength models are presented for iron (III) oxide red paste. For the other two pastes, the models were similarly constructed. The experiments on iron (III) oxide red paste were carried out as shown in Table 8 where the largest values of the tinting strength are highlighted. According to Table 8, the highest tinting strength values are achieved with the minimum bead size (which is the most important factor), minimum rotor speed and minimum bead

charge. The tinting strength is predicted on the basis of the minimum bead size to be within 14 to 16.5 % better than the standard value. The coefficients provided in Table 9 are computed for the quadratic model using scaled variables:

$$y = \beta_0 + \beta_1 \bar{x}_1 / 60 + \beta_2 \bar{x}_2 + \beta_3 \bar{x}_3 + \beta_{11} \bar{x}_1^2 + \beta_{22} \bar{x}_2^2 + \beta_{33} \bar{x}_3^2 + \beta_{12} \bar{x}_1 x_2 + \beta_{23} \bar{x}_2 \bar{x}_3 + \beta_{13} \bar{x}_1 \bar{x}_3$$
$$\bar{x}_1 = (x_1 - 960)/60$$
$$\bar{x}_2 = (x_2 - 1)/0.2$$
$$\bar{x}_3 = (x_3 - 1070)/130$$

The standard deviation of the model error is 0.80%, 0.57 %, 0.82% for 80%, 100% and, 120% of the specific energy, respectively, which agrees with the accuracy of the experiments. The captured R^2 is 63.0%, 76.6%, and 76.3%, so the models seem to be able to capture the character of the response of the tinting strength to the grinding conditions.

Table 8. The PR 101 experiments: N is a sample number, x_1 is a bead charge [ml], x_2 is a bead size [mm], x_3 is a rotor speed [rpm]

N	x_1	x_2	x_3	Tinting strength Es 80%	Tinting strength Es 100%	Tinting strength Es 120%
1	900	0.8	1070	13.99	14.88	15.46
2	960	0.8	940	14.46	15.23	15.39
3	960	0.8	1200	14.02	15.07	15.62
4	1020	0.8	1070	15.44	15.67	16.45
5	900	1	940	14.45	14.53	15.56
6	900	1	1200	14.05	14.11	14.99
7	960	1	1070	11.05	11.62	12.59
8	960	1	1070	12.41	12.63	12.90
9	960	1	1070	11.90	13.17	13.26
10	1020	1	940	11.14	12.43	13.19
11	1020	1	1200	12.19	12.76	13.29
12	900	1.5	1070	12.70	13.40	13.95
13	960	1.5	940	14.02	14.15	15.19
14	960	1.5	1200	13.02	13.67	14.12
15	1020	1.5	1070	14.19	14.90	15.61

Table 9. The coefficients of the tinting strength models for the PR 101 paste

SE	β_0	β_1	$\boldsymbol{\beta_2}$	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{23}	β_{13}
80%	11.78	-0.37	**-1.38**	-0.05	0.69	0.73	0.49	0.23	-0.13	0.36
100%	12.47	-0.24	**-1.48**	-0.07	0.58	0.76	0.40	0.25	-0.06	0.18
120%	12.92	-0.28	**-1.40**	-0.10	0.82	0.74	0.53	0.27	-0.18	0.17

3.2.3. Quadratic models of grinding power

Models of the average grinding power required for each paste were developed next. The average grinding power depends on the grinding conditions, but it stays at a constant level during the grinding process if the grinding conditions are constant. Thus a single quadratic model was constructed for all three cases when 80%, 100%, or 120% of the specific energy is used. The results of the experiments carried out with the three pastes are shown in Table 10.

Table 10. Three pastes experiments: N is a sample number, x_1 is a bead charge [ml], x_2 is a bead size [mm], x_3 is a rotor speed [rpm]

N	**x_1**	**x_2**	**x_3**	**Power [kW] PB 15:3**	**Power [kW] PR 101**	**Power [kW] PY 138**
1	900	1	940	2.56	3.00	2.46
2	900	1	1200	4.08	4.68	3.82
3	960	1	1070	4.92	4.14	3.23
4	960	1	1070	3.42	4.27	3.29
5	960	1	1070	3.49	4.28	3.31
6	1020	1	940	3.02	3.29	2.84
7	1020	1	1200	4.46	5.51	4.43
8	900	1.5	1070	3.37	5.22	3.29
9	960	1.5	940	2.78	3.36	2.78
10	960	1.5	1200	4.31	5.59	4.49
11	1020	1.5	1070	3.85	4.75	3.79
12	900	0.8	1070	3.17	3.82	3.17
13	960	0.8	940	2.51	3.07	2.58
14	960	0.8	1200	3.93	4.98	4.20
15	1020	0.8	1070	3.49	4.36	3.66

The models are presented in Table 11. The rotor speed appears to be the most important factor affecting the power intake. In addition, the energy consumption is usually larger with the big size beads.

Table 11. The coefficients of the grinding power models

SE	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{23}	β_{13}
PB 15:3	3.94	0.19	0.31	**0.73**	-0.16	-0.14	-0.24	0.02	0.01	-0.02
PR 101	4.23	0.21	0.15	**0.99**	0.09	0.03	-0.19	-0.16	0.05	0.13
PY 138	3.27	0.24	-0.02	**0.77**	0.03	0.04	0.07	0.00	0.02	0.06

3.3 Optimization of the grinding process

The models presented above were used to find the optimal grinding conditions for each paste. First, tinting strength and the average grinding power were simultaneously optimized. Next, the results of the optimization were presented in a form in which grinding time instead of the average grinding power is used as optimization criteria.

3.3.1 Optimization of tinting strength and grinding power

In the next stage, the full quadratic models of both tinting strength and grinding power were used in order to maximize the tinting strength for each power level, as described in section 2.4. In the case of Iron (III) oxide red paste (PR 101), the relationship between optimal tinting strength and grinding power, when 80%, 100%, and 120% of the required specific energy was used is given in Table 12, and it is presented in graphical form in Figure 2. The particle size models were used to obtain the particle size estimations provided in Table 12.

Table 12. The optimal points on the plot of the tinting strength vs. grinding power for the PR 101 paste for different levels of the specific energy.

N	Bead charge [ml]	Bead size [mm]	Rotor speed [rpm]	Power [kW]	Tinting strength [%]	The particle size μm
A_1	900	0.8	941	2.84	15.93	1.744
A_2	900	0.8	941	2.84	16.39	1.722
A_3	900	0.8	941	2.84	17.03	1.715
B_1	901	0.8	1200	4.44	15.37	1.739
B_2	901	0.8	1200	4.44	15.98	1.713
B_3	901	0.8	1200	4.44	16.85	1.715
C_1	1020	0.8	1200	5.44	14.91	1.727
C_2	1020	0.8	1200	5.44	15.38	1.722
C_3	1020	0.8	1200	5.44	16.09	1.714
D_1	1020	1.5	1200	5.73	14.27	1.757
D_2	1020	1.5	1200	5.73	14.89	1.752
D_3	1020	1.5	1200	5.73	15.42	1.721
E_1	900	1.5	1200	5.84	13.11	1.766
E_2	900	1.5	1200	5.84	13.72	1.763
E_3	900	1.5	1200	5.84	14.27	1.740

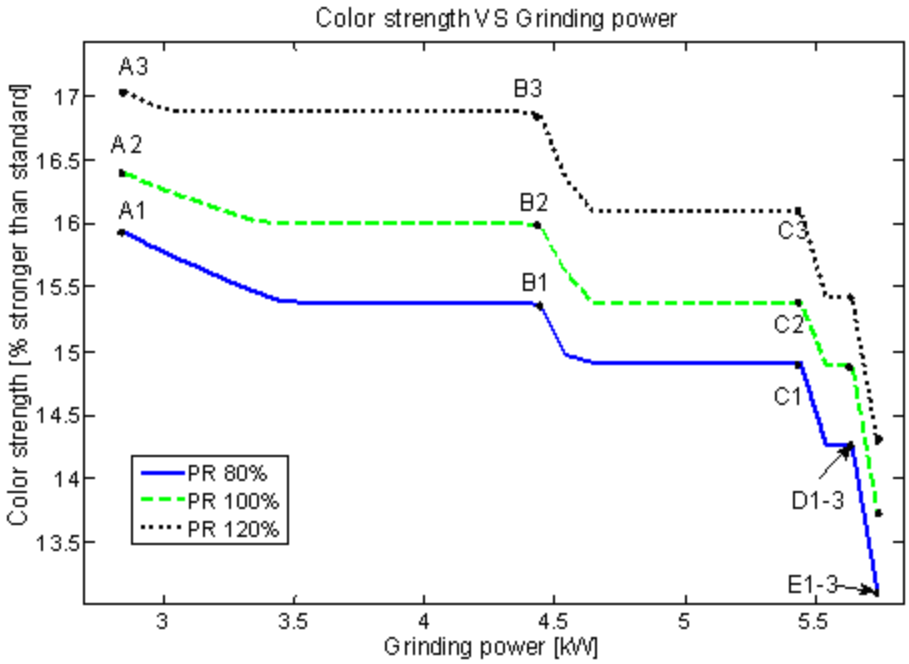

Figure 2 Relationship between the tinting strength and grinding power for the PR 101 paste, when 80%, 100% and 120% of the required specific energy was used.

It can be seen in Table 12, that tinting strength is highly correlated with mean particle size, which is mostly affected by specific energy and bead size. In fact, a good compromise between tinting strength and grinding power can be achieved through the small bead size (resulting in small particle size and high tinting strength) and high rotor speed (which provides high grinding power). These conditions correspond to points 'B' and 'C' in Figure 2 and in Table 12: The low bead charge used at 'B' points results in better tinting strength while the high bead charge used at 'C' points results in better grinding power.

Similar optimization was carried out for the other two pastes. The results obtained for the yellow PY 138 paste are quite similar to the ones presented above. Low bead size used together with low rotor speed achieved the highest tinting strength. Increasing rotor speed improves grinding power but reduces tinting strength. The highest grinding power can be obtained by increasing bead size, but at the price of a significant reduction in tinting strength. The bead charge was kept at the maximum level at all optimal points.

However, bead size did not play an important role in the case of the last paste studied, the PB 15:3paste. In this case, the bead size was kept in the middle of the range between 1.0 and 1.2 mm at almost all optimal points. In fact, only the rotor speed was adjusted, depending on the desired tinting strength and grinding power levels.

3.3.2 Optimization results of tinting strength and grinding time

In order to maximize productivity, optimization of tinting strength must be performed with respect to grinding time rather than grinding power. But since grinding time can be

expressed as a ratio of specific energy and grinding power, the results of the optimization can be represented in terms of tinting strength and grinding time.

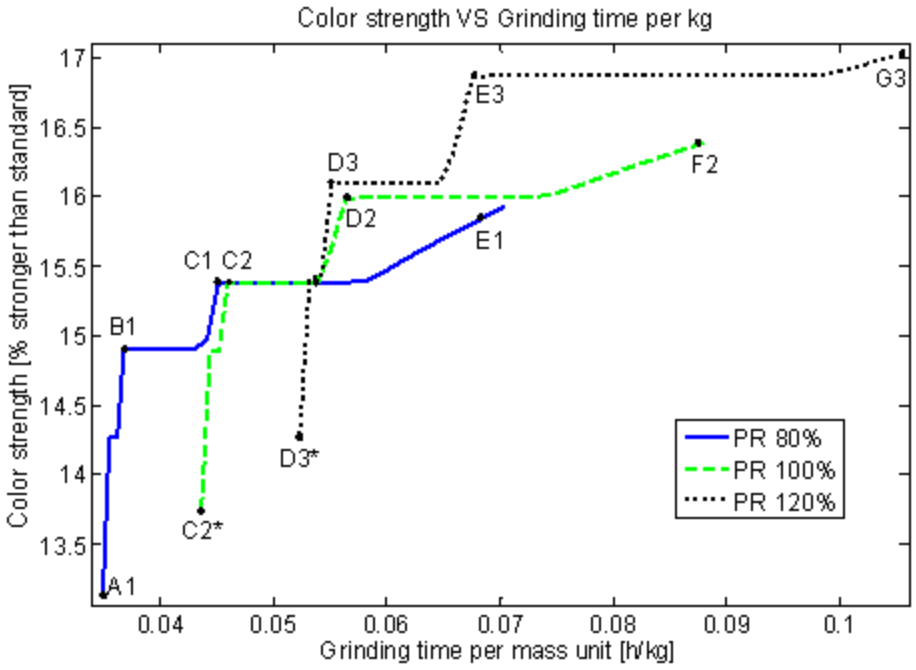

Figure 3. Optimized tinting strength vs. grinding time per mass unit, when 80%, 100% and 120% of the required specific energy was used, paste PR 101.

The relationship between optimal tinting strength and grinding time per mass unit for iron (III) oxide red paste (PR 101) when 80%, 100%, and 120% of the specific energy was used is shown in Figure 3. If the target tinting strength value is smaller than 15 %, the fastest way to reach it is by using the grinding variables at the optimum point ‘B1’ in Figure 3 and Table 13. This point is achieved by using 80% of the specific energy in grinding with a high bead charge (1020 ml), a small bead size (0.8 mm) and a high rotor speed (1200 rpm). It can be seen in Figure 3, that points ‘C1’ and ‘C2’ represent a similar tinting strength when 80% of the specific energy is used in comparison with 100%, while the grinding times are approximately the same. This means that if the required tinting

strength level is lower or equal to 15.38, the fastest and the most energy efficient way to reach it is by using the grinding variable values described at point ‘C1’ in Figure 3.

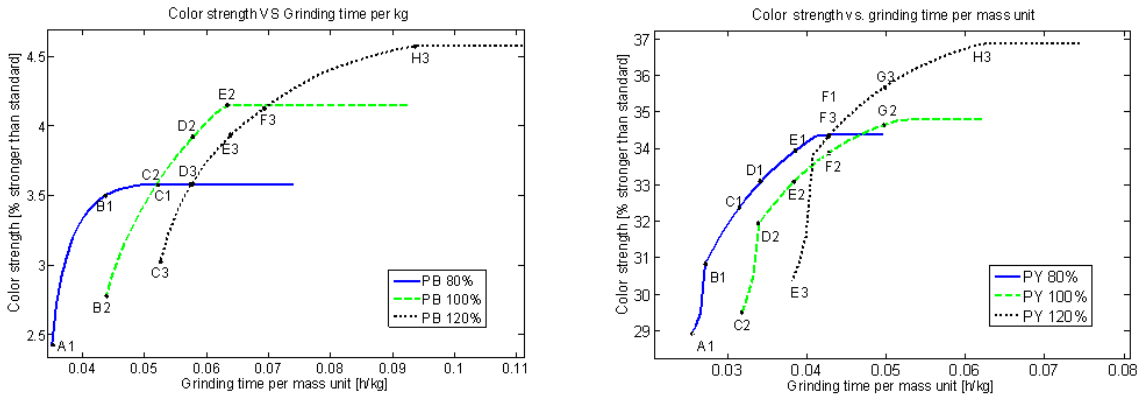

Figure 4. Optimized tinting strength vs. grinding time per mass unit, when 80%, 100% and 120% of the required specific energy was used, for PB 15:3 paste (left) and PY 138 paste (right).

Table 13. The optimal points on the plot of tinting strength vs. grinding time per mass unit for the PR 101 paste for different levels of the specific energy.

N	Bead charge [ml]	Bead size [mm]	Rotor speed [rpm]	Grinding time/mass unit [h/kg]	Tinting strength [%]
A_1	900	1.5	1200	0.034	13.11
B_1	1020	0.8	1200	0.037	14.91
C_1	900	0.8	1200	0.045	15.37
C_2	1020	0.8	1200	0.046	15.38
C_2*	900	1.5	1200	0.044	13.72
D_2	900	0.8	1200	0.053	15.98
D_3*	900	1.5	1200	0.052	14.27
D_3	1020	0.8	1200	0.055	16.09
E_1	1020	1.2	996	0.068	15.83
E_3	900	0.8	1200	0.068	16.85
F_2	900	0.8	941	0.088	16.39
G_3	900	0.8	941	0.106	17.03

Similarly, the points ‘D2’ and ‘D3’ represent almost similar tinting strengths and grinding time despite being at different specific energy levels. This means that if the required tinting strength level is <15.98 %, the fastest and the most energy-efficient way to reach it is by using the grinding variable values at the point ‘D2’ in Figure 3.

The results of optimization for the other pastes are presented in Figure 4.

4. Conclusions

In the study, five bead milling variables were analyzed using the Plackett & Burman method. The aim was to determine which variables affect the grinding process the most. The three most significant variables were found to be rotor speed, bead charge, and bead size, and they were selected for further analysis.

The Box-Bhenken experiment design was used to construct models for the three different pigment pastes iron (III) oxide red, β-phthalocyanine blue, and quinophthalone yellow. These models describe the dependence of the results of the grinding process (including tinting strength and average grinding power) on the grinding variables (bead charge, bead size, and rotor speed). Three of the models approximate the tinting strength with specific energy levels of 80%, 100%, and 120%, and a single model was constructed for the mean grinding power.

The tinting strength and the mean grinding power are then simultaneously optimized for the three pastes. A good compromise between the tinting strength and power is reached for all three pastes when the variables have the following values: bead charge 1020 ml

(the highest), bead size 0.8 mm (the lowest), and rotor speed 1200 rpm (the highest). At this point, significant progress in power is achieved at the price of medium losses in tinting strength. When grinding power is further increased, bead charge and rotor speed remain the same, but bead size has to be increased. As a result, only an insignificant improvement can be achieved in power at the price of a considerable losses in tinting strength.

However, grinding time turned out to be a much more interesting variable than average power because the final aim of the power increase is to decrease grinding time. Actually, the results presented in the form of tinting strength vs. grinding time clearly demonstrate that the fastest grinding versus tinting strength requires low specific energy for low tinting strength demands and higher specific energy for better tinting strength levels. Sometimes a low rotor speed combined with a low specific energy can provide better results compared with the high rotor speed strategy.

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