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Optimization of Flexographic Water-Based Ink Formulation for Polymer Films

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ABSTRACT

In this study, the optimization of flexographic water-based ink formulation was carried out. The aim is to minimize the contact angle of the ink on polymer substrates. A factorial design was used to evaluate the effects and interactions of three factors, which are dispersant, wetting agent and antifoam concentration on the wettability of ink. The optimal conditions obtained from the desirable response are dispersant of 9.65%, wetting agent of 0.58% and antifoam of 0.55 %. Under these conditions, about 15^{o} of contact angle is obtained. The validity of the statistical experimental strategies was verified by the ink samples prepared under the optimized conditions.

Keywords : Wettability, Water-Based Ink, Optimization, Experimental Design

I. INTRODUCTION

Water-based inks have been increasingly used in different applications, particularly in the packaging market since they contain little or no volatile organic compounds. However, the water-based inks are found to be incompatible with non-absorbent substrates (polymer materials), resulting in poor substrate wetting. The reason is that the surface tension of water is 73 mN/m which is much higher than the surface energy of the polymer substrate about 28–32 mN/m. To solve this problem, two ways have been proposed so far: surface treatment (substrate modification) or changes in the ink formulation (ink modification) [1]. The first method is complicated and is only effective for a short time before printing. Therefore, the second method is considered a more suitable solution. Studies in this direction focus on surfactants [2-5] or ionic liquid [6] that are added to the ink as additives (content less than 5%). However, choosing the right additives and their concentrations is not easy in the interaction between the ink and the polymer substrate. The surfactants tend to increase the foam formation during printing, affect the ink transfer and reduce the gloss of the ink film. To overcome this, some anti-foam agents are added. These substances have a negative effect of reducing ink adhesion [4]. all of these make the water-based inks unable to replace the solvent inks on polymer films.

To optimize the ink formulation of the flexographic printing process, in this study the 2k experimental design [5, 6] was applied for evaluation of the individual contribution of selected variables to the wettability of inks on polymer films. The investigated factors were dispersant, wetting agent, and antifoam.

II. METHODS AND MATERIAL

A. Materials

Pigment Blue 15:3 (Phthalocyanine Blue) was used for dispersion supplied by RAMDEV, India. The physical form is a blue powder. Dispersant PAT-ADD DA 103 is a product of PATCHAM Company, America. TEGO® Antifoam 1488 and TEGO® Dispers 760 W (non-ionic wetting additive) are from Evonik Operations GmbH.

B. Milling machine

High-speed dispersing machine BGD 740/1 is used in the milling state. Through mechanical energy (impact and shear forces), the pigment agglomerates are broken up and disrupted into smaller units and dispersed (uniformly distributed) evenly in the liquid. The schematic diagram of this machine is given in Fig.1. The speed of the stirrer can be adjusted from 0 to 4000 rpm. The dispersion blade size is 60 mm. The machine has excellent cooling properties.

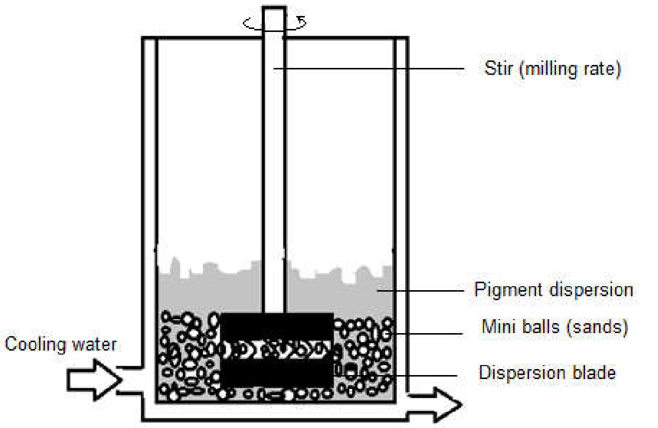

Figure 1: Schematic diagram of dispersing machine

C. Ink preparation

The pigment powder was mixed in the dispersant, wetting agent, anti-foam, additives and water with the required weight concentrations. Total weight of a batch was 500 g. This suspension was then added to the mill. The milling speed was set to the constant value of 3500 rpm and the temperature was kept at 25±2 oC during the milling process. Variations of ink fomulation were investigated.

D. Wettability determination

The wettability of ink was evaluated by the contact angle. A drop of liquid is placed on the polymer film and contact angle can be optically measured (Fig.2). Typically, 90° contact angle is considered as a threshold value. When the contact angle is above 90° the wettability is bad, when it is below 90° the wettability is good. Complete wetting is achieved when the contact angle is zero.

E. Experimental design and data analysis

In this work, a factorial design in which the influences of three experimental factors, e.g. wetting agent, dispersant, and antifoam concentration, on the response, i.e. the contact angle, was investigated. Two different levels were assigned to each factor. The factorial design is shown in Table 1. The levels of the factors are given by – (minus) for low level and + (plus) for high level. A zero-level is also included, a centre, in which all variables are set at their mid value. A sign table, or design matrix, used to calculate the main effects and the interaction effects from the factorial design is constructed in Table 2.

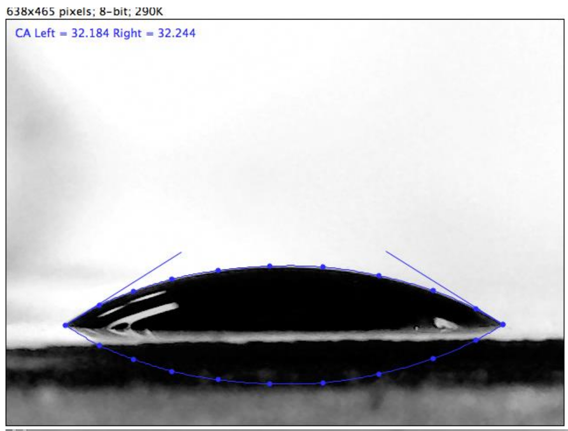

Figure 2: Contact angle image

TABLE I. FACTORIAL DESIGN

Exp. No	Variable			Response
	x_1	x_2	x_3	
1	-	-	-	y1
2	+	-	-	y2

3	-	+	-	y3
4	+	+	-	y4
5	-	-	+	y5
6	+	-	+	y6
7	-	+	+	y7
8	+	+	+	y8

TABLE III. MATRIX OF FACTORIAL DESIGN

Exp. No	X_0	x_1	x_2	x_3	x_1x_2	x_1x_3	x_2x_3	$x_1x_2x_3$	Response
1	+1	-1	-1	-1	+1	+1	+1	-1	y1
2	+1	+1	-1	-1	-1	-1	+1	+1	y2
3	+1	-1	+1	-1	-1	+1	-1	+1	y3
4	+1	+1	+1	-1	+1	-1	-1	-1	y4
5	+1	-1	-1	+1	+1	-1	-1	+1	y5
6	+1	+1	-1	+1	-1	+1	-1	-1	y6
7	+1	-1	+1	+1	-1	-1	+1	-1	y7
8	+1	+1	+1	+1	+1	+1	+1	+1	y8

The experiments were evaluated in order to fit a regression model

$$y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{23}x_2x_3 + b_{123}x_1x_2x_3 \quad (1)$$

Where, y_j ($j = 1 \div N$, $N = 8$) is the response variable to be modeled; x_j ($i = 1 \div 3$) is the independent variable which influence y; b_0, bi ($i = 1 \div 3$), b_{iu} ($i = 1 \div 3$, $u = 1 \div 3$) are model terms, that are estimated by

$$b_i = \frac{1}{N}\sum_{j=1}^{N} x_{ij}y_j \quad (2)$$

$$b_i = \frac{1}{N}\sum_{j=1}^{N} x_{ij}x_{uj}y_j \quad (3)$$

Analysis of variances (ANOVA) was used for graphical analyses of the data to obtain the interaction between the process variables and the responses. The quality of the fitted model was expressed with the coefficient of determination, R^2, and its statistical significance was checked by the F-test. Model terms were selected or rejected based on the p value (probability) with 95% confidence level.

The regression model for real variables (z) describing the relationship between the investigated factors was determined from (1) by replacing variables x with z:

$$x_i = \frac{2(z_i^+ - z_i^0)}{\Delta z_i} \quad (5)$$

Where $\Delta z_i = z_i^+ - z_i^-$; z_i^+ , z_i^0 , z_i^- are values of the i^{th} variable at high, low and mid level, respectively.

The optimum values of selected variables were obtained by using MATLAB 6.0. The interactive effects of the independent variables on the dependent ones were illustrated by three dimensional plots.

III. RESULTS AND DISCUSSION

Two different levels were assigned to each factor. These levels were experimentally determined to assure that the system has the particle size adapting to the requirement of flexographic printing technology (particle size is smaller than 1 μm). The investigated results are reported in Table 3 – 5. Corresponding to the requirement, the experimental domains of three investigated factors were determined (see Table 6).

Eight experiments in the factorial design and three experiments at the centre point were simultaneously performed. All the experiment parameters are reported in Table 7 and the model matrix is given in Table 8.

Coefficient values and statistical parameters obtained for the model are given in Table 9. For assessing the statistical significance of the result, a t-test (t-Student) was carried to the 95% confidence level.

TABLE III

EFFECT OF WETTING AGENT CONCENTRATION ON DISPERSED PARTICLE SIZE

Wetting agent (% wt)	Dispersant (% wt)	Antifoam (% wt)	Particle size (μm)
0.3	5	1	-
0.5	5	1	<1
1.0	5	1	<1
1.5	5	1	<1
2.0	5	1	-

- The formation of clots

TABLE IV

EFFECT OF DISPERSANT CONCENTRATION ON DISPERSED PARTICLE SIZE

Wetting agent (% wt)	Dispersant (% wt)	Antifoam (% wt)	Particle size (μm)
1	1	1	5
1	3	1	<1
1	6	1	<1
1	8	1	<1
1	10	1	<1
1	12	1	-

- The formation of clots

TABLE V

EFFECT OF ANTIFOAM ON DISPERSED PARTICLE SIZE

Wetting agent (% wt)	Dispersant (% wt)	Antifoam (% wt)	Particle size (μm)
1	5	0.5	<1
1	5	1.0	<1
1	5	1.5	<1

TABLE VI

INVESTIGATED FACTORS: LEVELS AND CONDITIONS

Factor	Experimental domain		
	Level (-)	*Level (0)*	*Level (+)*
z_1:Wetting agent (%)	0.5	1.25	2.0
z_2: Dispersant (%)	3.0	6.5	10.0
z_3: Antifoam (%)	0.5	1.0	1.5

TABLE VII

EXPERIMENT PARAMETERS

Exp No	Wetting agent (% wt)	Dispersant (% wt)	Antifoam (% wt)	Contact angle (°)
1	0.5	3.0	0.5	34.50
2	2.0	3.0	0.5	31.82
3	0.5	10	0.5	31.70
4	2.0	10	0.5	34.45
5	0.5	3.0	1.5	27.32
6	2.0	3.0	1.5	32.62
7	0.5	10	1.5	41.55
8	2.0	10	1.5	30.69
9	1.25	6.5	1.0	33.23
10	1.25	6.5	1.0	33.83
11	1.25	6.5	1.0	33.47

TABLE VIII

MODEL MATRIX AND RESPONSE

Exp. No	X_0	x_1	x_2	x_3	x_1x_2	x_1x_3	x_2x_3	$x_1x_2x_3$	Response
1	+1	-1	-1	-1	+1	+1	+1	-1	34.50
2	+1	+1	-1	-1	-1	-1	+1	+1	31.82
3	+1	-1	+1	-1	-1	+1	-1	+1	31.70
4	+1	+1	+1	-1	+1	-1	-1	-1	34.45
5	+1	-1	-1	+1	+1	-1	-1	+1	27.32

6	+1	+1	-1	+1	-1	+1	-1	-1	32.62
7	+1	-1	+1	+1	-1	-1	+1	-1	41.55
8	+1	+1	+1	+1	+1	+1	+1	+1	30.69
9									33.23
10									33.83
11									33.47

As the results shown in Table 9, with the confidence value > 95%, the coefficients, except b_{12}, are significant and the obtained equation is as follows.

$$y = 33.08 - 0.68x_1 + 1.5x_2 - 1.34x_1x_2 - 0.7x_1x_3 + 1.56x_2x_3 - 2.7x_1x_2x_3 \quad (6)$$

This model was then analyzed by F- statistical test for analysis of variance (ANOVA) to assess the "goodness of fit". The analysis results are presented in Table 10.

The value of F-statistic (the ratio of mean square due to regression to mean square to real error) of 925 is much larger than $F_{0.05,8,7}$ (3.73) so the model is significant at the chosen probability level and it is correct [6]. In addition, the lack of fit error was used to test whether the model can fit the data well. The ratio between lack of fit (SS_{lof}) and pure experimental error (SS_{pe}) is much smaller the critical $F_{0.05,1,2}$ (18.51). This result confirms that the model adequately fits the data. The R^2 of 0.99 also indicates that only 1% of the total variation could not be explained by the empirical model [6]. Clearly, at that significance level, it is acceptable to use the obtained model that does not include the rejected terms.

TABLE IX

COEFFICIENT VALUES AND STATISTICAL PARAMETERS OBTAINED FOR THE MODEL

Coefficient	**Coefficient value**	**Standard deviation**	**p-value**
b_0	33.08	0.32	< 0.05
b_1	-0.68	0.32	< 0.05
b_2	1.52	0.32	< 0.05
b_3	0.04	0.32	< 0.05
b_{12}	-1.34	0.32	< 0.05
b_{13}	0.70	0.32	< 0.05
b_{23}	1.56	0.32	< 0.05
b_{123}	-2.70	0.32	< 0.05

TABLE IX

STATISTICAL PARAMETERS OBTAINED FROM THE ANOVA TEST PERFORMED FOR THE MODEL

Source of variation	**Sum of square (SS)**	**Degree of freedom (ddl)**	**Average square**	**Fisher number**	**Signifi-cation**	**R^2**
Regression	14.80	8	1.85	925	3.73	0.99
Residues	0.01	7	0.002			
Lack of fit	0.17	1	0.17	1.89	18.51	
Pure error	0.18	2	0.09			

Replacing the x variables by the z factors, the model for real variables is obtained:

$$y = 51.46 + 1.09z_1 - 6.38z_2 - 15.89z_3 + 2.57z_1z_2 - 5.31z_1z_3 + 6.04z_2z_3 - 2.06z_1z_2z_3 \quad (7)$$

The function above is now describing how the experimental variables and their interactions influence the wettability. The model shows that the interaction of dispersant percentage has a significant effect on the wettability of the ink on the polymer substrate. An increase of dispersant with one scaled unit (e.g. from 3 to 4 %) results in an increase of the contact angle by about 7°. Therefore, to obtain the ink with good wettability on the polymer film, the dispersant concentration has to be enhanced.

The conclusion is clearly shown in Fig.1. A curvature in the surface of these factors indicates that they are interdependent. The response surface also implies that the optimal conditions were exactly located inside the design boundary (Fig.3).

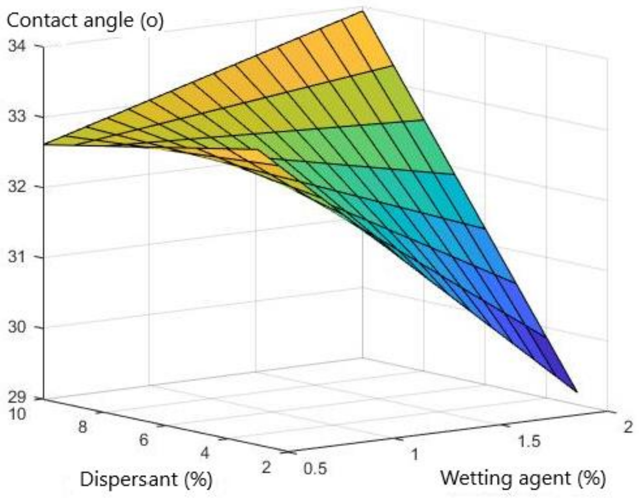

Figure 3: Surface graph of response y (contact angle) showing the effect of dispersant and wetting agent concentration (at antifoam concentration of 1%)

From (6), by using MATLAB software, the optimal conditions are calculated as follows. z1 = 0.58 %; z2= 9.65 %; z3 = 0.55%. Under these conditions the minimized value of contact angle is 15.93°.

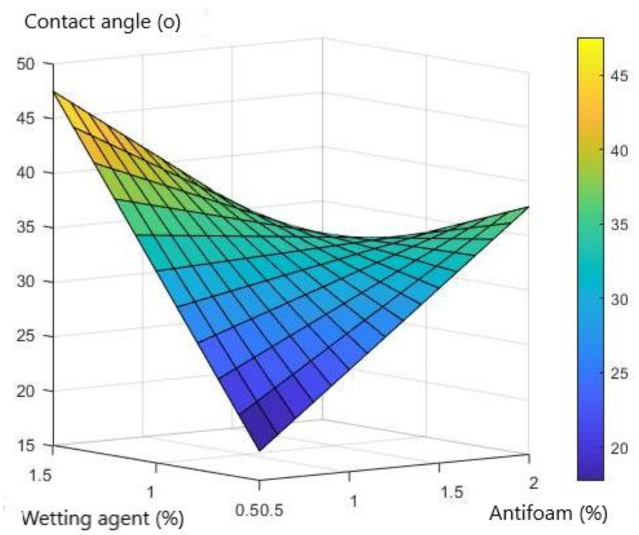

Figure 4: Surface graph of response y (contact angle) showing the optimal ink formulation

IV.CONCLUSION

The results showed that the three factors considered in this study play an important role in the water-based ink preparation. The optimal conditions obtained for wetting agent, dispersant, and antifoam concentration are 0.58%, 9.65% and 0.55%, respectively. Under these conditions, about 15° of contact angle is obtained. The conducted ink showed rheological properties, dispersion stability, and wettability suitable for flexographic water-based inks on polymer films.

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