On The Statistical Prediction of Performance of Coating Color Formulations Under Laboratory Air Knife Coater

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ABSTRACT:- Coating of paper and paperboard is a problem of extremum type. Many parameters like coating color composition, the coating process condition, base paper characteristics, economic factors and the flow behaviour of the coating color slurry etc. are involved to impart a desired property in the coated paper.

The coating color consists of various ingredients such as pigment, binder, dispersing agent, levelling agent, antifoaming agent, plasticisers, preservatives, flow modifier etc. are used. Therefore, the problem or a part of the problem is itself a multivariate system. In order to increase output with a reasonable accuracy within a limited time and effort is to use the statistical design planned experiment and computer to analyse test data. In the present investigation well known statistical and experimental methods are used through sequence of experimental design, experimental methods, regression estimation, optimisation techniques and a synthesis of this method into a unique algorithm for application to coating studies.

In the above planned experimental design well known methods like MacLean Anderson's design have been used for optimisation.

The input variables were % clay, % $CaCO_3$, %TiO₂, % starch, % Casein, nip pressure, coat weight, pigment to binder ratio, and starch to binder ratio to get the optical properties of paper like brightness, opacity, gloss and smoothness through air knife laboratory coaters and the regression equations were developed. The equations predict experimental data within the reasonable accuracy. The % deviation for each of the equation never become higher than 1.55% in the negative side. The equation can be used for predicting the performance. No satisfactory method for interference alleviating of air bubbles could be devised. Inspite of that slight randomness, the methodology can be used for better prediction for the process than the existing trial and error experimentation.

INTRODUCTION

Paper and paperboard is coated to improve primarily the printability of paper by enhancing smoothness and further, improving optical properties such as better appearance in terms of gloss, opacity and brightness. Pigment coating is a process in which

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the surface of paper or board is covered with a thin layer, mainly consisting of fine mineral pigment. The particles of the coating are combined to each other and to the fibers of the base paper through binders which are added to the coating color. The process can generally be divided into three different phases; Preparation of coating color. Coating, drying and calendering. The enhancement of print appearance and surface finish by providing a uniform and more ink receptive surface in comparison to the uncoated papers has to overcome the limitations imposed by the fibrous nature of the paper/coating colour formulations and the quality of the ingredients constituting the coating colour, the mechanism of the coating process, the rheology of the water, retention value of the coating mixture and finally the post treatment of the coated sheet during drying and calendering, while calendering, pressure, temperature and plastic flow are the parameters for responding to pigment applications and uniformity in coat weight distribution.

Out of the above parameters, the most important is the coating color composition. The coating color basically consists of a pigment- binder system with a few other additives to control the flow properties of the slurry.

The pigments commonly used are clay, precipitated $CaCO_3$, TiO_2 , Talc, $BaSO_4$, ZnO etc. either alone or in a combination to achieve a given set of properties for the coated stock. Similarly several types of binders are in wide spread use. Natural binders include Casein, Soy protein, etc. Several varieties of modified starches, synthetic binders (Polymeric), SB Latex etc. are generallor used.

Pigment-binder ratio. properties of different: pigments and ratios of the binders have to be fixed depending upon the properties desired. The coating formulations thus being a multi-component system: the number of possibilities becomes large, and a systematic study would involve extensive experimentation which is not always feasible.

The techniques employ regression analysis to develop regression equations for completion of the experimental design. The planning of experiments needs identifying the variables to be studied. Formulation components blended before the process, coat weight applied during the process and the Supercalendering nip load after the coating layer is dried are the variables chosen for the study. Besides the other variables affecting the process are also identified and kept constant at a level that maximises the coating performance e.g. dispersant quality, agitation time and pH of the coating color.

The entire methodology is discussed in the following paragraphs.

As already said coating of paper is a multivariate system(1,2) and obtaining quantitative estimates of the effect of each of the parameter on the process is difficult, to say the least. The set of variables is large and yet incomplete because not all of them have been investigated or analysed. The complexity of the situation is compounded by the interaction of the variables. The interaction effect cannot be separated from the main effect of a variable. Coating technology is well known to a selected few in the industry and it is generally shrouded in secrecy. The lack of coordinated, planned and complete investigation (which is due to the magnitude and complexity of the problem) gives rise to several misconceptions about the effect of some variables.

For producing a certain grade of coated paper the coating formulation components and the process variables are fixed by trial and error methods. A limited number of experiments are sometimes carried out and a step by step procedure is followed by fixing the level of each component in the formulation one at a time. Experience based estimates by the planned personnel play a major role in such an exercise. Step by step elimination of variables suffers from the drawback of eliminating the interaction effect at the same time. Besides a separate exercise has to be carried out if there is a change in the requirements of quality (in terms of the level of a property) in the finished product.

Experiment can be conducted with one of the two basic objectives: to just study or confirm the effect of some variables or to utilize the generated data set for further analysis. It is best to develop a mathematical model on the basis of theoretical arguments and mathematical analysis and then use the experimental data to find the most probable value of the unknown coefficients in the proposed model. Such a method could not be adopted in the problem

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under consideration due to the large number of variables and the difficulties of keeping all of them constant while one is being studied. The best compromise in such instances is the use of regression equations. From a theoretical basis the variables were identified. The range of values or region experimentation was also fixed to lie in the feasible range.

The number of experiments necessary for the seven variables chosen was very large. It was therefore essential to resolve two methods of experiment design by means of which the points at which the experiments were conducted were selected in a way that gave maximum information in a limited number of experiments. The data generated was subjected to regression analysis. A general equation comprising of 67 terms were chosen to begin with and the insignificant terms successively eliminated after statistical analysis. From these equations the possible ways of attaining a certain set of coated paper properties can be found. Non-linear programming techniques can be applied for optimisation. The whole exercise has resulted in the development to a methodology or a sequence of steps that can be used to study the coating processes and formulation components with respect to their effect on the properties. Coat weight, calendering pressure, %age clay, % age TiO_{2^*} % $CaCO_{3^*}$ % age casein and % age starch in the formulation were the variables investigated. The effect on the paper properties including gloss, opacitys smoothness and brightness were studied and regression equations developed for the same (3)

EXPERIMENT DESIGN:

From the survey of statistics it is found that the factorial designs are used in order to obtain the most complete representation of the system under study. However, if the number of levels of each factor is greater than 3 the number of experiments increases rapidly. Half or quarter replicates of factorial designs are used if some of the regression terms be neglected.(2, 4, 5).

Box Wilson composite design are used for studying regions close to the extremum. Box Hunfer rotatable designs are used for systems possessing some specific mathematical properties.

Latin squares and Graeco Latin squares are

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suitable when some qualitative factors are also being investigated in addition to the quantitative ones. Thus, several design methods are available with each differing in the type of problem to which it can be applied. Agarwal et.al. (2) have reported for the problem involving the study of coating formulation in relation to its effect on coated paper properties, and a mixture design was suggested. For studying the properties of a component mixture a separate set of designs had been developed. The factor space is a (q-1) simplex. In a 3 component system an equilateral triangle adequately represents the factor space. Gibb's and Rozenbum's triangles are used for the purpose. These two types of triangles differ in the way of locating point inside the triangle. The vertices of the triangle represent 1 component while the lines represent 2 components with the third at zero level; any point within the triangle has all the three components in the same proportion.

For quaternary (4 components) systems tetrahedron in which each face is an equilateral triangle represents the system. Each of the four faces is a ternary mixture representation with one component at zero level. Points within the body of the tetrahedron have all four components in the same ratio. Property curves for such system had been represented as a 3 dimensional simplex with the use of contour lines. Response surface in multi-component systems are intricate and polynomials with a large number of terms are needed. The number of experiments must at least be equal to the number of regression coefficient in the regression equation. Based on the Agarwal's (2) suggested lines which was for 4 component system, the present methodology is extended to five component system.

The properties of coated paper can be viewed as a property of the coating colour since it is the composition which affects the properties. Therefore mixture design methods can be applied to the present study. However, it is necessary in this case to carry out the exploration not over the whole range of concentration but in a localised region. The feasible region for preparing coating formulation is thus chosen. This region forms a polyhedron within the tetrahedron and a procedure for finding the design points in this polyhedron is given by *MacLean and Anderson*. The vertices are found out by the procedure outlined.

(1) Range of coating formulation components is chosen. All figures are in the % of the total solids in the formulation. The % solids of the formulation is kept constant at 45%. Its effect thus does not form a part of this study.

Other components used in small quantity are kept constant throughout the experiments.

- (2) All possible combinations of the maximum and minimum level of each component are put down but in each component the content of one component is omitted, thus 80 combinations are obtained for a 5 component system.
- (3) Among all the combinations those are selected whose sum of components is less than unity and which are within the range of investigation is specified by Table-1. Design points thus obtained lie at the vertices of the *bounding polyhedron*.
- (4) Vertices of each face are found and centroid of each face is chosen as a design point. Finally the centre of bounded region is selected as design point.

TABLE-1 RANGE OF EXPERIMENTS Component Range of composition Clay 55-75 % of solids CaCO₃ 10-25 % of solids TiO₂ 5-15 % of solids Starch 3-10 % of solids Casein 8-15 % of solids

PROCEDURE FOR OPTIMAL DESIGN OF EXPERIMENTS IN THE PRESENT INVESTIGATION:

The entire procedure for this particular experiment is illustrated below:

In order to find the relative orientation of the vertices in the space use is made of the concept that two vertices having any two of the four coordinates identical lie on one edge of the polyhedron. From comparison of the 20 co-ordinates the four vertices adjacent to the each vertex are found.

Now, the centroid of the each face has to be

selected. The vertices associated with each face must be known. For point 1 with adjacent points 3,5,12,19 it is necessary that (1,3,5,), (1,3,12), (1,3,19), (1,5,12), (1,5,19), (1,12,19), are sets of points that lie in the same plane. Similarly for all other points, we can find the other adjacent points.

Each face may have three or more vertices. Three co-ordinates of every face being known the remaining co-ordinates of each face can be deduced. Thus, the polyhedron has 9 faces with the vertices:

(1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11), (1, 3, 5, 13, 14, 15, 16, 17, 18, 19) (2, 4, 6, 20) (1, 3, 10, 11, 12, 17, 18, 19) (2, 4, 7, 8, 9, 13, 14, 15, 16, 20) (1, 5, 8, 9, 12, 15, 16, 19) (2, 6, 7, 10, 11, 13, 14, 17, 18, 20) (3, 5, 7, 9, 11, 14, 16, 18) (4, 6, 8, 10, 12, 13, 15, 17, 19, 20)

Centroid of each face is found by averaging the appropriate coordinates. Design points thus selected are distributed over the entire surface so that the best possible representation of the region is obtained in a limited number of experiments 30 formulations have to be prepared. One of the limitations of the laboratory coater is that a predetermined coat weight cannot be applied to the base paper. The coat weight can be varied however within a wide range by changing the air pressure or the knife angle. For each of the

Table-2								
	Selected	Vertices	of The	Polyhed	ron			
N	% Clay	/ % CaCO3	°, TiO2	% Starch	% Casein			
1	55	10	15	10	10			
2	55	2.5	5	3	12			
3	5.5	10	15	5	15			
4	5.5	25	5	7	8			
5	5.5	10	10	10	15			
6	55	25	9	3	8			
7	- 55	22	5	3	15			
8	55 .	22	5	10	8			
9	55	15	5	10	15			
10	55	19	15	3	8			
11	55	12	15	3	15			
12	55	12	15	10	8			
13	57	10	15	3	15			
14	57	10	15	- 10	8			
15	59	25	5	3	8			
16	60	10	5	10	15			
17	64	10	15	3	8			
18	67	10	5	3	15			
19	67	10	5	10	8			
20	74	10	5	3	8			

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formulations sheets of 3 coat weights are produced. At each of these weights 3 sheets are prepared which are calendered at nip loads of 5, 10, 15 kg/ cm^2 . A total of thus 270 sheets are to be prepared.

Sacrificing some accuracy, the centroids were not considered due to various restrictions. The *half* factorial method was applied to further reduce the number of design points.

EXPERIMENTAL PROCEDURE;

On the basis of experiment design, 20 points are selected and formulations for coating colour were prepared. A typical composition of some of these formulations has been shown in the table-3. In addition to these components several other additives in small quantities are used. Soldium hydroxide is added to the pigment slurry to maintain pH and enhance dispersion. Ammonium hydroxide (5.00 % on casein) is used to solubilize the casein. Excess alkali is driven off as ammonia and degradation due to prolonged alkalinity is prevented in the casein solution. Dispersants are used to maximise the zeta potential for mutual particle repulsion. Adsorption of anion on the particle neutralizes part of the surface charge. SHMP was dissolved in water to which the pigment was added. Casein has to be cooked at 60-70°C while agitating. A 20% solid casein solution and starch solution was prepared beforehand for adding to the pigment slurry. The general procedure followed in preparing a coating formulation is illustrated by taking a specific case from the formulation that were prepared. The weight of each component is given below:

The water added to the slurry with the casein,

Table-3						
A typical composition of coating formulation.						
Component	Percentage	Actual weight				
Clay	55.00 % of solids	61.88 gms				
CaCO ₃	10 % of solids	11.25 gms				
TiO ₂	15 % of solids	16.87 gms				
Casein	10 % of solids	11.25 gms				
Starch	10 % of solids	11.25 gms				
SHMP		1.00 gm				
NaOH		2.00 gms				
Water	55 % of slurry	160 ml				

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starch and alkali is subtracted from the total water demand of 160 ml for 45% solids. The dispersant is dissolved in water (70 ml) and pigments added while stirring. A thick paste results on kneading and adding alkali. For proper breakdown of aggregates, kneading of the pigments prior to dispersion in a high shear mixture is beneficial. When a uniform paste has been formed it is dispersed in the kneady, a mill at high shear. A small measured quantity of water is added to facilitate flow in the mixture. As agitation at a high viscosity is more effective because of larger fraction of the energy input is applied to the pigment clusters. The agitation time fixed are 20 mins with the highest attainable rpm or shear. Further increase in agitation time does not pose any significant advantage as the dispersion is already complete by them. The shear rate is more important than time and at high shear rates a short time is required. Increased time cannot compensate for a low shear.

Weighed quantities of binders were added while agitating. Casein initially causes the slurry to become very viscous and almost semi-solid with little flow possible. Continued agitation soon fluidises the slurry as casein also acts as a dispersant. No formulation is allowed to coof and the layer of foam at the top is removed after a stiff film has been formed. Presence of air bubbles is detrimental to coated paper uniformity and ideally, and hence a defoamer/antifoam was used. The slurry is applied to the base stock at ambient temperature.

The laboratory air knife coater is used for the purpose. Coat weight variation is brought about by adjusting the air pressure and/or the knife angle. The slit width of the air knife can also be changed but adjusting it to uniform opening across the width is a problem. Periodic cleaning of the air knife was necessary as any clogging of the slit shows up as non uniformities in the sheet. At each setting of the air knife three sheets were coated.

Drying of the coated sheet is critical in determining binder migration which in turn affects the optical properties and surface strength. To develop the paper properties to the greatest extent. the wet sheets were dried in air until all the free water was removed. The sheets were then finally dried at a constant temperature of 60° C in order to

reduce the time necessary to complete the drying. Sheets were conditioned at ambient conditions for about 24 hours after drying. The coated sheets were supercalendered in the laboratory supercalender at 5, 10 & 15 kg/cm² of nip load. Elevated temperatures during supercalendering were used to develop the properties further. The number of nips were kept constant at seven nips. The sheet was then evaluated for properties and the coat weight was measured.

The variables affecting the sheet properties can therefore be considered to be:

- 1. Coat weight
- 2. % Clay
- $3. \% CaCO_3$
- 4. % TiO₂
- 5. % Casein
- 6. % Starch
- 7. Nip pressure

To minimise random variations in the system, the following factors were kept constant during the experiment.

- 1. Dispersant (SHMP) level kept at 0.5 % of the pigment weight which is close to the optimum level.
- 2. pH of slurry by adding 0.1 % of Sodium hydroxide.
- 3. Ammonium hydroxide used for cooking casein (5%).
- 4. Percentage solids of cooked casein (20%).
- 5. Drying of coated sheets at 60°C.
- 6. Seven nip calendering.
- 7. Twenty minutes of agitation time for pigments.
- 8. Temperature during calendering and coating.

For each sheet the following properties have been evaluated;

- 1. Brightness
- 2. Opacity
- 3. Gloss
- 4. Smoothness

RESULTS AND DISCUSSIONS:

Based on the experimental design, the selection of polyhedron vertices in Maclean Anderson's design were made. The results are available elsewhere (1). A typical set of data for selected vertices of the polyhedron is shown in Table-2.

The experimental results for 49 runs with the variables: % $clay(x_1)$, % $CaCO_3(x_2)$, % $TiO_2(x_3)$, % starch (x_4) , % casein (5), Nip pressure (x_6) , Coat weight (x_7) , pigment to binder ratio (x_8) and starch to binder ratio (x_9) were changed according to the design and corresponding values for Brightness (y_1) , opacity (y_2) , Gloss (y_3) and smoothness (y_4) were estimated.

The Regression Equations:

Based on the techniques of multiple regression the data obtained from the experimental were fitted into equations. The dependent parameters were the properties of the coated paper.

- 1 Opacity
- 2. Brightness
- 3. Gloss
- 4. Smoothness

The independent parameters which were varied in the course of the experiments were

- 1. % clay of the total solids
 - % TiO,

2.

- 3. % CaO_3
- 4. % Casein
- 5. Coat weight
- 6. Nip load

To begin with, a general quadratic regression equation was chosen. This equation comprised of the square of each of the above mentioned parameters and all the possible interaction terms. Subsequently, the basic variables were somewhat modified to terms that are more relevant for coating. From the six variables a new set of basic variables was formed. This includes

- 1. % total pigment in the solids
- 2. % TiO₂ in the pigment
- 3. Pigment to binder ratio
- 4. Latex to binder ratio
- 5. % clay of total solids
- 6. Coat weight
- 7. Nip load

Regression was then carried out using these basic variables and their combinations. Any combination of these variables, to account for the interaction effect and quadratic terms, to account for dependence of a property on a variable were in

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turn represented by a new variable.

Thus, the methods of linear multiple regression were applied though some non-linear terms occured in the equation/high computer programme. The methods outlined before were then used to obtain regression equation for the coated paper properties. The initially assumed equation consisted of 67 terms. Each of the coefficients were tested for significance and the insignificant ones were successively eliminated. The equation obtained consisted of a fewer number of terms despite the large number of variables involved. All of the terms used in the final equation contribute significantly to the particular property. The equations are presented in Table-4 for brightness, opacity, gloss and smoothness.

From these equations the factors that are of relevance for each property can be deduced. The relative importance of each variable in determining of property can also be found. The curves that are finally left after dropping the insignificant coefficients are in concurrence with theoretical consideration while the equation have been obtained from a statistical basis and the insignificant coefficients have been dropped after applying the student-t test. The

Table-4					
· · · · · · · · · · · · · · · · · · ·	Multiple	Regression Equations			
BRIGHTNESS	OPACITY	GLOSS	SMOOTHNESS		
-30.9302X ₁	-10.5798X	79.9927X,	-104.474X,		
-2.2463X ₂	1.0368X ₂	-8.3419X,	24.2516X		
$-20.5281X_{3}$	$-11.2831X_{3}$	63.9206X	-93.31X		
-124.034X	-42.1382X	240.0868X	-237.17X		
68.7508X,	43.3687X	-191.398X	204.588X		
-4.0795X	-7.6997X	31.5728X	6.253X		
-2.4295X,	-3.1899X,	-6.7859X7	-18.2313X		
-48.9X	-1.2427X8	98.8595X	10.208X		
3261.235X	1150.938X9	-6931.5X	8283.191X		
0.1378X ₁ ²	0.0674X, ²	$-0.4141X_{12}^{2}$	0.726X, ²		
0.3129X,*X,	0.1509X ₁ *X ₃	-0.9338X,*X,	1.4273X ₁ *X ₃		
0.3592X,*X	0.1840X,*X	-0.9621X *X	-0.1549X,*X		
0.3424X,*X,	-0.2772X,*X,	0.41X *X	0.1177X ₁ *X ₅		
0.029X *X	0.0839X,*X	-0.2186X,*X	0.0239X,*X		
0.0016X *X,	-0.0032X *X,	0.0664X,*X,	-0.0552X,*X		
0.7194X ₁ *X	-0.0427X,*X	-1.825X ₁ *X ₈	-0.7526X1*X		
0.775X, ²	-0.0460X, ²	0.3215X,2	-0.8041X, ²		
0.4059X,*X	-0.3568X ₂ *X ₄	1.7019X ₂ *X ₄	$-1.2673X_2*X_4$		
-0.2208X,*X	0.2806X_*X	$-1.6855X_2*X_5$	1.023X,*X,		
0.1522X,*X	0.0152X,X,	0.2007X ₂ *X ₀	-0.04482X ₂ *X ₆		
-0.0067X,X,	0.1463X,*X	-0.4795X,*X	-0.0265X ₂ *X,		
0.0611X,*X	-0.005X,*X,	0.941X,&X,	-0.0593X,*X,		
-0.0055X,*X,	0.1198X * X,	$-0.7645 X_{6}^{2}$	0.5828X,*X		
0.0707X *X,	0.0778X *X	-0.7645X ²	0.5828X,*X		
0.1248X,*X,	0.0483X ²	0.0701X ₆ *X ₇	0.0103X ²		
0.3815X ²	-0.0131X ₆ *X ₇	$0.0X_{6}^{2}$	-0.0348X,*X,		
-0.0045X,*X,	0.3761X ₇ *X	0.3039X,*X,	0.0258X ²		
0.1614X,*X	7 - 8		2.0933X,*X ₈		
/here:		· · · · · · · · · · · · · · · · · · ·			
1 =	% Clay	X, =	% CaCO ₃		
1 = 3 = 5 = 7 =	% TiO ₂ %Casu in	X, =	% Starch		
s = .	Coat weight	$\begin{array}{ccc} X_{2} & = \\ X_{4} & = \\ X_{6} & = \\ X_{4} & = \\ \end{array}$	Nip pressure Pigment to hinder ratio		
=	Starch to binder ratio	• ` 8	Pigment to binder ratio		

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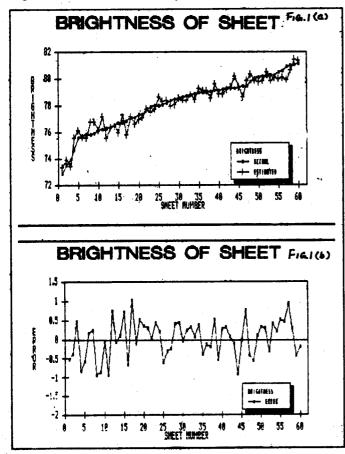
residual terms are the same as those that could have been obtained from a theoretical argument. The data obtained from the experimental observations are plotted in Figs 1-4. The data computed from multiple regression equations finally obtained are compared with the experimental data in 1(a) for brightness, 2(a) for opacity 3(a) for Gloss, 4(a) for smoothness. The relative errors for each output parameters were then calculated for each sheet. The spread of the errors for Brightness, opacity, Gloss and Smoothness were shown in Figs. 1(b). 2(b), 3(b) and 4(b) respectively.

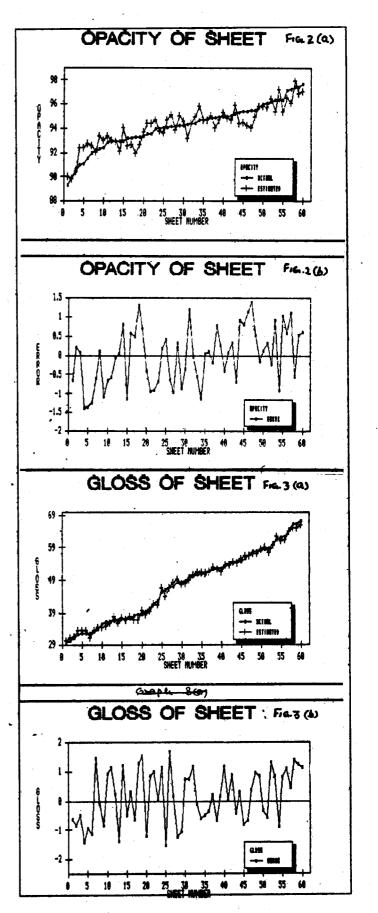
The percentage deviation for each of these equation is:

Gloss	- 1.25	%
Brightness	- 1.00	%
Opacity	- 1.55	%
Smoothness	- 1.3	%

CONCLUSION

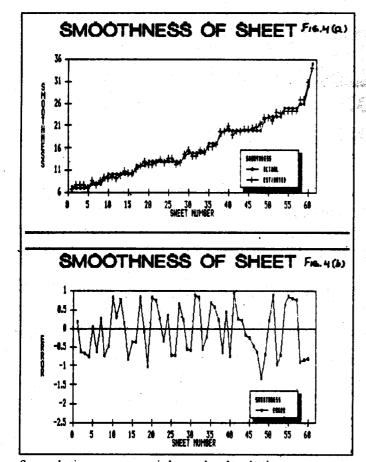
An attempt has been made in this investigation to develop and implement a procedure for quantifying the effect of the components of coating colour





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formulations, coat weight and calendering pressures.

The Maclean Anderson's design and other statistical methods have been adopted and combined into a procedure for statistical optimisation and further verified by experimental investigation. The effects of this exercise has been on a unification of coating concepts with optimisation techniques and well known statistical and experimental design methods. The development of the methodology has been to the sequence of experimental design, experimental methods, a multiple regression analysis, optimisation techniques and a synthesis of these methods into a single methodology for application to coating process.

The algorithm developed can be used for the selection of coating formulation, coat weight and nip load on the basis of the properties desired.

The nip variables for the experiments were coat weight, % clay, % TiO_2 , % $CaCO_3$, Casein, starch and calendering pressures and the output properties evaluated were gloss, opacity, brightness and smoothness. Some additives like SHMP, NaOH, Ammonium Hydroxide are also used for maintaining

process synchronisation. From the experimental results it is found that the surface properties, gloss and smoothness, showgreater randomness than optical properties (opacity and brightness). The evaluation of the properties was done for 61 odd sheets for each property. A regression equation obtained through the statistical optimisation methods can project the coated paper properties fairly well and also give an indication of the relative importance of each factor in a specific paper properties. The procedures illustrated in this investigation with the experimental validation can be applied very successfully not only to study on coating but also to any multi-variate problem. Adopting such procedure can help the designer to make the process more efficient and cost effective by being less wasteful of raw material. The developments can result in a data bank in the form of equations from which each information can be accessed in any desired form for coating applications.

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