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STATISTICAL EVALUATION OF HYPERBARIC FILTRATION FOR FINE COAL DEWATERING

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Abstract – A statistical design of parametric study of pressure filtration for fine coal dewatering is presented. The effects of five major parameters of the dewatering, i.e. applied pressure, filtration time, cake thickness, solids concentration and slurry pH, on cake moisture reduction and air consumption were investigated. The study was conducted starting with two level factorial experiments to identify the most significant parameters, and concluding with response surface methodologies to establish an optimum operating condition for the dewatering of fine coal. It was observed that applied pressure, cake thickness and filtration time were identified to be the key operating variables for reduction of filter cake moisture as well as air consumption. With the key parameters, an optimum condition for the dewatering was determined to be an applied pressure of 93 psi with a cake thickness of 2.5 cm and a filtration time of 4.8 minutes for the laboratory filtration system. At these optimum conditions the filter cake containing about 22 percent moisture by weight and consuming air by 4.1 $m^3/(m^2 \cdot min \cdot kg)$ on dry solid basis was obtained.

Key words: Pressure Filtration, Coal, Factorial Design, Response Surface Methodology

INTRODUCTION

In recent years greater quantities of wet pulverized fine coal have been produced in coal preparation plant. It must be stored, handled, and/or transported to be further processed; dewatered, pelletized or converted to coal-water fuel (CWF) before ultimate use. Such beneficiation is necessary because impuries, such as ash and, in particular, pyritic sulfur, are typically dessiminated through the coal matrix in the forms of inclusions of varying sizes and shapes. Effective removal of such materials requires reduction of the coal down to particle sizes small enough to liberate these inclusions, and then to separated them from the mixture using one or a number of wet processes; washing and flotation and their variants. There are considerable practical and economic advantages to reducing the amount of moisture in fine coal. Wet coal incurs higher transportation and handling charges. Moisture reduces the calorific value of the fuel in the combustion process. Wet coal may freeze and cause handling and utilization problems in cold weather [Parekh and Bland, 1988] . For these reasons the coal industry would prefer to have a product moisture in the range of 10 to 15 percent. Although the desired product quality can be obtained using thermal dryer, there are problems associated with this technology such as high capital costs and the greatest potential source of air pollution in a coal cleaning plant. Therefore, the development and improvement of mechanical methods for filtration of coal slurries and dewatering of coal fines are very important objectives in coal preparation technology for economic, conservation, calorific recovery and pollution abatement reasons.

In the present research project, an alternative to thermal drying, hyperbaric filtration which has shown potential in lowering moisture content in fine coal to less than 20 percent level [Groppo et al., 1995], was investigated using parametric design techniques. Of the most common statistical design methods, the Taguchi-type factorial design as well as central composite design (CCD) have been used in the present work to analyze the performance of a laboratory filtration/dewatering apparatus for the reduction of filter cake moisture and air consumption. This methodology provides several important operating guidelines to optimize the dewatering processes and products. The overall objective of this study was to obtain baseline laboratory data for eventual use in the evaluation of a continuous pilot-scale hyperbaric filter for fine coal dewatering at a preparation plant site.

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MATERIALS AND METHODS

1. Materials

The coal used in our studies was taken from a sample of Pittsburgh seam coal obtained from the Consol Inc. Representative samples of slurries were characterized for percent solids, particle size and ash distribution. Table 1 lists the size and ash analysis as well as the proximate and ultimate analyses for the froth sample. The sample was low in ash content of 8.6 percent. Note that it contained a significant amount of fine (−500 mesh) material. The solids concentration in the slurry was 11.5 percent by weight.

2. Filtration Procedure

The filtration experiments were carried out using a pressure/vacuum filtration cell obtained from Mott Metallurgical Corp. Initial experiments showed a significant segregation of fine particles (−500 mesh) during cake formation phase. To rectify this problem, the pressure cell was modified as shown in Fig. 1. A small stirrer shaft was installed into the filter chamber with a

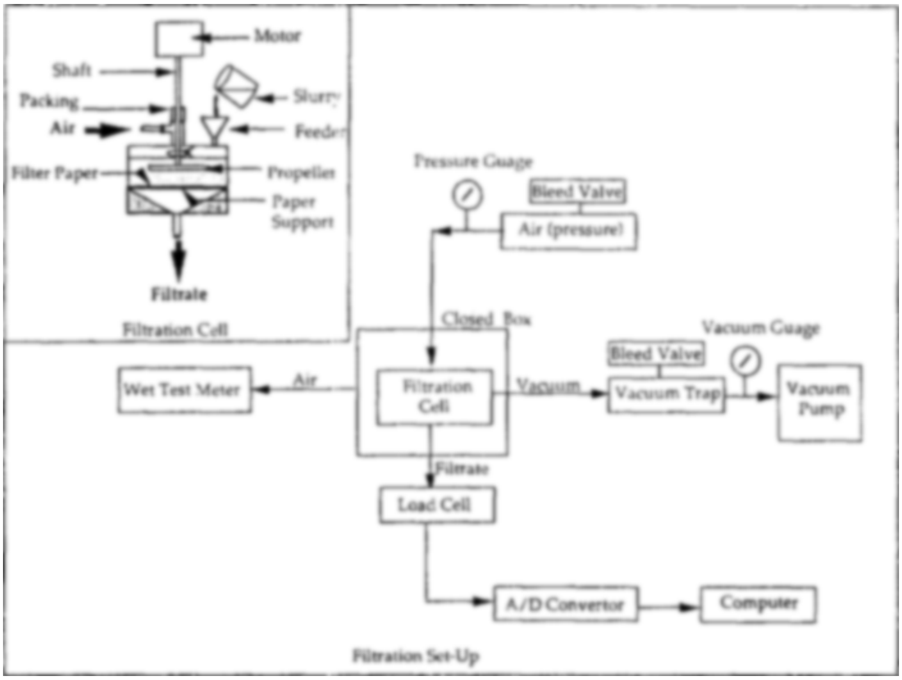

Fig. 1. Schematic diagram of filtration/dewatering apparatus.

Table 1. Size, ash, proximate and ultimate analyses on sample coal

Size, mesh	Weight, %	Ash, %	Ash distribution, %
+100	2.77	2.43	0.8
100×200	19.14	2.52	5.6
200×325	13.59	3.34	5.2
325×500	22.23	3.98	10.2
-500	42.27	15.92	78.2
Feed (calc)	100.0	8.60	100.0
Feed (actual)		8.78	
Proximate analysis		Ultimate analysis	
Moisture	1.59%	Carbon	77.51%
H-T ash	8.78%	Hydrogen	4.48%
L-T ash	9.64%	Nitrogen	1.36%
Volatile matter	29.38%	Sulfur	1.25%
Fixed carbon	60.6%		

Table 2. 2^{5-1} factorial design matrix

Run	A	B	C	D	E
1	1	-1	+1	+1	+1
2	+1	+1	-1	+1	1
3	+1	1	+1	+1	1
4	1	+1	+1	1	+1
5	+1	+1	+1	1	1
6	+1	1	1	+1	+1
7	1	1	1	+1	1
8	1	-1	+1	-1	1
9	+1	+1	+1	+1	+1
10	+1	-1	1	-1	1
11	+1	+1	-1	1	+1
12	-1	1	1	1	+1
13	1	+1	1	1	1
14	-1	+1	+1	+1	-1
15	-1	+1	-1	+1	+1
16	+1	1	+1	1	+1
17	0	0	0	0	0

Teflon packing to prevent air loss. The slurry was then stirred to prevent settling of coarse particles during cake formation stage. It has been described previously that the cake formed with agitation showed a significant moisture reduction compared to that without agitation due to the difference of cake structure [Sung et al., 1994]. A load cell system was used to measure the weight of the filtrate drawn from the filter cake. The load cell generates an electric signal for a small change in the weight, which is converted to a digital signal by passing through an amplifier/conditioner panel. The digital signal is then transmitted to a mini computer in which a code was developed to read and write the input signal in the forms of filtration time and weight of water collected. Wet Test Meter was used to estimate air consumption, which was connected to a closed box sealing the filtration cell. The air being passed through the filter cake enters the Wet Test Meter resulted in an indication of amount of air consumed during the experiments.

The sample slurry was prepared and poured into a sampling cell. The air inlet initially adjusted to a desired pressure was opened to expose a constant pressure to the test slurry. Whatman No.1 filter paper was used as a filter medium. The filtration was continued for a period of 1.5 or 4 minutes whereupon the cake thickness and weight were measured. The final moisture content of the filter cake was determined by drying the cake in an oven at 100 F for 24 hours.

3. Experiment Plan

Classical experiments, i.e. one variable at a time strategy were first conducted on the basis of exploratory test work. Once reasonable dewaterings were obtained, a Taguchi-type fractional factorial experiment was conducted to determine the approximate parameter space for the optimization of the dewatering. The fractional factorial design is useful in screening a large number of variables and helps to identify the most sig-

Table 3. Test variables and levels used in the parametric study

Parameter	Unit	Coded parameter	Test level		
			-1.0	0.0	+1.0
Pressure	psi	A	40	60	80
Filtration time	min	B	1.5	2.75	4.0
Cake thickness	cm	C	0.9	1.6	2.3
Slurry pH	---	D	5.5	7.5	9.5
Concentration	%	E	6.5	9.0	11.5

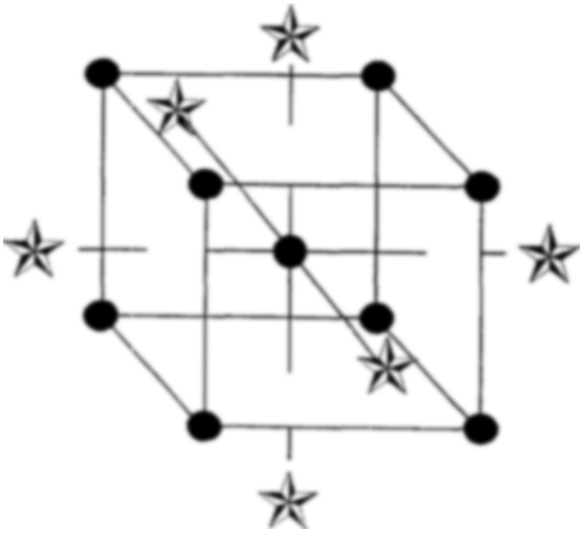

Fig. 2. Central composite design for 3 factors.

nificant parameters with a very small number of experiments [Montgomery, 1991; Box et al., 1978]. The construction of 2^{5-1} factorial design is shown in Table 2. Table 3 presents the five operating variables examined in the study as well as the relationship between the coded and experimental variables. The operating variables were coded as follows:

$$\text{Coded Variable} = \frac{\mathbf{V} - \mathbf{V}_c}{\mathbf{S}} \tag{1}$$

Here, **V** is the experimental variable at a given condition, $\mathbf{V}_c$ is the experimental variable at the center point, and **S** is the step size of the experimental variable. In this design, the five variables at two level were examined and each variable was tested eight times at the high level (+1) and eight times at the low level (− 1) allowing the clear evaluation of all five main factors (i. e. applied pressure=A, filtration time=B, cake thickness=C, slurry pH=D, slurry concentration=E) and all of two factor interactions (i.e. AB, AC, AD, AE and so on).

The factorial design was followed by central composite design (CCD) to extend the parameter space of original Taguchi experiment. The CCD is the most popular response surface design. It combines the two-level factorial with star point and center points as shown in Fig. 2. The star points in combination with the center points allowed the data to be fit to a second-order polynomial. This statistical analysis was accomplished with Design-Ease and Design-Expert software [Whitecomb, 1991, 1992]. The designs evaluate the effects of the dewatering processing variable on the quantitatively measured responses, i.

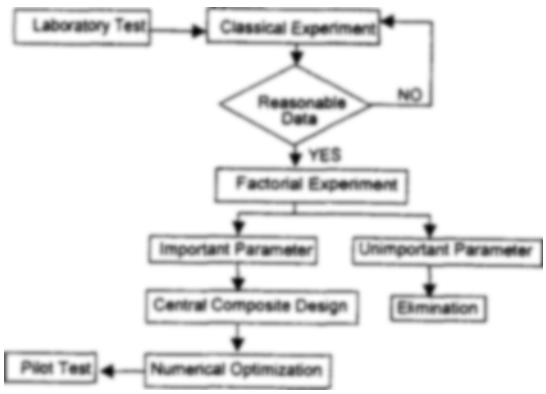

Fig. 3. Flowchart for experiment plan.

e. cake moisture and air consumption. The net effect of each variable is calculated by determining the difference between the positive and negative levels values divided by number of positive level tests as shown in the formula

$$E = \frac{R\ at(+) - R\ at(-)}{No.\ of + Levels} \tag{2}$$

where E is the effect of variable and R is the response (i.e. cake moisture or air consumption).

Numerical optimization was then used to determine a best combination of responses from parameter. The desirability function [Box and Draper, 1987] was utilized to combine the dewatering responses. It optimizes several responses simultaneously. The desirability function, D(x), reflects the desirable ranges for each response (d_i). The desirable ranges are from 0 to 1. The simultaneous objective function is a geometric mean of all transformed response:

$$D = (d_1 \times d_2 \times \cdots \times d_n)^{1/n} = (\Pi d_i)^{1/n} \tag{3}$$

where n is the number of responses in the measure. This simultaneous objective function evaluates to 0 if any of the responses are less than their least desirable state. Conversely if any response exceeds its desired value, it contributes at most 1. 0 to the product. A comprehensive treatment of desirability function is given elsewhere [Box and Draper, 1987].

After conducting laboratory experiments pilot plant tests using Andriz high pressure filter system were performed in order to verify the laboratory test results. Again, central composite design was used to identify variable effects and determine optimum operating conditions. The test program was conducted with the 28×0 and 100×0 mesh coal streams. The variables to be evaluated were chosen based on the laboratory test results. Based on these results, a 30-hour continuous test was conducted to determine performance and operation over an extended time period. Since the results of the field test at coal plant is out of scope in this paper it will be published elsewhere [Fonceca et al., 1996]. The over all flowchart for experimental plan is shown in Fig. 3.

RESULTS AND DISCUSSION

Classical experiments, i.e. one factor at a time experiments were conducted varying each operating parameter while keep-

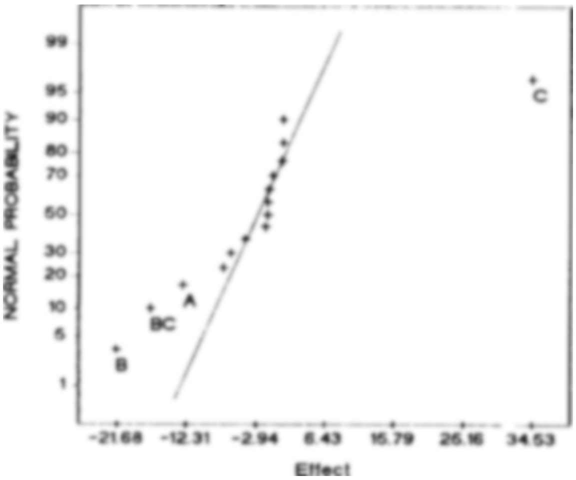

Fig. 4. Normal probability plot of effects.

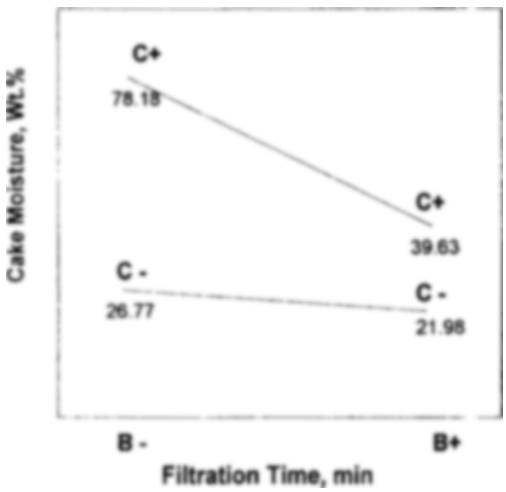

Fig. 5. B (filtration time) C (cake thickness) interaction plot.

ing the others constants. The results were illustrated on the previous paper [Groppo et al., 1995]. Five factors in dewatering process for fine clean coal were then investigated in a 2^{5-1} factorial design with the objective of reducing the filter cake moisture. Graphical procedures based upon normal probability plots were used to choose an appropriate model for the response. The principle underlying the probability plots is that the graph of the ordered values of a sample versus the expected ordered values from the true population will be approximately a straight line. Thus, only a few important effect shows up as outliers on the normal probability plot. Fig. 4 shows a normal probability plot of the effect estimates from this experiment for the filter cake moisture. The main effects of A (applied pressure), B (filtration Time) C (cake thickness) and the BC interaction, revealed as outliers in the normal probability plot, have the most significant influences on the filter cake moisture. The BC interaction illustrates that the effect of filtration time on cake moisture depends on the level chosen for cake thickness as shown in Fig. 5. Thus, the A, B, C and BC model was chosen for the filter cake moisture regression model.

Once the effects analysis was completed, the analysis of variance (ANOVA) calculations were conducted. As shown in Table 4, the A, B, C and BC model for filter cake moisture yielded

Table 4. Analysis of variance for selected model

Source	Sum of squares	Degree of freedom	Mean square	F value	Prob>F
Model (A,B,C,BC)	8421.2	4	2105.3	49.08	0.0001
Curvature	185.5	1	185.5	4.32	0.00618
Residual	471.8	11	42.9		
Total	9078.5	16			

Standard deviation	R-Squared	Coefficient of variation
6.549	0.9469	16.05

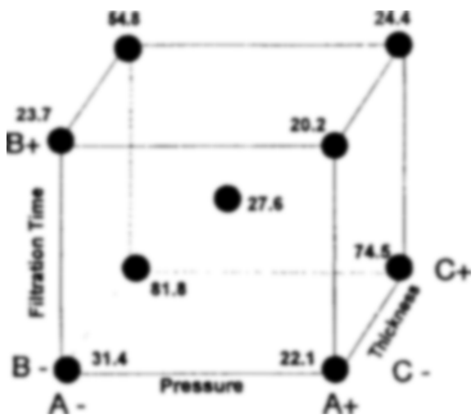

Fig. 6. Projection of 2^{5-1} design into 2^3 design A, B and C.

an F value (i.e. the comparison of the treatment variance with the error variance) of 49.08 and a probability value of 0.01% (i. e. the probability that the model terms are null). Also the coefficient of variation (C.V.) was 16.05%. Additional information on error analysis of ANOVA for selected model was given in Table 4 as well. Since large value of F(i.e. $F \gg 1$) for the model indicates that the error was relatively small for selecting the model term. On the other hands, the F value for the curvature test shows small F value indicating that the curvature between high and low levels is not significant. These statistics present that the A, B, C and BC model was robust for the filter cake moisture. This yielded the regression equation for coded variable:

$$\text{Cake Moisture} = 41.64 - 6.30{*}A - 10.84{*}B + 16.26{*}C - 8.44{*}BC \tag{4}$$

Fig. 6 illustrates the values for combinations of −1 and +1 levels of the three selected variables with center point. The minimum filter cake moisture would be obtained at the +1 value of pressure (80 psi) and filtration time (4 min), and the −1 level for cake thickness (0.9 cm).

Further statistical design of experiments, i.e. CCD has been performed with the three most significant factors, namely A (pressure), B (filtration time) and C (cake thickness) to establish an optimum operating condition for the dewatering of fine coal. Design-Expert program was used to analyze each response of cake moisture content and air consumption using the following methodology: (a) ANOVA analysis was conducted to determine the adequacy of linear, quadratic and cubic models; (b) one model was then chosen for an in-depth regression analysis; (c) diagnostic evaluation of the robustness of the model was determined; and (d) response surface analysis

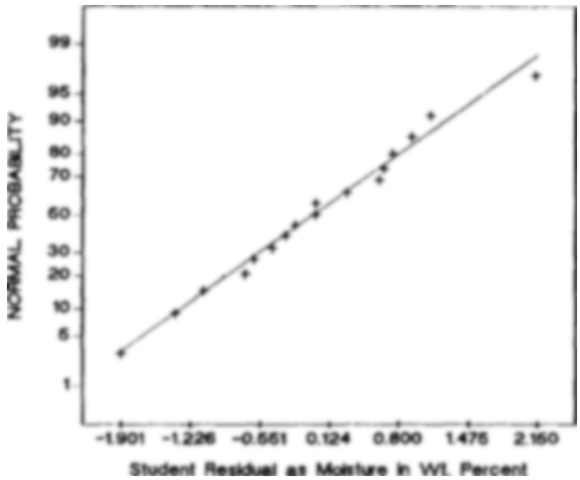

Fig. 7. Normal probability plot of residues.

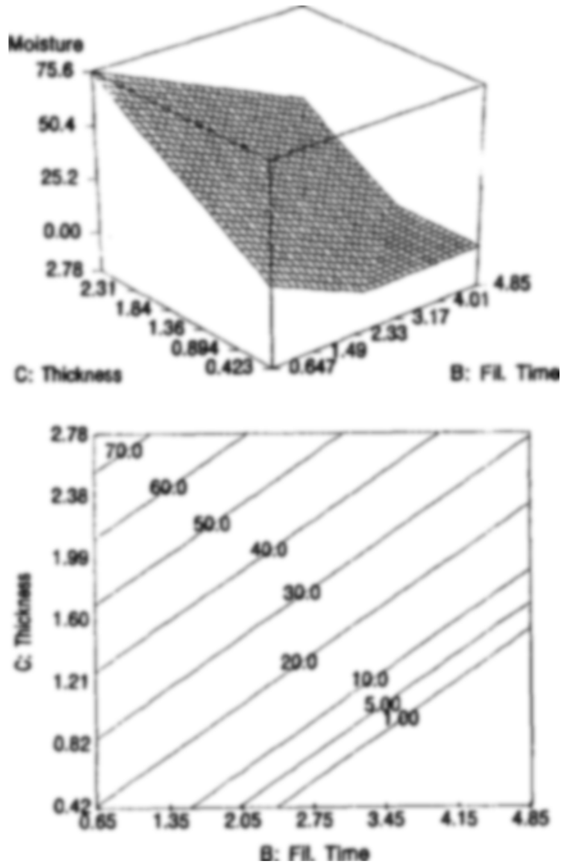

Fig. 8. Response surface and contours for cake moisture at pressure of 93.6 psi.

was conducted to optimize the dewatering attributes.

Central composite experimental design is a response surface methodology where the optimum operating conditions are identified and predicted by a quadratic response surface model. ANOVA calculations for the filter cake moisture showed that F values of 15.34 and 6.60 were obtained for the linear and quadratic models with corresponding probability values of 0.1 and 0.7%, respectively. For the CCD, the cubic model was aliased because of not enough design points to estimate the coefficients. A large F value and a small probability value indicate

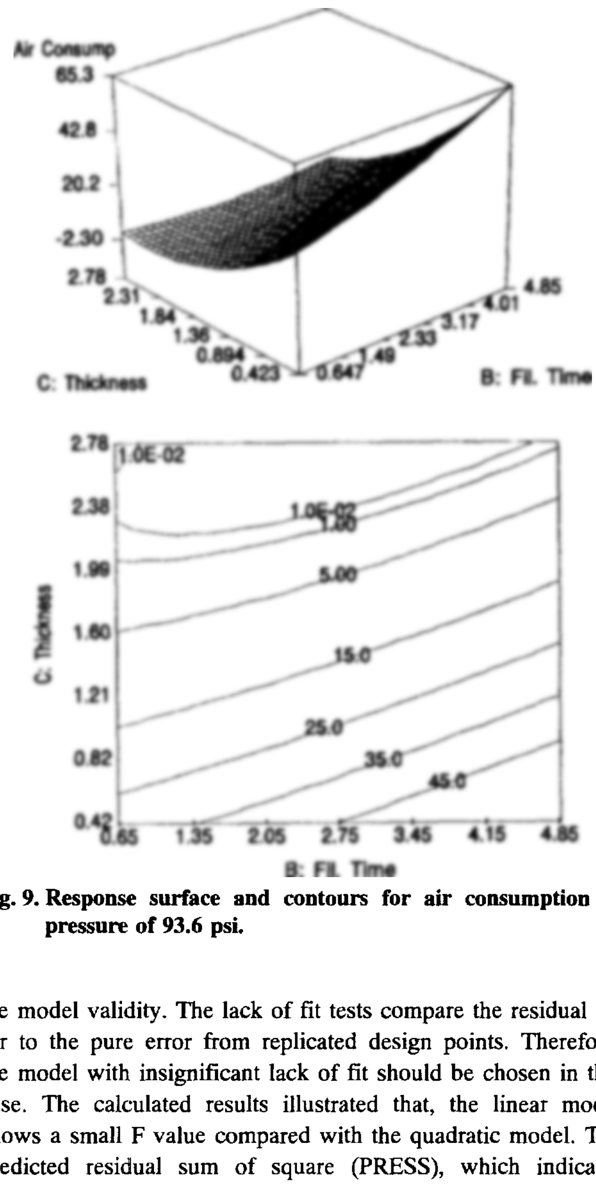

Fig. 9. Response surface and contours for air consumption at pressure of 93.6 psi.

the model validity. The lack of fit tests compare the residual error to the pure error from replicated design points. Therefore, the model with insignificant lack of fit should be chosen in this case. The calculated results illustrated that, the linear model shows a small F value compared with the quadratic model. The predicted residual sum of square (PRESS), which indicates how well the model fits the data, was 4227 for the linear model and 4508 for the quadratic model. The PRESS for the chosen model should be small relative to the other models. Thus, the linear regression model was chosen for the filter cake moisture. With the similar analysis, quadratic model was used for the air consumption. The CCD regression equations for each response are

$$\text{Cake Moisture} = 39.65 - 8.63*A - 13.36*B + 16.63*C \quad (5)$$

$$\text{Air Consumption} = 2.1 + 3.8*A + 2.9*B - 6.83*C + 0.88*A^2 + 1.02*B^2 + 3.62*C^2 + 1.37*AB - 4.28*AC - 3.1*BC \quad (6)$$

Diagnostic evaluation of the robustness of the model was determined with graphical means. The most important diagnostic is studentized residuals. This is illustrated in Fig. 7 for cake moisture in the CCD model. Departures from a straight line show non-normality of the error term. The diagnosis of residuals does not reveal any statistical problems in the regression analysis (i.e., predicted is close to actual). Analysis was then conducted to determined the optimum parameters for the CCD.

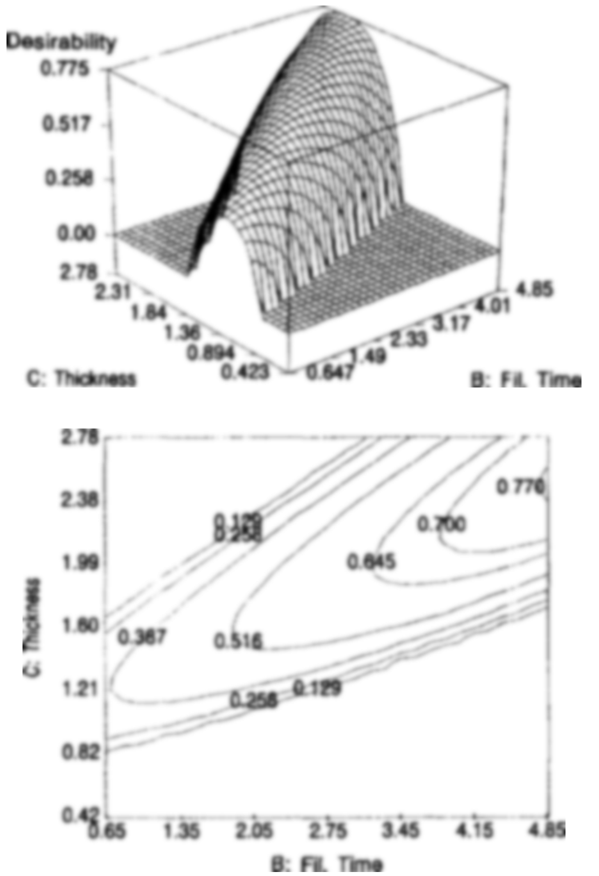

Fig. 10. Response surface and contours for desirability function at pressure of 93.6 psi.

Fig. 8 and 9 plotted the three parameters affecting dewatering performance as a function of filter cake moisture and air consumption, respectively. As shown, these parameters have a strong influence on both cake moisture and air consumption. At a given applied pressure thinner cake thickness and longer filtration time tend to lower cake moisture, but result in a sharp increase in air consumption. From Fig. 8 the minimum filter cake moisture was obtained at the + star point values of applied pressure of 93.6 psi and filtration time of 4.8 minutes and − star point value of cake thickness of 0.42 cm while for the minimum air consumption the trend are vice versa as shown in Fig. 9.

Numerical optimization was then used to determine a best combination of responses from parameter. The desirability function was utilized to combine the dewatering responses. As shown in Fig. 10 the function is at a maximum value of 0.775. The corresponding process parameters for this maximum are applied pressure 93.6 psi, filtration time 4.8 minutes and cake thickness 2.5 cm. Using these conditions the predicted dewatering responses would be cake moisture, 22 weight percent and air consumption, 4.1 $m^3/(m^2 \cdot min \cdot kg)$. The cake moisture and air consumption measurements using the predicted operating conditions provided a quite good agreement with the predicted results within 5 percent absolute variations.

CONCLUSIONS

From the results presented above it can be concluded that:

1. A Taguchi-type factorial experiment was conducted to identify the most significant variables in dewatering process. Of the five control variables studied, applied pressure, filtration time and cake thickness were found to be the most significant. Cake thickness had the most significant effect on both filter cake moisture and air consumption. Since the effect of filtration time on filter cake moisture depends on the level chosen for cake thickness, it was found that there was interactive effect between them.
2. Central composite design was then used to establish an optimum operating condition with the three most significant parameters, i.e. applied pressure, filtration time and cake thickness. It was observed that applied pressure of 93.6 psi with a cake thickness of 2.5 cm and a filtration time of 4.8 minutes provided the filter cake containing about 22 percent moisture by weight and consuming air by 4.1 $m^3/(m^2 \cdot min \cdot kg)$.
3. Since baseline dewatering data was provided as results of this study, the statistical design of experiment on dewatering could be expanded to other important control parameters such as chemical additives (flocculant, surfactant and metal ions), type of additive (anionic, nonionic and cationic) and additive dosage for the further reduction of moisture content of the filter cake.

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