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Response Surface Methodology Approach to Optimizing Process Variables for the Densification of Rice Straw as a Rural Alternative Solid Fuel

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Abstract: This research has been performed with the aim to find out the optimum condition of operating variables for the densification process of rice straw as an alternative solid fuel. A response surface methodology with three levels (-1, 0 and +1) was used as the experimental design. Independent variables to be optimized include densifying pressure, particle size and moisture content of the raw materials. The experiment was performed using a laboratory scale hydraulic press machine in which a number of 17 treatments were randomly implemented following the Bob-Behnken design. The experimental data on the density, relaxation and durability of solid fuel treated as the response variables were fitted into a quadratic polynomial model. The simultaneous optimization of the response variables has been implemented using Desirability Function (DF) approach, computed with the use of Design Expert software. The results of the research showed that the optimum conditions to produce solid fuels from rice straw biomass were obtained at a pressure of 3002.8 psi, the particle size of 34.90 mesh and raw material water content of 7.77%. The application of the optimum conditions enables to produce a solid fuel having a density of at least 1.0 g cm^{-3}. However, further research is still required to investigate the combustion performance of the produced solid fuels in the real combustor.

Key words: Optimization, response surface methodology, densification, rice straw, desirability function

INTRODUCTION

Biomass constitutes the third major natural energy source in the world after oil and coal (Ozbay *et al*., 2006). Up to present, biomass is utilized to supply energy for more than half of world population which is comparable to 1250 million tonnes oil equivalent (mtoe) (Purohit *et al*., 2006). If the biomass can be converted into various forms of energy economically and sustainably, it will not only provide a substitution for fossil fuel but also reduce the carbon emission into atmosphere as biomass has the potential to be CO_2 neutral. Therefore, the utilization of biomass as an alternative energy, particularly in the under-developed and developing countries can also be related to Clean Development Mechanism (CDM) for the investment of carbon trading.

In Indonesia, agricultural residues have become the most important source of biomass since it is an agricultural country with almost 70% of the population living in rural areas. It is estimated that the country is capable of supplying 146.7 million tons of biomass per year, equivalent to about 470 GJ year^{-1} (Abdullah, 2009). As the country has around 7-8 million hectares of rice fields, the largest source of the biomass comes from rice field residues, such as rice straws. To the farmers, rice straw residues do not have any economic value and are source of problem. In order to get rid of the residues, farmers either abandon or burn away most of the rice straws not long after harvesting the paddy. However, burning the rice straws not only releases a large amount of pollutants, affecting the ambient air quality but also substantially contribute to the formation of brown cloud which deteriorates the local atmospheric visibility that may cause the traffic accident. Therefore, a clean and an efficient method are required to convert the bulky rice straw residue into an alternative solid fuel to be used as rural household energy source. A mechanical densification process is proposed as a means which can be implemented to produce the solid fuel.

Biomass densification is not new idea and has been practiced for many years in a number of countries. At commercial scale, the densification is usually performed under high briquetting pressure by means of piston press, extrusion screws or by roll presses, supplemented with

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preheating raw materials (Rhen *et al.*, 2005; Husain *et al.*, 2001; Li and Liu, 2000). High pressure operation provides an advantage of producing strong compacted solid fuel without using binder (Rhen *et al.*, 2005; Li and Liu, 2000). Such an operating condition is however, difficult to implement for a manual scale production in the rural community due to the requirements of high energy. It is therefore necessary to determine the processing and raw materials conditions that lead either to a correct selection or design of machines for densification of biomass based upon manual operation.

A Response Surface Methodology (RSM) approach has been commonly used for optimization studies in recent years. The RSM is a collection of statistical and mathematical techniques useful to empirically study the relationship between a response and several input variables (Myers and Montgomery, 2002). It has been quite common that several response variables are involved in the product or process development. Therefore, the optimization procedure of the independent variables in this case requires a simultaneous consideration of all responses (Jeong and Kim, 2009). The multi-response problem consists of three stages: data collection, model building and optimization. This approach has been applied in many studies, such as mechanical characteristics of polymer concrete (Barbuţă and Lepădatu, 2008), surface roughness in turning process of mechanical parts (Doniavi *et al.*, 2007) and optimization of cement clinkering process (Amiri *et al.*, 2008), to name a few. The present study applies the RSM approach with the aim at finding out the levels of factors in the densification of rice straw biomass that provide optimum response from quality of the product and operational cost point of views through an investigation applying a laboratory scale pressing machine.

MATERIALS AND METHODS

The rice straw biomass was collected from rice fields in the rural areas outside the city of Banda Aceh, Indonesia. Prior to reducing its size, the rice straw was dried in the open air for a week. The dried rice straw was then ground in a milling machine and results were screened into three particle size categories of 20, 40 and 60 mesh. The moisture content of the samples was adjusted to 5, 10 and 15% by weight, respectively. A 10% starch was introduced into the sample to promote the binding among particles.

Proximate and ultimate analysis of the rice straw was performed at the Research and Development Center for Mineral and Coal Technology, Bandung, Indonesia. Proximate analysis provides information on the weight percent of the moisture, Volatile Matter (VM) when heated up to 950°C, Fixed Carbon (FC) and ash in a biomass material, whereas the ultimate analysis presents the weight percent of major elements such as carbon (C), hydrogen (H) and oxygen (O), as well as other elements of sulphur (S) and nitrogen (N). Both proximate and ultimate analysis was conducted in accordance with the recommendation of American Society for Testing Materials (ASTM). Determination of moisture content in dry sample was carried out consistent with ASTM D. 3173. Ash and volatile matter were analyzed in line with ASTM D.3174 and ISO 562, respectively. The fixed carbon was obtained through the following calculation: 100-total percentage of moisture content, ash and volatile matter. With respect to the ultimate analysis, elements of C, H and O were determined using ASTM standard method D. 5373 while total sulphur was performed along with ASTM standard D. 4239. Oxygen was calculated following Eq. 1 (Zhen, 1993):

$$O\ (\%) = 100-(C+H+N+S+Ash) \qquad (1)$$

where, C, H, N, S and Ash are carbon, hydrogen, nitrogen, sulphur and ash percentages in the biomass, respectively. Gross calorific value was measured using adiabatic bomb calorimeter following the ASTM standard D. 5685.

The independent variables being studied were densifying pressure X_1, particle size X_2 and moisture content X_3, keeping a 10% starch binder as a constant variable. The dependent variables analyzed were density, relaxation and durability of densified biomass produced. The densification experiments were conducted using a bench type manually operated laboratory hydraulic press having a capacity of 20 ton (Hydraulic Press Shop CMC ISO9002) and a densification die. The densification die was constructed from stainless steel cylinder of 30 mm in internal diameter and 250 mm in length, equipped with a stamp of 30 mm in external diameter. A Bob-Behnken design with three levels, low, medium and high coded as -1, 0 and +1 was applied to this study. The level values of each variable and code investigated in this study is presented in Table 1.

The density of the produced briquettes was found by a simple method as a ratio of weight and volume

Table 1: Experimental range and levels of independent variables

	Coded level and range		
Independent variable	-1	0	+1
Pressure X_1 (psi)	3,000	5,000	7,000
Particle size X_2 (mesh)	20	40	60
Moisture content X_3 (%)	5	10	15

Table 2: Bob-behnken design matrix along with experimental data predicted results

Run	Pressure X_1 (psi)	Particle size X_2 (mesh)	Moisture content X_3 (%)	Density (g cm^{-3})		Relaxation (%)		Durability (%)	
				Exp.	Predicted	Exp.	Predicted	Exp.	Predicted
9	0	-1	-1	1.0877	1.1054	0.1791	0.1872	52.9040	41.6800
12	0	1	1	1.2357	1.2240	0.0690	0.0700	1.7188	0.9400
10	0	1	-1	1.2358	1.2194	0.1346	0.1352	1.3586	-5.6400
11	0	-1	1	1.1156	1.1380	0.2522	0.2620	51.7320	46.7400
13	0	0	0	1.0812	1.0847	0.1155	0.1200	12.1523	12.1500
5	-1	0	-1	1.1282	1.1063	0.0982	0.1076	30.0903	36.4300
8	1	0	1	1.1282	1.1569	0.0982	0.0972	30.0903	23.7500
17	0	0	0	1.0812	1.0847	0.1155	0.1200	12.1523	12.1500
2	1	-1	0	1.1424	1.1006	0.2096	0.2156	22.7296	22.0800
14	0	0	0	1.0812	1.0847	0.1155	0.1200	12.1523	12.1500
1	-1	-1	0	1.0033	1.0166	0.2438	0.2400	69.2736	62.1400
6	1	0	-1	1.0812	1.1143	0.1439	0.1444	10.0529	9.9300
3	-1	1	0	1.1208	1.1686	0.1048	0.1088	5.3517	-5.9800
15	0	0	0	1.0812	1.0847	0.1155	0.1200	12.1523	12.1500
16	0	0	0	1.0812	1.0847	0.1155	0.1200	12.1523	12.1500
4	1	1	0	1.1548	1.1486	0.0892	0.1028	1.9477	-2.9200
7	-1	0	1	1.1282	1.1009	0.1552	0.1644	34.1316	34.2500

determined from the briquette geometric shape. Relaxation in volume was measured after the briquettes were stored for a week, utilizing a vernier calliper. The durability of the produced solid fuels was measured in accordance with ASAE S269 (ASAE, 1996).

A number of 17 runs were randomly performed to optimize the process variable, as shown in Table 2 together with the experimental and predicted results of the dependent variables: the density, relaxation and durability of the densified rice straw biomass. The experimental data were analyzed by RSM using Design Expert software (Version 6.06, State-Ease Inc. Minneapolis, USA) to fit the second order polynomial relationship, as shown in Eq. 2:

$$Y_k = \beta_o + \sum_{i=1}^{3}\beta_i X_i + \sum_{i=1}^{3}\beta_{ii} X_i^2 + \sum\sum_{i<j}\beta_{ij} X_i X_j + \varepsilon_j \tag{2}$$

where, Y is the predicted response and X_1, X_2 and X_3 are coded independent variables corresponding to the pressure, particle size and moisture content, respectively. Constants β_o, β_i and β_{ij} are linear term, quadratic term and cross product term coefficients, respectively. The coded values are related to the real values through Eq. 3:

$$Z = \frac{(X - X^{\circ})}{\Delta X} \tag{3}$$

where, Z is the coded value (-1, 0 or +1) and X is the corresponding original un-coded value, while X_0 the mid value of the domain, ΔX represents as the increment of X for every unit of Z.

For the purpose of optimizing multiple response variables, it is necessary to establish the optimum criteria in accordance to the Desirability Function (DF) approach, as proposed by Derringer and Suich (1980). The maximum or minimum value of the variable response is determined on the basis of technical and/or economical considerations. The general approach is to first convert each response Y_k into an individual desirability function $d_k = h\,(Y_k)$ that may vary over the range of $0 \le d_k = 1$. If the response Y_k meets the goal or target value, then $d_k = 1$ and if the response falls beyond the acceptable limit, then $d_k = 0$. The individual desirability functions are then combined into a single composite response, the so-called Desirability Function (DF), defined in Eq. 4 as the geometric mean of the different d_k-values:

$$DF = \left[\prod_{1}^{3} d_k\right]^{1/3} \tag{4}$$

It is clear from Eq. 4 that DF will be close to 1.0 if all individual desirability functions are also close to 1.0. Therefore, DF = 1.0 implies that all response variables are at their respective optimum or target value condition. This type of methodology has been successfully applied for the optimization of mechanical densification process of agricultural crop residues for the production cattle feed (Munoz-Hernandez *et al*., 2006).

RESULTS AND DISCUSSION

Table 3 presented the results of proximate and ultimate analysis of rice straw from this study and compared with the one of Calvo *et al*. (2004). With the exception to the volatile matter in the proximate analysis, other data in both analyses of the two studies are comparable. With regard to HHV, the result obtained from Calvo *et al*. (2004) is slightly higher than that of this study, due to its higher carbon content. However, both HHVs are in the range of normal values, as shown by

Table 3: Properties of rice straw biomass

Proximate analysis					
Moisture (%)	Ash (%)	Volatiles (%)	Fixed carbon (%)		
9.35[a]	18.28[a]	57.67[a]	14.70[a]		
7.43[b]	19.07[b]	67.95[b]	12.98[b]		
Ultimate analysis					
C (%)	H (%)	N (%)	S (%)	O (%)	HHV (cal g^{-1})
35.64[a]	4.90[a]	0.84[a]	0.14[a]	40.20[a]	3432[a]
37.87[b]	4.61[b]	0.63[b]	0.14[b]	34.87[b]	3515[b]

[a]This study, [b]Calvo *et al.* (2004)

Table 4: The fitted model equations

$Y_1 = 1.08+0.016X_1+0.050X2+0.0093X_3-0.014-X12+0.038X22$
$+0.049X32-0.026X-1X2+0.012X1X3-0.007X2X3$
$Y_2=0.12-0.0076X1-0.061X2+0.0024X3+0.0058X12+0.041X22$
$+0.0026X32+0.0046X-1X2-0.026X1X3-0.035X2X3$
$Y_3 = 12.15-9.25X1-23.28X2+2.91X3+0.0058X12+5.92X22$
$+8.02X32+10.78X-1X2+4.00X1X3+0.38X2X3$

Y_1 = Density, Y_2 = Relaxation and Y_3 = Durability of solid fuel

Table 5: Analysis of variance (ANOVA) of the fitted models

Sour	SS	DF	MS	F-V	Prob>F
For Y_1					
Model	0.044	9	4.8×10^{-3}	4	0 . 0 407
Residual	8.5×10^{-3}	7	1.21×10^{-3}		
Lacko of fit	8.5×10^{-3}	3	$2..83\times10^{-3}$		
Pure error	0	4	0		
Cor total	0.0 5 2	16			
R^2 = 0.8371; adjust-R^2 = 0.6276; Pre-R^2 = -1.6068 adeq; Prec 7.761; C.V. = 3.12; PRESS= 0 .14					
Sour	SS	DF	MS	F-V	Prob>F
For Y_2					
ModEL	0.045	9	5.0×10^{-3}	11 2	<0.0001
Resid	3.1×10^{-3}	7	4.53×10^{-5}		
Lacko of fit	3.1×10^{-3}	3	1.01×10^{-3}		
Pure error	0	4	0		
Cor	0.045	16			
R^2 = 0.9931; Adjusted R^2 = 0.9842; Pre-R^2 = 0.8897; adeq Prec = 37.289; C.V. = 4.82; PRESS = 0.005					
Sour	SS	DF	MS	F-V	Prob>F
For Y_3					
Model	6.3×10^3	9	6.2×10^2	24.9	2.1×10^{-4}
Resid	2×10^2	7	28.09		
Lack of fit	2×10^2	3	65.54		
Pure	0	4	0		
Cor	6.52×10^3	16			
R^2 = 0.9697; Adjusted R^2 = 0.9308; Pre-R^2 = 0.5158; adeq = 16.763; C.V. = 24.21; PRESS = 3145.70					

Y_1 = Density, Y_2 = Relaxation and Y_3 = Dlurability of solid fuel

Huang *et al.* (2008) who studied 172 rice straw samples from around China and found out that the minimum and maximum values of HHV are 3051 cal g^{-1} and 4000 cal g^{-1}, respectively.

Table 2 presented the design matrix in the coded units in conjunction with the experimental data and the predicted values of three response variables, the density, relaxation and durability of densified rice straw. The predicted values of the response were calculated from quadratic model fitting techniques utilizing Design Expert software. The experimental data, the density, relaxation and durability of solid fuel produced were utilized to develop the statistical model using multiple regression analysis method to fit the response function in accordance to Eq. 2. The resulted relationships between each response variable and independent variables of pressure, particle size and moisture content are presented in Table 4, where Y_1, Y_2 and Y_3 are density, relaxation and durability of the solid fuel produced, respectively.

The significance of the statistical model shown in Table 4 was evaluated by the F-test analysis of variance (ANOVA) presented in Table 5. In Table 4, the value of “Prob>F” less than 0.0500 revealed that the quadratic model of the response variable is significant at 95% confidence level. From inspection of the value of “Prob>F”, it can be concluded that all models representing the response variables are significant. The model showed a relatively high determination coefficient, R^2 and low the coefficient of variation C.V. These values are obtained as follows: $R^2 = 0.8371$ and C.V. = 3.12 for Y_1, $R_2 = 0.9931$ and C.V. = 4.82 for Y_2 and $R^2 = 0.9697$ and C.V. = 24.21 for Y_3. The closer the determination coefficient to unity, the better agreement of the model suits the experimental data, showing less the difference between the calculated and measured values.

Myers and Montgomery (2002) also suggested that the model adequacy can be evaluated not only from R^2 but also from adjusted R^2, predicted R^2 and prediction error sum of squares (PRESS). A good model is indicated by a large R^2 and a low PRESS. In this case, $R^2 = 0.8371$; adjusted-R^2 = 0.6276; predicted-R^2 = -1.6068; adeq precision = 7.761 and PRESS = 0.14 for Y_1. A negative "Pred R-Squared" implies that the overall mean is a better predictor of the response than the current model. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The current study showed ratios of 4.801 for Y_1, 37.289 for Y_2 and 16.763 for Y_3, respectively which indicate an adequate signal, confirming each model can be used to navigate the design space.

It is clearly seen that the residual values are normally distributed on both sides of the line indicating that the experimental data are in excellent agreement with the predicted values. The above findings indicate outstanding adequacy of the proposed quadratic model to represent the variable responses of density, relaxation and durability of the densified rice straw in the range of pressure: 3000-7000 psi, particle size: 20-60 mesh and moisture content: 5-15%, respectively.

The normal probability plots showing the distribution of residual value defined as the difference between the predicted and experimental data for all response variables of density, relaxation and durability of the solid fuel are forming a straight line, as shown in Fig. 1a-c, respectively.

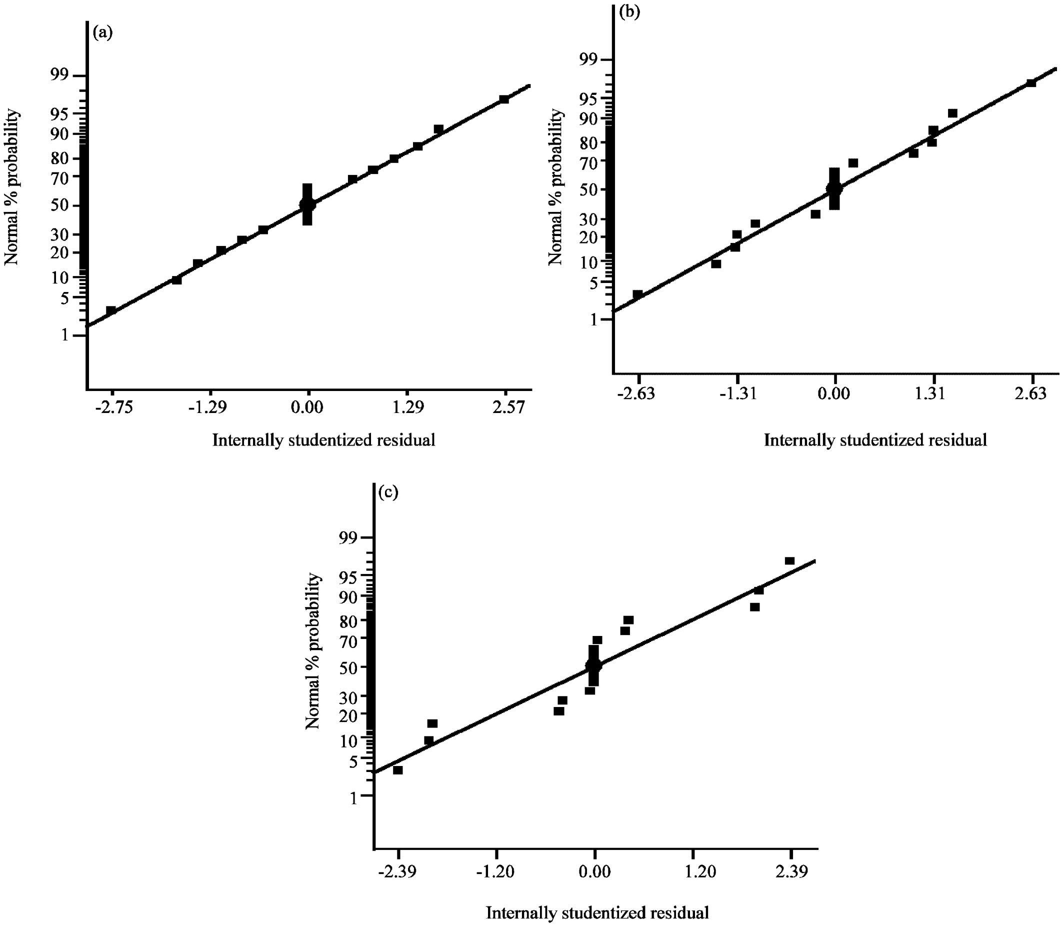

Fig. 1: Normal probability plot of residuals for (a) density, (b) relaxation and (c) durability of rice straw briquette

OPTIMIZATION OF THE RESPONSE VARIABLE

The determination of optimum operating conditions for the density of densified rice straw is aimed at obtaining high quality of solid fuel and minimum operating cost of production. One feature of high quality densified solid fuel is indicated by its density of higher than 1.0 g.cm^{-3}. Minimum operating cost can be achieved when the pressure is at minimum (X1<5000 psi), particle size is at maximum (X_2 >40 mesh) and moisture content is at maximum (X_3>10%) levels. The second order polynomial model to represent all response variables, namely the density, relaxation and durability of densified rice straw, were utilized to optimize the operating conditions of the independent variables. This model is merely valid in the selected experimental domain. In this study, pressure, particle size and moisture content were chosen in the range of 3000-7000 psi, 20-60 mesh and 5-15%, respectively.

Applying the desirability function (DF) method, the Design Expert software produced a number of 10 solutions of which each has a DF = 1, as shown in Table 6. However, among 10 solutions only three meet the predetermined criteria. The first is 3244.0 psi for pressure, 58.08 mesh for particle size and 14.63% for moisture content. The second is 3002.8 psi for pressure, 34.90 mesh for particle size and 7.77% for moisture content and the last is 3912.4 psi for pressure, 48.39 mesh for particle size and 12.77% for moisture content.

The first, second and the third acceptable solutions yield the density of densified rice straw of 1.1856, 1.0390 and 1.1378 g cm^{-3}, respectively. As the pressure of the third solution is the highest among the available solutions, coupled with somewhat higher in moisture content, this solution yields the highest density of the product and the highest operational cost. The first solution has a higher pressure and finer particle size in comparison to the second solution. As a consequence,

Table 6: Alternative solutions that meet DF=1 for optimization of process parameter

Solution No.	Pressure X_1 (psi)	Particle size X_2 (mesh)	Moisture content X_3 (%)	DF	Selection analysis	Judgment
1	5877.2	47.51	13.03	1	$X_1>5000$ psi	Rejected
2	5324.4	52.86	5.42	1	$X_1>5000$ psi;$X_3<10\%$	Rejected
3	3244.0	58.08	14.63	1	In accordance to criteria with a slight lower in X_2	Acceptable
4	6157.6	40.40	14.13	1	$X_1>5000$ psi;$X_2<40$ mesh	Rejected
5	3002.8	34.90	7.77	1	In accordance to criteria, with a slight lower in X_3	Acceptable
6	3912.4	48.39	12.77	1	In accordance to criteria, with a slight lower in X_2	Acceptable
7	6964.8	50.23	12.75	1	$X_1>5000$ psi	Rejected
8	5650.8	20.37	13.99	1	$X_1>5000$ psi; $X_2<40$ mesh	Rejected
9	3270.0	42.10	6.19	1	$X_3<10\%$	Rejected
10	5483.2	28.88	13.38	1	$X_1>5000$ psi; $X_2<40$ mesh	Rejected

the first solution requires higher operational cost than that of the second solution. Although the second solution produces the lowest briquette density, it gives the lowest operational cost. In addition, the density produced by the second solution is still higher than the minimum density of 1.0 g cm^{-3} required for producing a solid fuel briquette. It is therefore appropriate to select the second acceptable solution as the optimum.

CONCLUSION

A desirability function approach has been utilized to optimize the process variables of pressure, particle size and moisture content on the multi-response variables of density, relaxation and durability of solid fuel produced through a mechanical densification of rice straw biomass. The optimum conditions to produce solid fuels from rice straw biomass were obtained at a pressure of 3002.8 psi, the particle size of 34.90 mesh and raw material water content of 7.77%. With a minimum number of experimental runs, this technique is an efficient way for solution of optimization problems.

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