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Statistical inferential models for flexible polyurethane foam properties

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ABSTRACT

Models of foam properties in terms of raw material mix that forms the thrust of this paper is uncommon in the foam industry. Eighty sets of data on foam formulation and physical properties were collected from four certified companies using central composite design (CCD). To verify data reliability, ten of the mixes were randomly selected, physical properties value determined and compared to the existing formulation using t-statistics. Density (P_1); compression set (P_2); elongation (P_3); hardness-index (P_4) and tensile strength (P_5) were formulated as functions of the raw materials (toluene-di-isocyanate, water, amine, silicone-oil, stannous - octoate and ethylene-chloride.) using regression analysis. The mean density, compression-set, elongation, hardness-index and tensile strength of the data verification sample were 23.40kgm^{-3} , 8.46%, 170.01%, 150.13N and 117.54kNm^{-2} , respectively, while 23.26kgm^{-3} , 8.49%, 169.26%, 150.14N and 117.48kNm^{-2} for the existing foams. Both sets were not significantly different ($p < 0.05$). The functions P_1 , P_2 , P_3 , P_4 , P_5 with respective standard errors of 0.539, 0.097, 0.989, 0.987 and 0.513 were not significant ($p < 0.05$) while the coefficients of determination were 0.983, 0.898, 0.997, 0.976 and 0.896 respectively. These models are useful for making optimal decisions under various economic conditions for foam production. © 2011 Trade Science Inc. - INDIA

KEYWORDS

Polyurethane foam;
Density;
Compression set;
Elongation;
Hardness index;
Tensile strength.

INTRODUCTION

The behavior of most chemical processes is generally indicated by the states of output variables, which are dependent on the operating conditions and the adjustments made to the process^[1,2]. However, the productivity of such process may be quantified by a subset of these output variables; normally the specifications upon which the product is sold, e.g. purity, physical or chemical properties. These are usually the primary variables and are often difficult to measure on-line. The other outputs, like temperatures, flows and pressures

are called secondary variables and these are easily measured on-line^[1-3]. Inferential measurement systems are thus designed to overcome such measurement problems. The model thus generated can then be used to generate estimates of the difficult to measure primary output at the frequency at which the easily measured inputs and secondary variables are measured. If sufficiently accurate, the inferred states of primary outputs can then be used as feedback for automatic control and optimization^[3,4].

Inferential measurement systems essentially mimic what experienced process operators and engineers do

daily in running process plants, but are more advantageous as they can alleviate the problems of process complexities that lead to inconsistencies in human judgments¹. The procedure of building an inferential measurement system is essentially that of developing a model that relates a primary or quality variable to other, more easily measured secondary variables. Thus any modeling concept may be employed, including the development of first principles models or a statistical model which is data based modelling methods^[3,4].

The production of flexible polyurethane foam involves strong interrelationship of chemical reactions and process variables that are consequential on the final properties of foam^[5-8]. Coping with these complex interrelationships of variables involved in the production as well as meeting the required product qualities in a most profitable way has always been the dream of its manufacturers. Flexible polyurethane foams are produced by the controlled expansion of a gas during the polymerization process. They are designed to be open-celled which allow the free movement of gas within the foam cells. The properties of flexible polyurethane foams depend on both, the electrometric character of the polymer comprising the foams, as well as the geometry of the cells which is a function of the production conditions^[9-12].

The polyurethane technology is a one-shot system using new catalysts and silicone-based surfactants. In the one-shot process; the isocyanate, polyol, water, and other ingredients are rapidly and intensively mixed and immediately poured to carry out the foaming^[11,12]. Subsequent foaming reactions lead to the polymerization of the reacting mixture which gives the foam its integrity; and also release carbon-dioxide, which helps the entrained air bubbles to develop into foam cells. The formation of flexible polyurethane foams relies on a complex interaction between physical and chemical phenomena where there are no independent chemical or process variables^[13-15]. Therefore, the effect of altering a single variable such as a foam component or a process condition cannot be taken in isolation, since changing a particular parameter will affect the strong interplay which exists between the different variables^[16,17]. Several researchers have worked on the structure – property relationships of foam and have been widely reported in literatures^[18-22]. Villworcks^[23] in his own

TABLE 1 : Classification of set of quantities

Type	Description	Symbol	Units
Raw materials			
Inputs	Quantity of polyol	W ₁	kg
	Quantity of toluene di isocyanate (TDI)	W ₂	kg/kg polyol
	Quantity of water	W ₃	kg/kg polyol
	Quantity of amine	W ₄	kg/kg polyol
	Quantity of silicone oil	W ₅	kg/kg polyol
	Quantity of stannous octoate	W ₆	kg/kg polyol
	Quantity of ethylene chloride	W ₇	kg/kg polyol
Physical Properties			
Output	Density	P ₁	kgm ⁻³
	Compression set	P ₂	%
	Elongation	P ₃	%
	Hardness index	P ₄	kN
	Tensile strength	P ₅	kNm ⁻³

work presented some mathematical models for production of foam fortified with recycled foam particles.

In this paper, development of statistical models of the physical properties of foam in terms of raw material mix was executed. This work is of paramount importance to the foam industry as manufacturers will have a basis for foam formulations based on a combination of properties instead of formulation based on density which is the present practice^[24].

Development of polyurethane foam production models

Identification of the flexible polyurethane foam production quantities

Foam production processes were considered in four ISO- certified foam companies for the purpose of identifying the production inputs, outputs and parameters. After thorough interviews of the production personnel and review of relevant literatures, the input variables are the quantities of raw materials while the output variables are the physical properties of foam. The classification of these variables is presented in TABLE 1.

EXPERIMENTAL

Experimental design

The design was based on the fact that physical properties are functionally related to the specific raw mate-

Full Paper

rial mix. For this study, a centre point for the design was selected with raw material mix at a level of medium-density foam having quality standards. TABLE 2 lists the raw materials mix for the centre point. In multiple factor analysis, these raw materials (using the notations in TABLE 1) were transformed to ratios, which can be varied independently. Equations 1 to 6 were ratios selected as the x_i variables while the increment of the variation for each variable spaced around the centre point ratios are presented in TABLE 3.

$$x_1 = \frac{W_2}{W_1} \quad (1)$$

$$x_2 = \frac{W_3}{W_1} \quad (2)$$

$$x_3 = \frac{W_4}{W_1} \quad (3)$$

$$x_4 = \frac{W_5}{W_1} \quad (4)$$

$$x_5 = \frac{W_6}{W_1} \quad (5)$$

$$x_6 = \frac{W_7}{W_1} \quad (6)$$

where the x_i and the coded Y_i ratios are related by the following equations:

$$Y_1 = \frac{(x_1 - 0.4540)}{0.1} \quad (7)$$

$$Y_2 = \frac{(x_2 - 0.0370)}{0.01} \quad (8)$$

$$Y_3 = \frac{(x_3 - 0.0026)}{0.0005} \quad (9)$$

$$Y_4 = \frac{(x_4 - 0.0120)}{0.001} \quad (10)$$

$$Y_5 = \frac{(x_5 - 0.0024)}{0.0005} \quad (11)$$

$$Y_6 = \frac{(x_6 - 0.0455)}{0.02} \quad (12)$$

By substituting these equations, compositions were coded for solution of the multiple regression equation.

Data collection

Production data on flexible polyurethane foam production of diverse grades from the four ISO-certified

TABLE 2 : Formulation at the design centre point

Raw materials	Weight (g)
Polyol	100.00
TDI	45.40
Water	3.70
Amine	0.13
Silicone oil	1.20
Stannous octoate	0.24
Methylene chloride	4.55

Source: Field Survey, 2006

TABLE 3 : Experimental increments, values of coded levels

Raw materials	± Increment	Y_i coded levels				
		-2	-1	0	+1	+2
x_1	± 0.1000	0.2540	0.3540	0.4540	0.5540	0.6540
x_2	± 0.0100	0.0170	0.0270	0.0370	0.0470	0.0570
x_3	± 0.0005	0.0016	0.0021	0.0026	0.0031	0.0036
x_4	± 0.0010	0.0100	0.0110	0.0120	0.0130	0.0140
x_5	± 0.0005	0.0014	0.0019	0.0024	0.0029	0.0034
x_6	± 0.0200	0.0055	0.0255	0.0455	0.0655	0.0855

TABLE 4 : Descriptive statistics of raw materials and physical properties of foam

Raw materials	N	Minimum	Maximum	Mean	Standard deviation
Polyol (kg)	80	85.00	500.00	363.1250	202.1844
TDI (kg)	80	33.41	268.00	192.8025	110.3172
Water(kg)	80	2.52	22.00	15.7242	9.1052
Amine(kg)	80	0.19	4.40	1.2828	1.0867
Silicone oil(kg)	80	0.78	6.00	2.7879	1.9206
Stannous octoate(kg)	80	0.18	1.00	0.6773	0.3487
Methylene chloride(kg)	80	0.00	3.60	0.3000	1.0392
Physical properties	N	Minimum	Maximum	Mean	Standard deviation
Density (kgm ⁻³)	80	17.90	32.40	22.9917	4.1677
Compression Set (%)	80	4.00	15.52	8.5133	3.1616
Elongation (%)	80	139.00	274.00	167.0533	36.0811
Hardness index (kN)	80	97.70	168.00	150.9917	19.1242
Tensile strength(kNm ⁻³)	80	106.94	126.00	117.4314	4.7545

Source: Field survey, 2006

companies were gathered for examination. Data collected included foam formulations, costs of raw materials and physical properties. Using Karunakaran^[25] method, eighty (80) data sets were selected based on the experimental design (Central Composite Design (CCD) principle). This design was used to select data in order to minimize the effects of unexplained variability in the observed responses due to extraneous factors. A descriptive statistics of the data set selected is

TABLE 5 : Experimental verification of the foam production data collected

Raw materials	Experimental foam formulation (kg)									
	1	2	3	4	5	6	7	8	9	10
Polyol	500.00	500.00	500.00	500.00	500.00	500.00	500.00	500.00	500.00	500.00
TDI	270.00	270.00	270.00	270.00	270.00	265.00	295.00	305.00	185.00	190.00
Water	22.00	22.00	22.00	22.00	22.00	21.00	23.25	25.30	14.00	14.28
Amine	1.25	1.25	1.25	1.25	1.25	1.50	1.00	2.59	1.50	1.60
Silicone Oil	6.00	4.00	2.00	1.00	3.80	5.00	5.73	6.00	4.35	4.40
Stannous Octoate	0.90	0.90	0.90	0.90	0.90	1.00	0.96	1.18	1.25	1.25
MC	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	20.00
Physical properties										
Density(kg/m ³)	20.14 (20.10)	20.35 (20.40)	23.59 (23.60)	24.03 (24.00)	20.40 (21.00)	22.40 (22.60)	21.80 (22.00)	17.90 (18.00)	32.40 (32.30)	29.60 (30.00)
Compression Set (%)	6.89 (6.90)	6.89 (6.90)	13.10 (13.15)	15.52 (14.80)	6.89 (6.90)	7.14 (7.20)	7.10 (7.20)	9.60 (9.80)	4.00 (3.90)	7.80 (7.90)
Elongation (%)	160.10 (160.50)	160.00 (160.10)	147.00 (147.00)	140.00 (141.00)	160.04 (160.00)	189.00 (190.10)	162.00 (163.40)	139.00 (138.90)	274.00 (278.20)	161.50 (162.50)
Hardness Index(KN)	161.80 (161.80)	160.00 (160.00)	143.00 (143.20)	138.00 (140.00)	161.70 (161.80)	161.40 (162.00)	161.80 (162.10)	97.70 (98.20)	168.00 (170.00)	148.00 (150.20)
Tensile Strength(KN/m ³)	119.44 (119.50)	118.44 (118.40)	113.89 (114.00)	106.94 (107.10)	119.10 (118.90)	119.10 (119.40)	119.60 (120.00)	112.50 (114.00)	126.00 (128.10)	119.80 (120.00)

NB: Values in brackets are the experimental verification values for the physical properties

given in TABLE 4.

Data verification experiment

Foam samples of ten (10) production data selected by stratified random sampling from the data collected were produced and their physical properties were tested in one of the companies to verify the reliability of the data obtained which is in agreement with Karunakaran²⁵. The results obtained from this verification experiments were compared with those from the data using t-statistics to test for significant difference using the following hypotheses:

Null hypothesis H₀

There is no significant difference in the two samples

$$H_0: \beta_0 = \beta_1$$

Alternative hypothesis H₁

There is significant difference in the two samples,

$$H_1: \beta_0 \neq \beta_1$$

Model development by regression analysis

As pointed out by Karunakaran²⁵ and Jouhaud et al.^[26], regression analysis provides a conceptually simple method for investigating functional relationships among variables. Regression models were formulated for each of the five physical properties of foams as a function of

the raw materials mix using the data collected from the companies. A statistical package SPSS version 11.0 was used for this analysis. In multiple regression as in the present case, R², which is the square of the adjusted coefficient of determination and standard error are the indices. F statistics shows the significance of the overall model while the t statistics tests the significance of each of the variables of the model. All the six independent variables were included due to reaction requirements and theoretical consideration as pointed out by Chatterjee and Price^[27] and Argyrous^[28]. It was assumed that all the production process variables like raw materials and operation conditions were consistent.

RESULTS AND DISCUSSION

Experimental verification of foam production data

Ten foam samples were produced using the formulation of the randomly selected data and physical property test were carried out on the foam samples. The foam formulations, its accompanying physical properties from the companies and the results of the physical test conducted on the foam samples produced are presented in TABLE 5. The bracketed values indicate the experimental verification results of the physical properties. The summary of the t-test at 5% level of signifi-

Full Paper

TABLE 6 : Summary of t-test for experimental verification of the foam production data collected

Source of variation	t-value calculated	t-value critical	p- value (2 tail)	Remark
Density	-2.007*	2.262	0.076	No significant difference
Compression set	2.147*	2.262	0.073	No significant difference
Elongation	-2.251*	2.262	0.051	No significant difference
Hardness index	-2.165*	2.262	0.062	No significant difference
Tensile strength	-1.962*	2.262	0.081	No significant difference

*Significant level at $p < 0.05$

cance for experimental verification of the five critical physical properties as compared to the data collected is presented in TABLE 6.

The t-value for the comparison of density was -2.007 which indicated no significant difference at $p < 0.05$ between mean experimental validation value (23.30kgm^{-3}) and data (23.27kgm^{-3}) samples. The corresponding t-values for compression set, elongation, hardness index and tensile strength were 2.147, -2.251, -2.165 and -1.962, respectively, indicating no significant difference at $p < 0.05$ between experimental (8.46%, 170.10%, 150.93kN and 117.94kNm^{-2}) and data (8.49%, 170.08%, 150.87kN and 117.86kNm^{-2}) samples. The t-test showed that there were no significant differences in the sets of data obtained thereby upholding the null hypothesis which confirmed the reliability of the data for model formulation and analysis. It is generally expected that reliable data set should lead to reliable conclusions.

Estimation of the model parameters and adequacy test of the models

TABLE 7 shows the factors of the models, their parameter estimates and the statistics of the estimates for the best functions adopted, bearing in mind the reactivity of the system among other factors. Analyses were conducted to evaluate the adequacy and consistency of the models and the analysis of variance on the models are presented in TABLE 8. The analysis of variance calculated assessed how well the model represented the data. As shown on the TABLE 8, the F-value for the density model is 693.303 that is significant at 95% level implying good model fit. Similar significant values were also evaluated for compression set, elon-

TABLE 7: Estimated coefficients of the fitted model for properties based on t-statistic

	Model factors	Coefficients	t-Values	p-values
Density (P ₁)	Constant	-585.622*	-19.220	0.001
	x ₁	59.150*	4.701	0.003
	(x ₂) ⁻¹	1.305*	5.011	0.000
	x ₃	-111.452*	-4.834	0.002
	exp(-x ₄)	475.808*	17.691	0.002
	x ₅	6632.265*	3.250	0.002
	exp(-x ₆)	61.600*	8.166	0.001
N = 80		R ² = 0.983		
Compression set (P ₂)	Constant	-82.212*	-7.121	0.002
	x ₁	-214.419*	2.933	0.001
	x ₂	3247.530*	3.366	0.002
	x ₃	-283.371*	3.198	0.002
	(x ₄) ⁻¹	0.023*	20.829	0.000
	(x ₅) ⁻¹	-0.077*	2.808	0.000
	exp(x ₆)	103.561*	7.068	0.002
N = 80		R ² = 0.898		
Elongation (P ₃)	Constant	758.664*	12.094	0.004
	(x ₁) ⁻¹	-55.380*	-3.406	0.002
	x ₂	-7024.813*	-7.291	0.003
	x ₃	-602.856*	-5.981	0.002
	(x ₄) ⁻¹	-0.052*	-23.453	0.000
	(x ₅) ⁻¹	-0.326*	-17.756	0.000
	x ₆	-2812.097*	-99.182	0.003
N = 80		R ² = 0.997		
Hardness index (P ₄)	Constant	-9709.406*	11.094	0.047
	exp(x ₁)	-97.065*	-4.559	0.002
	exp(x ₂)	1266.549*	-3.432	0.001
	x ₃	-1430.642*	-19.604	0.004
	(x ₄)	2707.739*	19.072	0.003
	exp(x ₅)	8183.550*	-13.345	0.002
	exp(-x ₆)	492.117*	12.426	0.003
N = 80		R ² = 0.976		
Tensile strength (P ₅)	Constant	6364.025*	3.094	0.004
	x ₁	14.636*	3.436	0.003
	(x ₂) ⁻¹	1.225*	-4.726	0.001
	x ₃	-49.511*	-6.897	0.038
	exp(x ₄)	1172.897*	16.262	0.042
	exp(x ₅)	-7594.094*	-7.301	0.003
	exp(-x ₆)	142.906*	7.056	0.001
N = 80		R ² = 0.896		

*Significant at p value < 0.05 at 95% confidence interval

gation, hardness index and tensile strength on TABLE

TABLE 8 : Analysis of variance for physical properties models

Physical properties	Sources of variation	Sum of squares	d.f.	Mean square	F-value	Adjusted R ²	Standard error
Density	Regression	1306.062	6	217.677	692.303*	0.983	0.539
	Residual	22.953	73	0.314			
	Total	1329.015	79				
Compression Set	Regression	621.517	6	124.303	130.839*	0.898	0.097
	Residual	70.304	73	0.950			
	Total	691.821	79				
Elongation	Regression	98482.919	6	16413.820	4150.345*	0.997	0.989
	Residual	288.701	73	3.955			
	Total	98771.620	79				
Hardness index	Regression	27185.286	6	5437.057	613.333*	0.976	0.987
	Residual	655.993	73	8.865			
	Total	27841.279	79				
Tensile strength	Regression	1442.358	6	240.393	105.032*	0.896	0.513
	Residual	167.080	73	2.289			
	Total	1609.438	79				

*Significant level at $p < 0.05$

8. The density, compression set, elongation, hardness index and tensile strength curves respective standard errors of estimate were 0.539, 0.097, 0.989, 0.987 and 0.513, and were not significant ($p < 0.05$) while the coefficients of determination (R^2) were 0.983, 0.898, 0.997, 0.976 and 0.896.

Response equation for physical properties of flexible polyurethane foam

Following the adoption of the aforementioned standard procedures, equations 13, 14, 15, 16 and 17 were obtained for density, compression set, elongation, hardness index and tensile strength respectively.

$$59.15x_1 + 1.31x_2^{-1} - 111.45x_3 + 475.81\exp(-x_4) + 6632.27x_5 + 61.60_{\exp(-x_6)} - 585.62 = P_1 \quad (13)$$

$$-214.42x_1 + 3247.53x_2 - 283.37x_3 + 0.023x_4^{-1} - 0.077x_5^{-1} + 103.56_{\exp(x_6)} - 82.21 = P_2 \quad (14)$$

$$-55.38x_1^{-1} - 7024.81x_2 - 602.86x_3 + 0.05x_4^{-1} - 0.33x_5^{-1} - 2812.10x_6 + 758.66 = P_3 \quad (15)$$

$$-97.09_{\exp(x_1)} + 1266.55_{\exp(x_2)} - 1430.64x_3 + 2707.74x_4 + 8183.55_{\exp(x_5)} + 492.12_{\exp(-x_6)} - 9709.41 = P_4 \quad (16)$$

$$14.64x_1 + 1.23x_2^{-1} - 49.51x_3 + 1172.90_{\exp(x_4)} - 7594.09_{\exp(x_5)} + 142.91_{\exp(-x_6)} + 6364.03 = P_5 \quad (17)$$

Generally, in any process involving chemical reactions, it is always necessary to compute the mass balance of components of the reaction around the process. In order to obtain the actual mass of foam pro-

duced in any batch operation, the mass balance can be taken around the process box as follows:

$$x_1 - 1.44x_2 + x_3 + x_4 + x_5 = -0.018P_1 + 0.010P_2 + 0.001P_3 - 0.001P_4 + 0.005P_5 + 0.325 \quad (18)$$

CONCLUSION

In this study, mathematical models of foam properties in terms of raw material mix were developed as non linear multiple regression models. The mathematical functions developed for physical properties in term of raw material mix for polyurethane foam production adequately compared with conventional formulation practice with each having high coefficient of determination and insignificant standard error of the estimate at 5% level. The validity and sensitivity of the model over a wide range of changes in physical properties requirements show that the models will be useful for making optimal decisions under various economic conditions for foam production. Therefore, the application of this frame work by the foam manufactures is highly recommended.

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Full Paper

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