Modeling of Fiber-reinforced Phenolic Foam

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Abstract: A statistical predictive model is developed that describes the compression properties of phenolic foam reinforced with glass fibers. An analysis of variance is applied to determine the behavior of composite phenolic foam. The material variables used in the study are fiber length, fiber weight fraction and weight percentage of blowing agent. The responses analyzed are density, compressive modulus, and strength. The foam cell size distribution as a function of density is also studied. Comparison of the experimental results with statistical data indicates that the elastic properties of glass–fiber-reinforced phenolic foam do not depend on the fiber lengths used. Also, the results showed that the density and morphology of composite foam exhibit a strong influence on the responses of the model. The statistical approach has utility for predicting the effects of material variables on elastic properties of foams.

Key words: foams, glass fibers, modeling, mechanical properties.

1. INTRODUCTION

Phenolic foam, unlike most polymer foams, exhibits excellent flame resistance, low peak heat release rate (PHRR), and exceptional fire, smoke and toxicity properties (FST) [1, 2]. Furthermore, the low cost of phenolic foam relative to most other polymer foams, makes it well-suited to applications where fire resistance is critical, such as aircraft interiors, naval vessels, and building

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materials. However, structural applications of phenolic foam have been limited due to the friable and brittle nature of these foams. Fiber reinforcements have been introduced in phenolic foams to address the deficiencies in mechanical performance [3–5]. For example, Shen et al. [3] reported significant improvements in peel strength, and compression and shear properties of foam reinforced with glass and aramid fibers. Desai et al. [4] used hybrid reinforcements to tailor the properties of phenolic foam to specific requirements by varying fiber proportions. In both cases, several-fold improvements in compression and shear properties were demonstrated for foams by the judicious selection of hybrid reinforcement compared to foams reinforced with only glass or aramid fibers.

Several approaches have been employed to model the properties of composite foams, and these can be classified into three categories. In the first, the modeling is based on micro-mechanical methods which require precise representation of the internal structure of the material [2]. The second category involves a macro-modeling approach in which deformation mechanism based on analysis of constitutive equations derived from experimental data [6, 7]. The third category includes models derived from homogenization theories [8]. A good example of the modeling based on foam microstructure is the well-known theory of Gibson and Ashby, which offers the virtue of simplicity [1]. On the other hand, Saint-Michel et al. modeled polyurethane foams (PU) using the Christensen and Lo equations [7], an example of the macro-modeling approach. Both of these examples involve analysis of conventional unreinforced foams.

The presence of filler in composite foams raises special challenges for modeling mechanical behavior, and although there are a few examples in the literature, success has been limited. In one case, Siegmann et al. [8] studied the low- to medium-density foams filled with different fillers below 10 mm. They attempted to model the linear behavior of filled foams using Kerner equations in two steps. However, their analysis did not account for foam microstructure or for the influence of filler size on foam properties. In more recent work, Desai et al. [4] evaluated conventional models (parallel, series and Halpin-Tsai) for fiber-reinforced composites to predict the mechanical properties of hybrid foams. However, none of the models provided accurate fits to the experimental data, suggesting the need for a mechanistic model incorporating fiber length, loading, orientation, and foam density to predict the complex behavior of hybrid foams.

Different analytical tools have been employed to predict the elastic behavior of foams as a function of cell irregularity and relative density. For example, random Voronoi models have been constructed to simulate the linear elastic behavior of open cell foams [8]. Similarly, the dependence of elastic properties of the foams on the relative density has been simulated using finite element analysis (FEA) [9,10], providing good accuracy and utility [9]. FEA analysis was combined with experimental observations by Youssef et al. [11], when they obtained the microstructure of polyurethane foam directly form X-ray tomographic data and implemented a predictive finite element model of the mechanical behavior of foams.

An alternative approach to the mechanical modeling of foams involves the use of statistical methods. Statistical approaches greatly reduce the number of experimental iterations required compared to other modeling approaches while simultaneously yielding a wealth of information about multiple, interacting variables which influence the system. Several recent works demonstrate the utility of a statistical approach, known as analysis of variance (ANOVA) [12, 13]. For exmple, Alonso et al. [13] applied this approach to analyze the behavior of composite foams. They developed a statistical model to describe the compressive properties of glass–fiber-reinforced epoxy foams and investigated the effects of simple variables such as density, fiber weight fraction, and fiber length on the modulus and compressive strength. A 2^3 central composite design was applied, which included three main effects and three two-factor interactions. Such methods combine increased efficiency with good accuracy.

The aim of the present work is to determine the relationship between composition, final morphology, density, and elastic properties of fiber-reinforced composite phenolic foam. A statistical model is utilized to describe the behavior of glass–fiber-reinforced phenolic foams when length, weight fraction, and density are varied within a prescribed range.

2. EXPERIMENT

2.1. Materials and Foam Preparation

Phenolic foam samples were produced using a proprietary formulation [14] and patented technology [15], as described elsewhere [5]. Pentane was used as the blowing agent for foaming and polysulphonic acid (PSA) was the acid catalyst. Short glass fibers were used with chop lengths of 3, Modeling of Fiber-reinforced Phenolic Foam 3, 6, 9, and 12 mm and an average diameter of 11 μm (Lauscha Fiber International). The fibers included a silane sizing.

Synthesis of reinforced phenolic foams was carried out by blending the resin along with the surfactants, blowing agent, and catalyst in a high-speed, dual-axis mixer (Keyence, HM-101). After mixing, the glass fibers were incorporated into the mixture, which was blended for an additional 2 min. The reinforced phenolic foam was poured into a mold and held for 1 h at 80 °C. The phenolic foam was then cooled to room temperature before neutralizing overnight in a closed chamber of ammonia. A total of 14 different reinforced phenolic foam samples were synthesized with different amounts of blowing agent, glass–fiber weight fractions, and glass–fiber lengths.

2.2. Statistical Experimental Design

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Experimental design is widely used in the scientific and engineering communities for product improvement and optimization. Statistical approaches to experimental design are useful for extracting the most meaningful conclusions from experimental data. Only a brief discussion of statistical analysis of experimental design is presented here, with a focus on factorial experiment, although more detailed accounts appear elsewhere [16, 17].

The two aspects common to nearly all experimental investigations are the design of experiment and the statistical analysis of the data, and these two aspects are complementary. Choice of experimental design involves consideration of sample size (number of replicates), selection of suitable run order, and identification of randomization restrictions, if any [16]. The key task for selecting an appropriate model is identifying dependent and independent variables. One must then identify factors or variables which cause the response and estimate the magnitude of the response change. In the current work, a 2^k factorial design was implemented. The design of experiments was divided into two sets, as explained below.

A 2^3 factorial design was chosen for the first part of the design of experiments (Experimental Design I). The design involved three factors (variables), each with two levels of interest. High and low levels of the factors were designated as '+' and '-', respectively. Table 1 summarizes the design with all possible high/low combinations from the range of three factors, and Figure 1 represents graphically the ranges of the three factors considered in the 2^3 design. Each one of the numbers from the 'cube' represents an iteration of the experimental design. Additionally, in the first part of the design, one additional experiment is included which corresponds to the midpoint of the ranges studied for the independent variables. The iteration of the central point of the experimental design can be used as an estimate of the experimental variation [17].

Table 1. Matrix of two level factorial designs with one central point (Experiment Design I).

Run	Factor 1	Factor 2	Factor 3
1	0.0	0.0	0.0
2	-1.0	-1.0	-1.0
3	1.0	-1.0	-1.0
4	-1.0	1.0	-1.0
5	1.0	1.0	-1.0
6	-1.0	-1.0	1.0
7	1.0	-1.0	1.0
8	-1.0	1.0	1.0
9	1.0	1.0	1.0

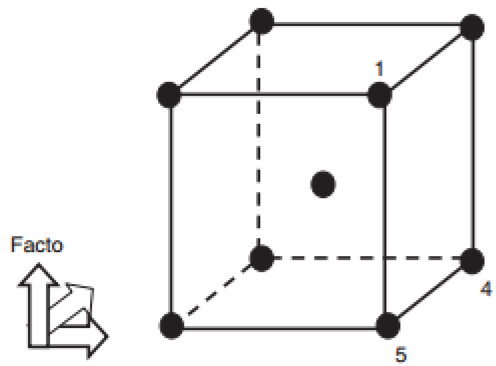

Figure 1. Cube for 2 cube design.

A 2^2 factorial design was chosen for the second part of design of experiments (Experimental Design II), in which, there were two factors (variables), each with two level of interest. Table 2 summarizes the design with all possible combinations (high/low) from the range of the two factors. As with the first design, one additional experiment is added which corresponds to the midpoint of the range studied for the independent variables. Figure 2 represents the range of the three factors considered in the 2^2 design. The various dependent variables (or responses in the present case) were density, fiber length, and fiber weight fraction, discussed in a later section.

Table 2. Matrix of two level factorial designs with one central point (Experiment Design II).

Run	Factor 1	Factor 2
1	1,0	-1,0
2	-1,0	-1,0
3	-1,0	1,0
4	1,0	1,0
5	0,0	0,0

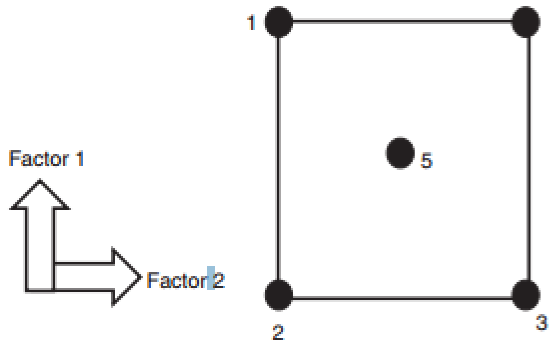

Figure 2. Cube for 2 square design.

The experimental designs were employed to fit a linear statistical model to the response results, with factor levels coded as 'low' or 'high'. Statistical software was used to perform ANOVA and multiple regression methods. For the 2^3 design, this yields a statistical model that can be expressed as:

$$Z = a_0 + \sum_{i=1}^{3} a_i X_i + \sum_{i=1}^{3} \sum_{j=1}^{3} a_{ij} X_i X_j \qquad (1)$$

where Z is the response considered, a_{ij} are the regression coefficients, and X_{ij} are the effects studied. After the regression coefficients are determined, an estimation or validation of the results is performed. Thus, each effect yields a p-value which signifies the significance of that effect, either low or high. For example, a higher p-value (>0.05%) indicates that an effect should be rejected in the variance analysis because that effect lacks significance in the model. In addition, for determining

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the fit of the model, the data must show a low scatter and an R^2 value (fraction of the variance) near 100%. The responses can be plotted as a contour map or a response surface (2D and/or 3D plot). Such graphs provide a visual depiction of the evolution of several factors that influence the response studied, allowing one to optimize the system conditions within the ranges considered.

For the 2^2 design, the statistical model is represented as:

$$Z = a_0 + \sum_{i=1}^{2} a_i X_i + \sum_{i=1}^{2}\sum_{j=1}^{2} a_{ij} X_i X_j \qquad (2)$$

2.3. Plan of Experiments

In the first part of the experimental work, nine phenolic foams were formulated with different fiber lengths (6 mm, 9 mm, and 12 mm), fiber weight fractions, and amount of blowing agent, resulting in composite foams with varied density. In addition, five composite phenolic foams were formulated with similar fiber lengths (3 mm), but with different fiber weight fractions and blowing agent content. Compression tests were performed on each of the specimens (parallel direction only), and the data were analyzed using the Gibson and Ashby model [1]. Cell size distributions for the fiber-reinforced composite foams were obtained from SEM images.

In the second part of this work, factorial design was employed to study the effects of the main process variables on composite foam manufacture. For Experimental Design I, a 2^3 central composite design was applied (9 runs: 2^3 + 1 central point), which included three main effects and three two-factor interactions. Similarly, for Experimental Design II, a 2^2 central composite design was applied (5 runs: 2^2 þ I central point), which included two main effects and two two-factor interactions. The variables studied included density (ϸ), fiber weight fraction (W), and fiber length (L) for Experimental Design I, and for Experimental Design II, the variables included density (ϸ)

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and fiber weight fraction (W). The measured responses included compression modulus (E) and the compressive strength (c).

Tables 3 and 4 summarize the experimental conditions employed and the results for Experimental Design I and II. Commercial software (Statgraphics Plus) was used to process the data. The model equations included first-order terms to describe main effects, and second-order terms for interactions, yielding the expression below for the response:

$$Z = a_0 + a_\rho \rho + a_L L + a_W W + \alpha_{\rho L} \rho L + a_{\rho W} \rho W + a_{WL} WL \qquad (3)$$

The nonsignificant effects were discounted by applying analysis of variance, which were included from the model regression, and to check the suitability of the model.

2.4. Compression Tests

Compression tests were performed in accordance with ASTM D1621. Specimens, 30 mm square by 25.4 mm thick, were placed between steel platens, and load was applied with a crosshead speed of 0.5 mm/min (0.02 in./min). Compressive modulus was determined from the steepest initial slope of the stress-strain curve, and strength was determined from the maximum load (in a range of strain <10%). At least five replicates were tested for each material, and the results were presented as the average value of all replicates.

Table 3. Foams with 6, 9 and 12 mm fibers.

Runs	Blowing agent (wt%)	Glass fiber loading (wt%)	Glass fiber length (mm)	Density (pcf)	Compression modulus (MPa) (Parallel)	Strength (MPa)	Avg. cell diameter (mm)
1	2.5	5.0	12.0	10.85	46.5	1.65	0.012
2	3.5	5.0	12.0	8.58	34.70	0.95	0.09
3	3.5	5.0	6.0	7.892	36.50	1.025	0.111
4	2.5	1.0	6.0	5.83	29.58	0.81	0.012
5	2.5	5.0	6.0	15.17	57.86	2.48	0.1
6	3.5	1.0	12.0	5.92	28.8	0.76	0.007
7	3.5	1.0	6.0	5.5	19.09	0.58	0.09
8	3.0	3.0	9.0	12.2	99.7	2.54	0.083
9	2.5	1.0	12.0	5.13	15.1	0.522	0.014

Table 4. Foams with 3 mm fibers.

Runs	Blowing agent (wt%)	Glass fiber loading (wt%)	Glass fiber length (mm)	Density (pcf)	Compression modulus (MPa) (Parallel)	Strength (MPa)	Avg. cell diameter (mm)
1	2.5	5.0	3.0	7.62	31.902	0.665	0.024
2	2.5	1.0	3.0	5.89	23.30	0.6075	0.15
3	3.0	3.0	3.0	5.21	19.5	0.38	0.083
4	3.5	5.0	3.0	5.62	21.052	0.4225	0.01
5	3.5	1.0	3.0	4.38	15.174	0.398	0.11

2.5. Scanning Electron Microscopy

Fracture surfaces of foam samples were examined with a scanning electron microscope (SEM, Cambridge 360) operated at 10 kV. The samples were submerged in liquid nitrogen to avoid deformation and structural damage to the foams, and samples were sputter coated with gold to impart electrical conductivity. SEM images were analyzed with image analysis software.

3. RESULTS AND DISCUSSION

3.1. Characterization of Unreinforced and Reinforced Foams

The density of phenolic foam increased sharply as the amount of blowing agent (alkane) was decreased in the formulation, as shown in Figure 3. Thus, the amount of blowing agent was critical to the density of phenolic foams. The compressive modulus (E) and compressive strength (c) are plotted as functions of foam density in Figure 4 (a) and (b). The figures indicate that the foam strength and modulus increase substantially with increasing foam density. The compressive modulus and strength can be fitted to a simple power law, as follows:

$$E = A\rho^{n} \qquad (4)$$

$$\sigma_c = B\rho^{m} \qquad (5)$$

where A and B are constants related to the physical properties of the foams, and n and m are density exponents related to the structure and deformation mechanics of the foams. Although, Equations (4) and (5) were developed for open-cell foams [1], these expressions have been widely used to predict other properties for closed-cell foams as well [13,18], and they accurately describe the compressive strength and modulus over the present density range. The corresponding value of coefficients in Figure 4 (a) and (b) are 0.95 and 0.97, respectively, while the values obtained for 'n' and 'm' are 1.5 and 1.9.

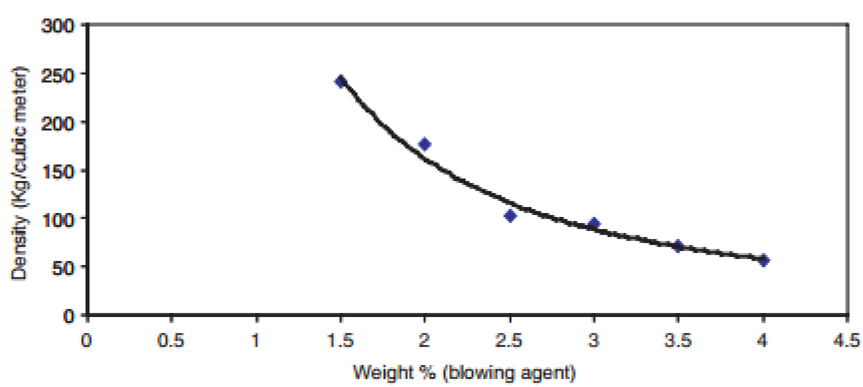

Figure 3. Variation of density with weight percentage of blowing agent for unreinforced foam.

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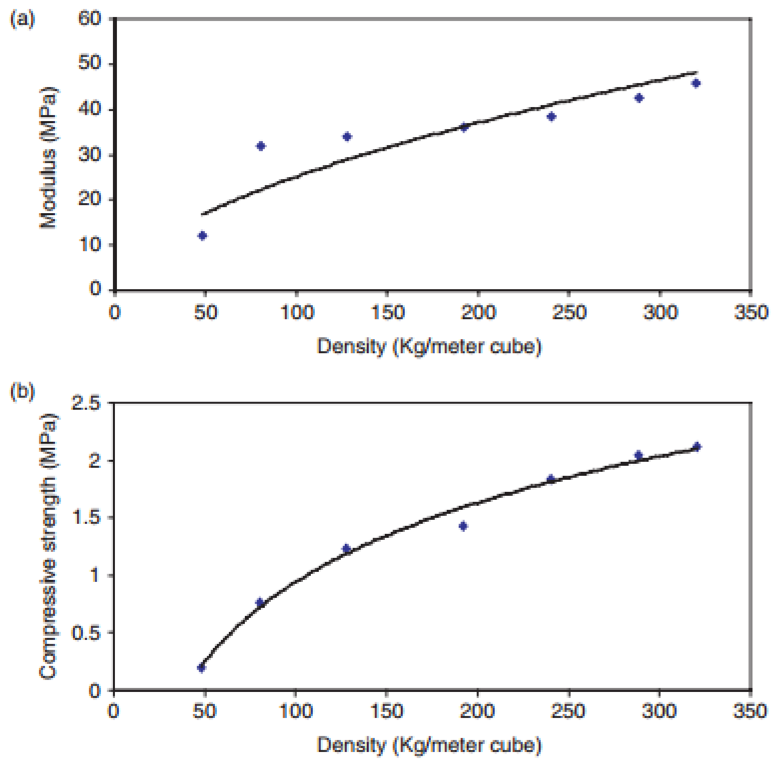

Figure 4. Variation of Modulus and Compressive Strength with Density of foam (unreinforced): (a) Modulus vs density of foam, (b) Compressive strength vs density of foam.

3.2. Effect of Fiber Length on Cell Diameter

The effect of fiber length on cell diameter for composite phenolic foam (with 1 wt% fiber and 2.5 wt% blowing agent) is shown in Figure 5. The average cell diameter for foams with 3 mm fibers is ~0.15 mm. However, for the foams with 6 and 12 mm fiber lengths the average cell is reduced by an order of magnitude, to 0.012 and 0.014 mm, respectively.

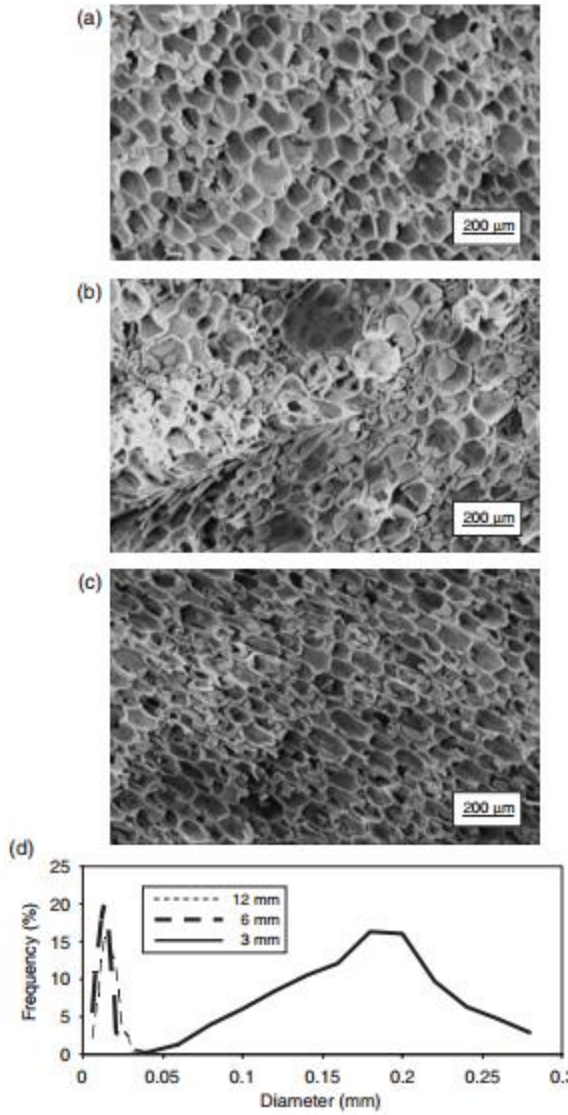

Figure 5. Effect of fiber length on cell diameter: foams with 1 wt% fibers and varied fiber length: (a) 12 mm, (b) 6 mm, (c) 3 mm, (d) frequecy (%) vs cell diamater.

We assert that the decrease in average cell size with increasing fiber length can be attributed in part to heterogeneous nucleation of cells on the fibers. In addition, significant fiber breakage occurs during processing, and the resulting fiber clustering interferes with cell expansion locally and compromises foam expansion globally. Longer fibers (6 and 12 mm) experience more extensive breakage compared to shorter (3 mm) fibers, resulting in greater reductions in average cell diameter. The phenomenon of fiber breakage is consistent with computer tomography (CT) observations by

Shen et al. [19], who reported that glass fibers added to phenolic foam were substantially reduced in length after mechanical mixing. In future work, we will perform CT imaging of foams studied in this work to support the theory. Similar observations were noted for foams with 5 wt% fiber and 2.5 wt% blowing agent using fiber lengths of 3, 6, and 12 mm. Figure 6 compares these foams with unreinforced foam at the same composition. The average cell diameter was 0.28 mm for unreinforced foams, while for composite foams with 3 mm, 6 mm, and 12 mm fibers, the mean cell diameters dropped to 0.24 mm, 0.007 mm, and 0.012 mm, respectively.

3.3. Effect of Fiber Weight Fraction on Cell Diameter

The average cell diameters for foams with 1 wt% fibers (Figure 5) were 0.15 mm, 0.012 mm, and 0.014 mm with fiber lengths of 3 mm, 6 mm, and 12 mm, respectively, whereas for foam samples with 5 wt% (Figure 6), cell diameters were 0.24 mm, 0.007 mm, and 0.012 mm, respectively. Thus, altering the fiber weight fraction (from 1 wt% to 5 wt%) had a neglible effect on the cell size distribution. These results are consistent with data for epoxy foams [13], where the size cell distribution was not significantly affected by the weight fraction of fibers.

3.4. Effect of Percentage of Blowing Agent on Cell Diameter

Table 3 summarizes the effect of blowing agent on cell size of composite phenolic foams. Runs 1 and 2 produced a large increase in average cell size (diameter). The increase in cell size was 650% when the amount of blowing agent was increased by only 1 wt%, while all other constraints (12 mm fiber length and of 5 wt% fibers) remained unchanged. However, the change in average cell diameter with amount of blowing agent was negligible when fiber lengths were 6 mm and 3 mm, as shown in Tables 3 and 4. For example, comparing runs 3 and 5, at a fiber length of 6 mm with 5 wt% fibers, there was no significant change in the average cell size with an increase in the amount of blowing

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agent. Thus, the amount of blowing agent affected cell diameter only when fiber lengths were long, while the cell size for foams with shorter fibers (6 and 3 mm) was essentially unchanged.

3.5. Statistical Model for Glass–Fiber-Reinforced Composite Phenolic Foams

The behavior of fiber-reinforced foams was evaluated on the basis of measured values of compressive modulus (E) and strength (c), as summarized in Tables 3 and 4.

Part A: Foams with 6 mm, 9 mm, and 12 mm Glass-fibers

In the first Experimental Design I, a factorial design was followed to study the effects of key process variables on the manufacture of glass– fiber-reinforced phenolic foams. A 2^3 central composite design was applied (9 runs), which included three main effects and three two-factor interactions. The variables studied were blowing agent (B), fiber weight fraction (W), and fiber length (L), and the respective ranges were 2.5– 3.5 wt%, 1–5 wt%, and 6–12 mm. The response measured for model development was density (þ).

The influence of blowing agent (B), fiber length (L), and fiber weight fraction (W) was studied through statistical design. The analysis of variance for density of the reinforced foams was carried out for a level of confidence of 95%, and is shown in Table 5.

In cases where the factor had a distribution F518.58% and p-values 40.05, the factor or factors were rejected because the effects presented a significance level 595%. Thus, as shown in Table 5, the factors L, BL, and WL were rejected from the analysis of variance. The new analysis of variance values for the density are shown in Table 6. All factors display a confidence level of 95%. The corresponding revised model describing the density for the composite foams (in the range studied), is given by:

$$\rho(\mathrm{kg/m^3}) = 12.5615 + 21.3525\,B + 71.9488\,W - 17.6675\,B\,W \quad (6)$$

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Table 5. Analysis of variance for density in Experimental Design I.

Source	SDQ	Mean square	df	Test F	p-values
B	2003.45	2003.45	1	1.44	0.3526
W	11486.7	11486.70	1	8.27	0.1026
L	238.06	238.06	1	0.17	0.7190
BW	2497.12	2497.12	1	1.80	0.3119
BL	784.08	784.08	1	0.56	0.5308
WL	150.16	150.16	1	0.11	0.7735
Error	2777.16	1388.58	2	–	–
Total	19936.8	–	8	–	–

SDQ: sum of squares, df: degrees of freedom.

The response surface of the density as a function of fiber weight fraction and amount of blowing agent is shown in Figure 7 (a). Statistical results indicate that the foam density is independent of fiber length.

In the Experimental Design II, variables studied included density (β), fiber weight fraction (W), and fiber length (L). The measured responses used for model development were modulus (E) and compressive strength (σ_c). The analysis of variance for composite foam modulus was carried out for a level of confidence of 95%, and is shown in Table 7. In this case, the factors L, βL, and WL had a distribution F<18.58% and p-values>0.05 and thus were rejected from the analysis of variance (Table 8). Thus, the corresponding revised model describing the modulus for the composite foams (in the range studied), is given by:

$$E(\mathrm{MPa}) = -81.8488 + 1.16321 \cdot \rho + 17.2642 \cdot W - 0.185056 \cdot \rho W \quad (7)$$

Statistical results indicate that the composite foam modulus is independent of fiber length, similar to results obtained in Equation (6). Figure 7 (b) shows the response surface of the modulus as a function of fiber weight fraction and density. Finally an operation similar to the one for evaluating modulus was performed for strength, and the factors L, βL, and WL were rejected from the analysis of variance. This is shown in Table 9, and the new analysis of variance values for compressive strength are shown in Table 10. The model obtained for compressive strength is found to be:

$$\sigma_c(\mathrm{MPa}) = -2.08192 + 0.0316814\,\rho + 0.19398\,W - 0.00314429\,\rho W \quad (8)$$

Table 6. Analysis of variance for density with significance effects in Experimental Design I.

Source	SDQ	Mean square	df	Test F	p-values
B	2003.45	2003.45	1	5.70	0.754
W	11486.7	11486.70	1	32.66	0.0046
BW	2497.12	2497.12	1	7.10	0.0561
Lack of fit	2542.8	2542.8	1	7.23	0.0547
Error	1406.66	351.67	4	–	–
Total	19936.8	–	8	–	–

Table 7. Analysis of variance for modulus in Experimental Design I.

Source	SDQ	Mean square	df	Test F	p-values
β	2049.61	2049.61	1	248.22 0.0040	
W	710.59 710.59	1	86.05	0.0114	
L	2.034	2.034	1	0.25	0.6688
βW	406.90 406.90	1	49.28	0.0197	
βL	5.20	5.20	1	0.63	0.5108
WL	6.87	6.87	1	0.83	04580
Error	16.51	8.26	2	–	–
Total	3236.07	–	8	–	–

SDQ: sum of squares, df: degrees of freedom.

Table 8. Analysis of variance for modulus with significance effects in Experimental Design I.

Source	SDQ	Mean square	df	Test F	p-values
β	2338.31	2338.31	1	454.88	0.0000
W	812.35	812.35	1	158.03	0.0001
βW	736.52	736.52	1	143.28	0.0001
Error	25.70	5.14	5	–	–
Total	3236.07	–	8	–	–

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Table 9. Analysis of variance for strength in Part A.

Source	SDQ	Mean square	df	Test F	p-values
β	2.723	2.723	1	372.82	0.0027
W	0.427	0.427	1	58.47	0.0167
L	0.00143	0.00143	1	0.20	0.7016
βW	0.107	0.107	1	14.69	0.0618
βL	0.00736	0.00736	1	1.01	0.4211
WL	0.0162	0.0162	1	2.22	0.2748
Error	0.0146	0.0073	2	–	–
Total	4.8897	–	8	–	–

SDQ: sum of squares, df: degrees of freedom.

The results obtained from the statistical model support the assertion that the compressive modulus and strength are independent of fiber length. This lack of dependence is attributed in part to the fiber breakage that apparently occurs during foam processing, as noted previously.

Table 10. Analysis of variance for strength with significance effects in Experimental Design I.

Source	SDQ	Mean square	df	Test F	p-values
β	3.131	3.131	1	363.27	0.0000
W	0.498	0.498	1	57.73	0.0006
ƤW	0.213	0.213	1	24.67	0.0042
Error	0.0431	0.0086	4	–	–
Total	4.8897	–	8	–	–

SDQ: sum of squares, df: degrees of freedom.

Table 11. Analysis of variance for density in Experimental Design II.

Source	SDQ	Mean square	df	Test F	p-values
B	790.453	790.453	1	8.65	0.2086
W	565.726	565.726	1	6.19	0.2433
BW	15.406	15.406	1	0.17	0.7520
Error	91.378	91.378	1	–	–
Total	1462.96	–	4	–	–

SDQ: sum of squares, df: degrees of freedom.

Previous work on similar composite foams showed that the fiber lengths were substantially reduced as a result of foam processing [19].

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The co-relations obtained above for density, compressive strength, and modulus leads to the next part of this work Experimental Design II in which statistical modeling was applied by varying the blowing agent and fiber proportion while keeping fiber length constant.

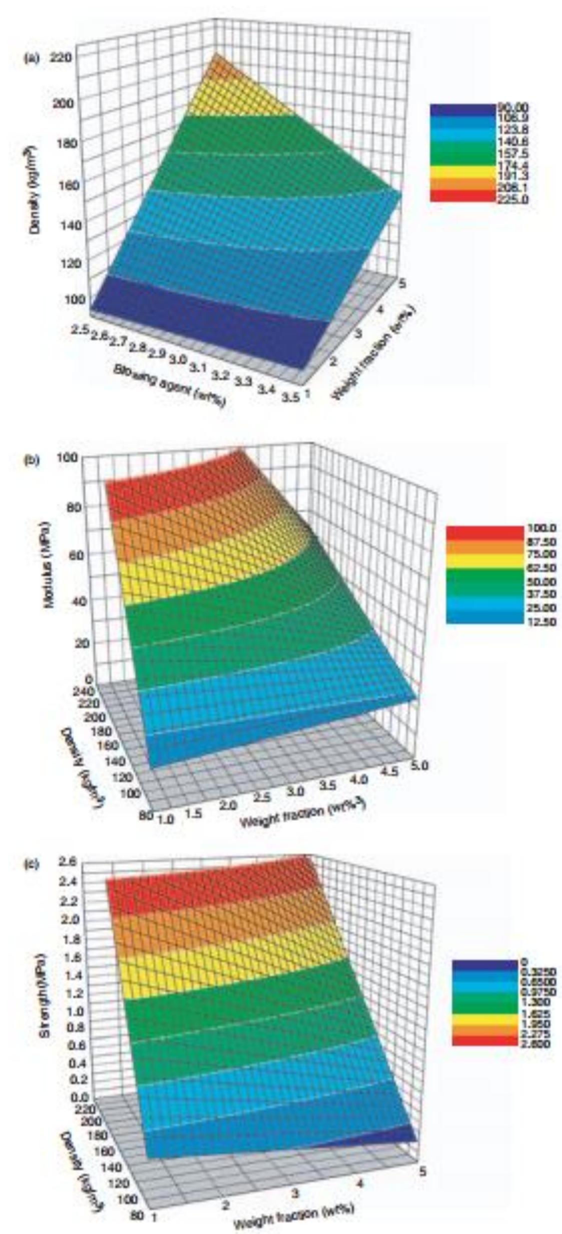

Figure 7. Response curve surfaces for 23 designs: (a) Variation of density as a function of amount of blowing agent and weight fraction of fibers, (b) Variation of modulus as a function of density and weight, (c) Variation of strength as a function of density and weight fraction of fibers.

Part B: Foams with 3 mm Glass Fibers

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A 2^2 central composite design was applied (5 runs), which included two main effects and one two-factor interaction. The variables were blowing agent (B), and fiber weight fraction (W), and the respective ranges were 2.5–3.5 wt%, and 1–5 wt%, while the response measured for model development was density (β). The influence of the variables (B and W) was studied through statistical design. The analysis of variance for foam density, carried out for a level of confidence of 90%, is shown in Table 11.

Table 12. Analysis of variance for density with significant effects in Experimental Design II.

Source	SDQ	Mean square	df	Test F	p-values
B	790.453	790.453	1	14.80	0.0614
W	565.726	565.726	1	10.60	0.0828
Error	106.784	53.392	2	–	–
Total	1462.96	–	4	–	–

In cases where the factor had a distribution F <9.29% and p-values >0.10, the factor was rejected. Therefore, the factor BW was rejected from the analysis of variance, as shown in Table 11, and the new analysis of variance values for modulus are shown in Table 12. All factors display a confidence level of 90%. The corresponding revised model obtained describing the composite foam density (in the range studied), is given by:

$$\rho(\mathrm{kg/m^3}) = 158.516 - 28.115\,B + 5.94625\,W \qquad (9)$$

Figure 8 (a) represents the response surface of density as a function of fiber weight fraction and amount of blowing agent. The 3D curve shown in Figure 8 (a) bears resemblance to the curve obtained earlier for the 2^3 design for density (Figure 7 (a)). In both these curves we observe a similar pattern where the density of the foams increases with increase in weight percentage of fibers whereas with an increase in amount of blowing agent the density of foams decreases.

Next, a factorial design was followed to study the effects of key process variables on foam manufacture. The variables studied were density (β) and fiber weight fraction (W), and the responses measured were modulus (E) and compressive strength (σ_c).

Analysis similar to the one performed in Part A was carried out using statistical design to study the influence of density (β) and fiber weight fraction (W). Table 13 shows the analysis of variance for modulus of reinforced phenolic foam. The factors presenting a significant level less than 95% (modulus) and 90% (strength) were rejected and the revised models describing the modulus and strength were:

$$E(\text{MPa}) = -8.09045 - 0.189865\,W + 0.335407\,\rho \qquad (10)$$

$$\sigma_c(\text{MPa}) = -0.169761 - 0.0411862\,W + 0.00856372 \cdot \rho \qquad (11)$$

The response surface of modulus as a function density and weight fraction of fiber is shown in Figure 8(b). The response surface resembles the surface of Figure 7 (b) obtained for the 2^3 design of composite foam. Also, the response surface of compressive strength as a function of density and weight fraction (Figure 8 (c)) resembles the surface obtained for the 2^3 design of composite foam (Figure 7 (c)). These figures depict the variation of compressive strength and modulus as a function of density and weight fraction of fibers. Also, for the foams studied, the density and compression modulus is not affected much by the length of fibers used. Tables 13–16 show corresponding data before and after lack of fit for modulus and strength.

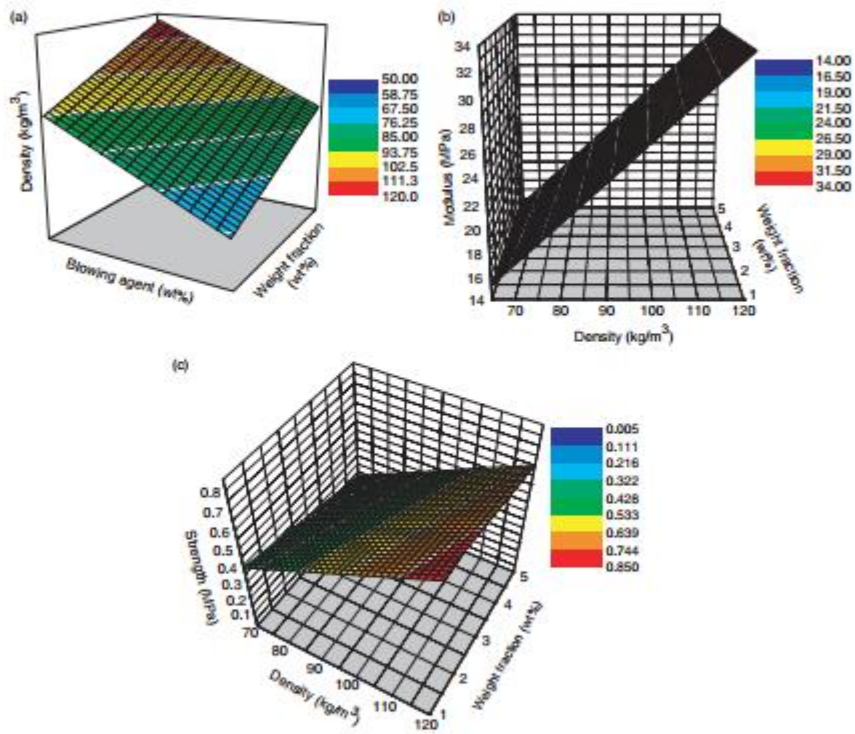

Figure 8. Response curve surfaces for 22 designs: (a) Variation of density as a function of amount of blowing agent and weight fraction of fibers, (b) Variation of modulus as a function of density and weight fraction of fibers, (c) Variation of strength as a function of density and weight fraction of fibers.

Shen et al. [19] studied similar composite foams and observed a large variance in the fiber length distribution (FLD) after foam processing, although the original fiber length was constant (6.4 mm). The majority of fibers in the final foam samples were 3 mm or less. The approach and experimental design can be used as a predictive tool to reduce the number of experimental iterations required to obtain foams with the specific strength and modulus values by simply varying the amount of blowing agent and the weight fraction of fibers. Finally, the modeling approach presented here can be applied to the design of more complex foams with hybrid fibers [4] to obtain specific ranges of mechanical properties.

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Table 13. Analysis of variance for modulus in Experimental Design II.

Source	SDQ	Mean square	df	Test F	p-values
W	0.313	0.313	1	8.60	0.2093
β	88.487	88.487	1	2429.89	0.0129
βW	0.0001	0.0001	1	0.00	0.9662
Error	0.0364	0.0364	1	–	–
Total	153.332	–	4	–	–

SDQ: sum of squares, df: degrees of freedom.

Table 14. Analysis of variance for modulus with significance effects in Experimental Design II.

Source	SDQ	Mean square	df	Test F	p-values
W	0.352	0.352	1	19.33	0.0480
β	100.878	100.878	1	5524.69	0.0002
Error	0.0365	0.0182	2	–	–
Total	153.332	–	4	–	–

SDQ: sum of squares, df: degrees of freedom.

Table 15. Analysis of variance for strength in Experimental Design II.

Source	SDQ	Mean square	df	Test F	p-values
W	0.0149	0.0149	1	5.72	0.2522
β	0.0577	0.0577	1	22.16	0.1333
βW	$1.274\,10^{-7}$	$1.274\,10^{-7}$	1	0.00	0.9955
Error	0.00260	0.00260	1	–	–
Total	0.0699	–	4	–	–

SDQ: sum of squares, df: degrees of freedom.

Table 16. Analysis of variance for strength with significance effects in Experimental Design II.

Source	SDQ	Mean square	df	Test F	p-values
W	0.0166		1	12.76	0.0702
β	0.0657		1	50.54	0.0192
Error	0.00260		2	–	–
Total	0.0699	–	4	–	–

SDQ: sum of squares, df: degrees of freedom.

4. CONCLUSIONS

Composite foams with different fiber loadings, fiber lengths, and amount of blowing agent were produced to obtain a broad range of densities, and the compressive strength and modulus were

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measured. While a simple power-law expression was sufficient to describe the mechanical properties of unreinforced foams, composite foam systems required a more sophisticated description. For this purpose, a statistical design approach was employed to analyze the mechanical properties of composite foams. This tool was used to investigate the effects of simple process factors (such as fiber loading, fiber length, etc.) on the mechanical properties of the composite foams.

Analysis of the experimental data and statistical design led to the conclusion that the compressive properties of the composite foams did not depend on the fiber length, at least over the range of lengths employed, and depended instead on other parameters, such as the amount of blowing agent and the fiber weight fraction.

Similar methods and approaches can be applied to other fiber-foam systems to reduce the number of experimental iterations and understand the effects of process variables on associated mechanical properties. For reinforced phenolic foams, it would be useful to extend this method to the analysis of more complex systems, such as 'hybrid' foams reinforced with more than one type of fiber. In particular, the statistical method employed here would be well-suited to investigating the effects of multiple process parameters on foam mechanical properties.

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