NC STATE
Wilson College of Textiles
JTATM
Journal of Textile and Apparel, Technology and Management

Volume 11, Issue 4, 2020

Application of Statistical Experimental Design for Optimization of Mechanical Properties of Protein Fiber Hybrid Composites

G. Rajkumar,
Department of Fashion Technology,
Kumaraguru, College of Technology, India

P. Shanmugapriya,
Department of Mathematics,
Ramakrishna Engineering College, India

ABSTRACT

Mechanical properties are important next to thermal properties especially a composite used in thermal insulation applications because it affects composite's life. Wool fibre composites is known for better thermal insulation properties and their poor mechanical properties act as a barrier to effectively utilize them in thermal insulation applications. Development of hybrid composites help to enhance the mechanical properties compared to single fibre composites. In the present work, hybrid composites were developed using wool and silk fibres by compression moulding technique. Polypropylene fibre was used as a matrix material. Also the manufacturing conditions of the composites were optimized using Box-Behnken experimental design with response surface methodology with respect to mechanical properties of the composites. The optimized manufacturing conditions arrived from the study were 180 °C temperature, 7.50 min time and 35 bar pressure. At this optimized manufacturing conditions the mechnaical properties of 30.66 MPa, 25.43 MPa and 23.67 KJ/m^2 were obtained for tensile strength, flexural strength and impact strength respectively for the protein fiber hybrid composites.

Keywords: Silk, wool, hybrid composite, tensile strength, flexural strength

1. Introduction

The global building and construction industry is growing rapidly from past few years. This industry consumes huge materials and energy, emitting greatest amount of CO_2 and producing huge amount of residues. The current research and innovation in this sector mainly focuses on the development of sustainable materials with energy efficient strategy. There is a huge demand for thermal insulation materials especially in the envelope-load-dominated buildings located in harsh climatic conditions.

Natural fiber composites find an immense place in the insulation field due to low environmental impact and low cost, which supports their potential across a wide range of applications. Several investigations were carried out with natural fibre composites and have shown that they are comparable with standard building materials [1]. For instance, lingo cellulosic fibres from flax [2], sunflower [3-5] and from date palm [6] have already been used as insulating materials in natural fibre composites.

Sheep wool is a natural protein fibre and good thermal insulator. It has comparable thermal conductivity to glass wool and polystyrene foam [7] which are used in building insulation. Sheep wool is used in civil engineering, for thermal and acoustic insulations [8]. Composites has been developed with wool fibres as reinforcement phase with different matrices (polymeric, earthern or cementitious) [9, 10]. Even though wool has better thermal insulation properties, their low mechanical properties restrict its applications.

Hybrid composites are the materials which have two or more fibres as reinforcement phase in single matrix and also the properties of the hybrid composites lie on the weighted sum of the individual components where there is a more balance between the advantages and disadvantages of individual parts [11]. Huge volume of literatures is available on the hybrid composites with natural base such as banana/sisal [12], sisal/silk [13], coir/silk [14] hybrid composites.

Silk is a natural protein fibre. They are biodegradable and highly crystalline with well aligned structure. The vast properties of silk fibres are mainly due to its high strength, elongation and stiffness consequently enhancing its mechanical property [15]. Shuttle-less loom silk weaving process generates huge amount of trimmed silk selvedge waste [16-18]. This is not utilized for any value added purposes except for making embroidery threads. Hence in the present work, an attempt is made to develop hybrid composites using wool and silk fibres retrieved from trimmed silk selvedge waste by compression moulding technique. The manufacturing conditions of the compression moulding are optimized with respect to mechanical properties of the composites using Box Behnken experimental design with response surface methodology.

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2. Experimental Work

2.1 Materials

In this study, wool and silk fibres were used as reinforcement phase and polypropylene fibres as matrix phase for the development of hybrid composites. Wool fibres were sourced from M/s Tirupati fibres, Panipat, India. Silk fibres used in this work are recycled fibres from shuttleless-loom silk selvedge waste which was obtained from from M/s Ethnic Fashions, Bengaluru, India. Polypropylene fibres were supplied by Zenith fibres, Vadodara, India. The physical properties of these fibres are given in Table.1.

2.2 Method

The reinforcement phase (wool and silk fibres) and matrix phase (polypropylene fibre) were blended in the ratio of 30:70 using a lab model blending machine. At this blend ratio better mechanical properties were obtained for protein fibre composites [11]. Webs were prepared using Trytex lab model carding machine. The blend was carded four times to get better fibre orientation [19, 20]. The web obtained from carding machine were compression moulded. Seventeen composites samples were developed as per the experimental design.

2.3 Measurement of mechanical properties

The tensile strength tests of the composites was carried out according to ASTM D 638-Type I in an Universal Testing Machine (Instron tensile tester model 3345) at a cross-head speed of 50 mm/min. Flexural strength tests were performed using 3-point bending method according to ASTM D790-03. The specimens were tested at the cross head speed of 50 mm/min. The flexural strength (FS) was determined using the following equation,

$$\mathrm{FS} = \frac{3PL}{2bt^2}$$

where P is the maximum load (Kgf), L is the span length (mm), b and t are width (mm) and thickness (mm) of the specimen respectively. Notched Izod impact tests were carried out as per ASTM D256 using pendulum impact strength tester. The standard specimen size is 64×12.7×3 mm^3, and the depth under the notch is 10.2 mm. The precise ten tested results were chosen for each type of test.

3. Results and discussion

The ranges of the experimental design for compression moulding are given in Table 2. The experimental design has 17 runs which were developed using Design expert software (trial version 8.1). The results of the mechanical properties of the composites for individual experimental runs are shown in Table 3.

Second order polynomial equations developed for tensile strength, flexural strength and impact strength are given in Table 4. Determination coefficient R^2, adjusted R^2, predicted R^2 and coefficient of variation (CV%) was determined to check the adequacy and accuracy of the developed models. The R^2 value indicates the proportion of the total variation in the response predicted by the models. The analysis of variance (ANOVA) for the responses tensile strength, flexural strength and impact strength is shown in Table 5.

Table 1. Physical properties of fiber

Fiber	Staple length (mm)	Fineness (Denier)	Strength (g/den)	Elongation (%)
Wool	45	2.1	1.5	26.30
Silk	75	1.1	4.5	13.20
Polypropylene	51	2.5	5.5	11.12

Table 2. Ranges of experimental design

Variables	Factors	Levels		
	X	-1	0	+1
Temperature (°C)	X_1	165	175	185
Time (min)	X_2	7	11	15
Pressure (bar)	X_3	35	40	45

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The R^2 values obtained for the mechanical properties of the hybrid composites were 0.9933, 0.9841 and 0.9921 which ensures a satisfactory fit of the model to the experimental data. The adjusted R^2 obtained were 0.9847, 0.9637 and 0.9819. The predicted R^2 values obtained were 0.9496, 0.9752 and 0.9698. It is observed that the relationships between tensile, flexural and impact strengths and process variables are accounted for the variability of the data satisfactorily. The values of adjusted- R^2 and predicted - R^2 indicated high correlation between observed and predicted values.

The ANOVA of the quadratic regression model demonstrated that the model is highly significant. This is evident from the Fisher *F*-Test (*F* value = 115.73 for tensile strength, 48.23 for flexural strength and 97.55 for impact strength) and a very low probability value (*P* = 0.0001). The coefficient of variation obtained was 3.67, 5.65, and 5.09 for the three responses. The adequate precision values obtained in this study were 40.88, 23.08 and 32.76 for the responses tensile strength, flexural strength and impact strength respectively which is higher than 4. This indicated the model is adequate.

The contour plots representing the interaction effect between the variables (a) time and temperature (b) pressure and temperature (c) pressure and time on the response tensile strength are given in Figure 1 (a) to (c). It is observed from Figure 1(a) that an increase in time with an accompanying increase in temperature resulted in an increase in tensile strength of the composites from about 9.45 MPa to 22.76 MPa. The lower viscosity of the matrix at high temperature improves the flow capability of the matrix which results in better fibre embedment. This leads to higher stability of the composite [21, 22]. The trend observed in Figure 1 (b) indicates that an increase in pressure and temperature yields increase in tensile strength of the composites. Figures 1 (a) and 1 (b) exhibited nearly linear relationship of variable temperature with the variables time and pressure in the form of straight line up to the medium level of temperature. At higher temperature, this become curvilinear or nonlinear. It is also noted in Figure 1(c) that a decrease in time and pressure yields increase in tensile strength of the composites. The variables time and pressure had shown non-linear relationship at all levels of the two variables.

Figures 2 (a) to (c) demonstrate the contour plots representing the effects of (a) time and temperature (b) pressure and temperature (c) pressure and time on flexural strength of the composites. Figures 2 (a) and 2 (b) have shown nearly linear relationship of variable temperature with the variables time and pressure in the form of straight line up to the medium level of temperature. At higher temperature, this becomes curvilinear or nonlinear. The variables time and pressure had non-linear relationship at all levels of the two variables as shown in Figure 2(c). The flexural strength of the composites increases with increase in temperature due to better wetting of the fibre in the matrix which leads to better consolidation of the parts at low moulding pressure [23, 24].

Table 3. Mechanical properties of the hybrid composites

Run	**X_1**	**X_2**	**X_3**	**Y_1**	**Y_2**	**Y_3**
	Temp (°C)	**Time (Min)**	**Pressure (Bar)**	**Tensile strength (MPa)**	**Flexural strength (MPa)**	**Impact strength (KJ/m^2)**
1	175	7	45	20.12	18.12	16.21
2	185	11	45	21.11	15.1	10.98
3	165	11	45	11.1	9.18	9.73
4	175	11	40	21.18	22.12	9.73
5	175	15	35	26.68	24.48	21.89
6	165	7	40	6.18	6.4	5.6
7	165	11	35	13.12	11.69	11.92
8	175	11	40	22.42	22.13	10.67

9	175	15	45	30.52	25.07	23.54
10	175	7	35	28.18	24.54	23.22
11	175	11	40	21.56	24.12	11.8
12	175	11	40	22.5	20.18	10.34
13	175	11	40	23.1	22.07	9.73
14	185	15	40	20.95	16.21	10.98
15	185	11	35	24.16	18.52	14.43
16	185	7	40	24.5	20.07	17.1
17	165	15	40	15.9	17.1	16.85

Table 4. Model equations for the mechanical properties of hybrid composites

Property	Model Equations for the mechanical properties
Tensile strength	$-2221.63+26.64X_1+6.46X_2-8.50X_3-0.08X_1X_2-0.005X_1X_3+0.14X_2X_3-0.07X_1^2+0.11X_2^2+0.09X_3^2$
Flexural strength	$-2742.50+30.56X_1+11.30X_2+0.16X_3-0.09X_1X_2-0.004X_1X_3+0.08X_2X_3-0.08X_1^2+0.07X_2^2-0.007X_3^2$
Impact strength	$-961.25+14.28X_1+7.02X_2-16.19X_3-0.10X_1X_2-0.006X_1X_3+0.10X_2X_3-0.03X_1^2+0.36X_2^2+0.19X_3^2$

Table 5. ANOVA for the mechanical properties of hybrid composites

Source	Response value		
	Tensile strength	Flexural strength	Impact strength
R^2	0.9933	0.9841	0.9921
Adjusted R^2	0.9847	0.9637	0.9819
Predicted R^2	0.9496	0.9752	0.9698
F - value	115.73	48.23	97.55
p - value	0.0001	0.0001	0.0001
CV%	3.67	5.65	5.09
Adequate precision	40.88	23.08	32.76

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The contour plots for the effects of (a) time and temperature (b) pressure and temperature (c) pressure and time on impact strength of the composites are indicated in Figure 3 (a) to (c). The interaction effect of the variable temperature with the variables time and pressure has given nonlinear relationship. An increment in impact strength was also observed with increase in temperature initially and later on decrement in impact strength was observed. Variables time and pressure in Figure 3 (c) had non-linear relationship at all levels of the two variables. The impact strength increased with increase in moulding time. The impact strength decreases with increase in molding time. A decrease in impact strength was observed with increase in molding pressure.

The interface between the fibre and resin may be good at the increased molding time due to the better melting of the resin and the application of high pressure will make the composite more stiff which reduces the impact strength [25].

The optimum conditions for the fabrication of wool/silk hybrid composites were arrived using the second order polynomial models developed in this work. The optimum values of tensile strength, flexural strength and impact strength of 30.66 MPa, 25.43 MPa and 23.67 KJ/m^2 was obtained at the temperature of 180 °C, at the time of 7.50 min and at the pressure of 35 bar. Figure 4 depicts the optimum conditions of the hybrid composites.

Triplicate experiments were conducted at optimized conditions and mean values of the experimental results were compared with the predicted values to validate the developed model. The mean values obtained from the experimental work were 30.21 ± 0.21 MPa, 26.22 ± 0.24 MPa and 23.03 ± 0.7 KJ/m^2 for tensile strength, flexural strength and impact strength respectively. The results obtained from the experimental work are closely related with the data obtained from the optimization analysis. This indicates the suitability of the developed quadratic model for effectively optimizing the mechanical properties of the wool/silk hybrid composites.

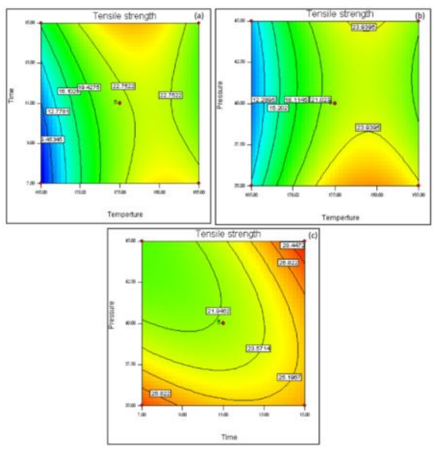

Figure 1. Contour plots of tensile strength showing the interaction effect between the variables (a) time and temperature (b) pressure and temperature and (c) pressure and time

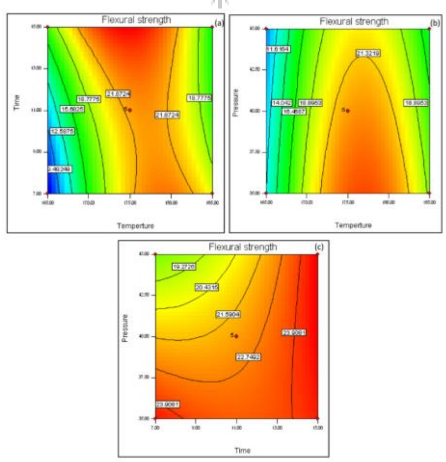

Figure 2 Contour plots of flexural strength showing the interaction effect between variables (a) time and temperature (b) pressure and temperature and (c) pressure and time

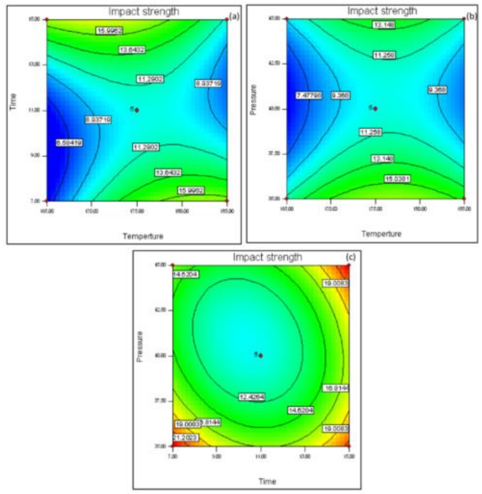

Figure 3. Contour plots of impact strength showing the interaction effect between the variables (a) time and temperature (b) pressure and temperature and (c) pressure and time

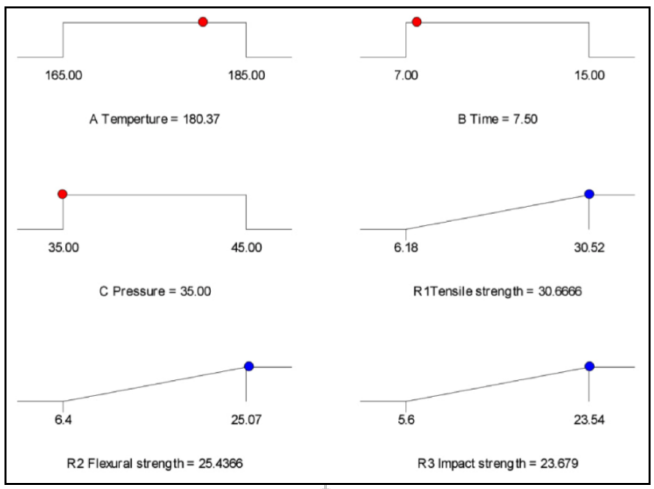

Figure 4. Optimum conditions for the mechanical properties of the hybrid composites

4. Conclusion

Wool and silk hybrid composites are developed using compression moulding technique. The manufacturing conditions of the composites are optimized with respect to its mechanical properties using Box-Behnken experimental design. It is observed from the results that the composites with better mechanical properties are obtained at 180 °C temperature, 7.50 min time and 35 bar pressure of compression moulding machine. The wool and silk hybrid composites developed from this work can be used as thermal insulation materials in building sector.

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