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Application of Design of Experiment (DoE) for Parameters Optimization in Compression Moulding for Flax Reinforced Biocomposites

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Abstract

Lately, widespread research on polymer composites that consist of natural fiber as reinforcement have been widely discussed. In this work, an attempt on optimizing the hot press forming process parameters using Response Surface Methodology (RSM) have been made to improve the mechanical properties of the woven flax/PLA composites. Three independent process variables, including moulding temperature, time and pressure were studied. Through the Box Behnken approach, a set of experiment runs based on various combination of compression moulding via Minitab 16 were established. As a results the optimum value for the variables of compression moulding technique parameters were 200°C, 3 min and 30 bar in order to yield 48.902 kJ/m^{-2}ofimpact strength.

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Keywords: Response surface methodology; Flax fibre reinforced composites; Compression moulding

1. Introduction

In recent years, there are growing interests in the use of natural fibers as reinforcement in fiber reinforced plastics in order to minimize the environmental problem associated with disposal of non-biodegradable polymer.[1–3]

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As a natural fiber, flax fiber has been regarded as alternative material to synthetic fiber because it is abundant, low cost environmental friendly and good specific strength properties.[3] Recent developments in natural polymer composites have heightened the need to improve its mechanical properties and one of the solution is the utilization of woven fibre as reinforcement. Woven fibre promotes higher fibre packing and offers good toughness through its mechanical interlocking.[4]

Compared to many of the forming process for polymer composites, compression moulding is frequently used as manufacturing process that requires specific design, especially for woven natural fibre. In compression moulding process, selection of suitable parameters is important in order to yield the optimum composites products. Among the compression moulding parameters, moulding temperature, pressure, and heating time are the most important parameters that influence the mechanical properties.[5]Traditonally, most researchers follow the classical experimental method where by changing one parameter at a time while keeping others factors at fixed level.[6,7]However, due to its time-consuming nature and cost issues, response surface methodology (RSM) have been identified to be efficient statistical tool because of less number of experiments required, thus experiments are faster and more effective.[2,6,8]

Although, a considerable amount of literature has been published on applying RSM on compression moulding process however optimisation of compression moulding process for flax fiber reinforced PLA has not been reported yet. In this works, the Box Behnken design approach was employed on woven flax/PLA aiming improve the mechanical properties through optimization series of the compression moulding factors via Minitab16.

2. Experimental

2.1. Materials

The material studied is a commercially available Biotex 40% flax/PLA woven fabric supplied from Composites Evolution based in Chesterfield, United Kingdom. The material comes in preconsolidate sheet form that have 2x2 twill woven weaves style as shown in Fig.1. The flax/PLA preconsolidated sheet has a measured average thickness of 0.80 mm. The composite woven fabric properties as given by the manufacturer are summarized in Table 1.

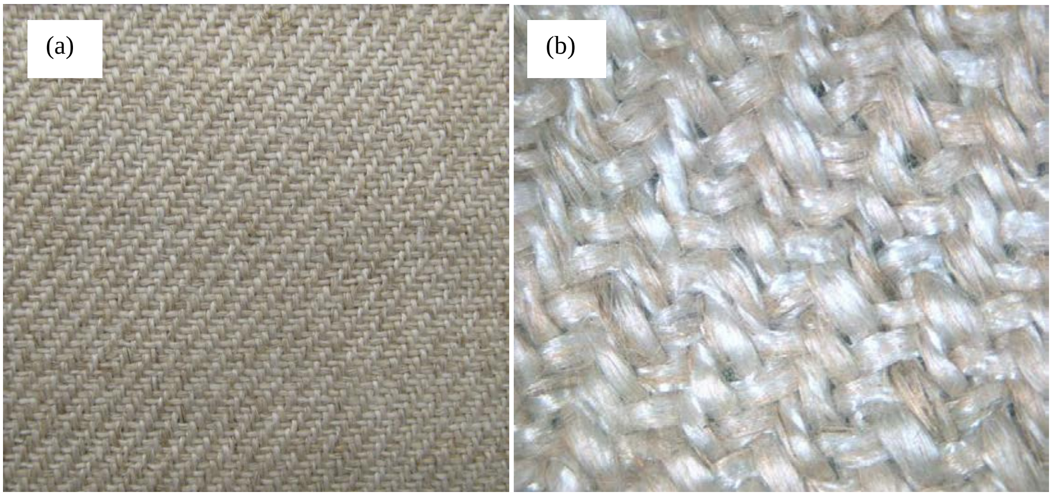

Fig.1. Woven flax/PLA 2x2 twill fabric at (a) visual observation; (b) 10x magnification.

Table 1. Composite properties.

Property	Woven flax/PLA composites
Fibre content by volume (%)	40
Density (g/cm^3)	1.33
Weave style	2x2
Warp count (ends/cm)	7
Weft count (picks/cm)	7
Weight per square meter (g/cm^2)	400

2.2. Compression Moulding process

Compression moulding branded as (model GT-7014-H50C) is the processing machine used to fabricate all samples. A total of 9 layers of woven fabrics were prepared to achieve a composite thickness 3 mm. The pre-preg was covered with Teflon and pressed into a stainless steel mould of 150 mm x 120 mm square cavity, possessing a thickness of 3 mm, to produce a composite sheet of dimensions of 150 mm x 120 mm x 3 mm. The composites are moulded with variation of hot pressing parameters; with moulding temperature range from 180-200^{o}C and moulding pressure between 30-70 bar and moulding time for 3 -9 min.

2.3. Impact test

Charpy V-notch impact test was conducted on the samples complying with ASTM D 6110-10. At least 5 specimens of each set were executed to ensure accuracy. The results are reported as impact strength where it is defined as energy lost per unit cross-sectional area at the notch (kJ/m2)

2.4. Experimental Design and Optimization

The optimization study of compression moulding process was carried out according to the box behnkan design tool (BBD) of Response Surface Methodology (RSM) using Minitab 16 software for selected 3 factors. The three factors selected were moulding temperature, time and pressure as shown in Table 2. Equation 1 shows the generalized response surface model. Total of 15 experimental points consist of 12 factorial design runs and 3 replicates at the centre points were shown in Table 3:

$$Y = b_0 + \sum_{i=1}^{3} b_i X_i + \sum_{i=1}^{3} b_{ii} X_i^2 + \sum_{i=1}^{2} \sum_{j>1}^{3} b_{ij} X_i X_j + \qquad \ldots \qquad (1)$$

Where Y was the response variable (impact strength), $b_0$was the constant, ε was the residual (error) term, bi was the linear coefficients, b_{ii} was the quadratic coefficient, b_{ij} was the interaction coefficient and X_i was the dimensionless coded independent variable.

Table 2. Parameters for experimental design.

Factor	Variation Levels		
	Low (-1)	Medium (0)	High (+1)
Moulding Temperature (°C)	180	190	200
Moulding Time (min)	3	6	9
Moulding Pressure (bar)	30	50	70

Table 3. Arrangement of experimental design.

Test Number	Variables (Coded value)			Variables (Real value)		
	Moulding Temperature (°C)	Moulding Time (min)	Moulding Pressure (bar)	Moulding Temperature (°C)	Moulding Time (min)	Moulding Pressure (bar)
1	-1	0	1	180	6	70
2	0	0	0	190	6	50
3	0	1	-1	190	9	30
4	-1	0	-1	180	6	30
5	1	0	-1	200	6	30
6	1	-1	0	200	3	50
7	0	-1	-1	190	3	30
8	1	1	0	200	9	50
9	0	0	0	190	6	50
10	-1	1	0	180	9	50
11	0	0	0	190	6	50
12	1	0	1	200	6	70
13	0	1	1	190	9	70
14	0	-1	1	190	3	70
15	-1	-1	0	180	3	50

3. Results and Discussion

Table 4 represents the experimental results obtained from RSM on charpy V-notch impact strength at different variation combination of compression moulding parameter.

Table 4. Experimental design and results of PLA/flax Biocomposites.

Test Number	Independent Variables			Response
	Moulding Temperature (°C)	Moulding Time (min)	Moulding Pressure (Bar)	Charpy notched Impact Strength (kj/m^2)
1	180	6	70	42.23
2	190	6	50	51.49
3	190	9	30	39.84
4	180	6	30	41.18
5	200	6	30	42.09
6	200	3	50	37.70
7	190	3	30	41.10
8	200	9	50	47.60
9	190	6	50	44.10
10	180	9	50	28.69
11	190	6	50	40.10
12	200	6	70	46.12
13	190	9	70	42.10
14	190	3	70	37.85
15	180	3	50	35.12

3.1. Analysis of Variance (ANOVA)

ANOVA can be useful in determining the significant of each linear, quadratic and interaction term in the models through probability value (P-value). The significant of quadric models obtained in Table 5 for all terms were determine by P values less than 0.05.[1,6–8]Allindependent and interaction terms were significantly influenced impact strength.However for quadratic terms, only X_3^2 linear square term were insignificant to the response. A test of lack-of-fit is determine to ensure the fitness of the models and to predict the variation. It evidence that the lack of fit test was not significant with P-value more than 0.05.[9,10] While in Table 6, the adjusted determination coefficient value (R-sq(adj)) for woven flax/PLA was near to 1. Besides, plot of residuals versus predicted, normality plot, histogram plot and randomness of the residual suggested in Fig.2 proved that the residuals follow normal distribution and the variances are at constant. Overall, this indicates that RSM approaches are effective tools for the optimization of compression molding parameter and developed models was a good fit and satisfactory.

Table 5. Analysis of variance for woven flax/ PLA impact strength.

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	9	8	333.028	37.003	158.9	0
Linear	3	82.01	82.01	27.337	117.39	0
Temperature	1	10.881	10.881	10.881	46.73	0.001
Time	1	62.161	62.161	62.161	266.94	0
Pressure	1	8.968	8.968	8.968	38.51	0.002
Square	3	23.185	23.185	7.728	33.19	0.001
Temperature*Temperature	1	11.708	9.755	9.755	41.89	0.001
Time*Time	1	11.188	11.399	11.399	48.95	0.001
Pressure*Pressure	1	0.289	0.289	0.289	1.24	0.316
Interaction	3	227.833	227.833	75.944	326.13	0
Temperature*Time	1	204.776	204.776	204.776	879.36	0
Temperature*Pressure	1	16.606	16.606	16.606	71.31	0
Time*Pressure	1	6.452	6.452	6.452	27.7	0.003
Residual Error	4	1.164	1.164	0.233		
Lack-of-Fit	3	0.543	0.543	0.181	0.58	0.681
Pure Error	1	0.621	0.621	0.31		
Total	13	334.192				

Table 6. Estimated Coefficient of woven flax/PLA.

Term	Coef	SE Coef	T	P
Constant	39.5733	0.2786	142.039	0
Temperature	-1.1663	0.1706	-6.836	0.001
Time	-2.7875	0.1706	-16.338	0
Pressure	-1.0587	0.1706	-6.206	0.002
Temperature*Temperature	-1.6254	0.2511	-6.472	0.001
Time*Time	1.7571	0.2511	6.997	0.001
Pressure*Pressure	0.2796	0.2511	1.113	0.316
Temperature*Time	-7.155	0.2413	-29.654	0
Temperature*Pressure	2.0375	0.2413	8.444	0
Time*Pressure	1.27	0.2413	5.264	0.003

S = 0.482564 PRESS = 10.0925 R-Sq = 99.65% R-Sq(pred) = 96.98% R-Sq(adj) = 99.02%

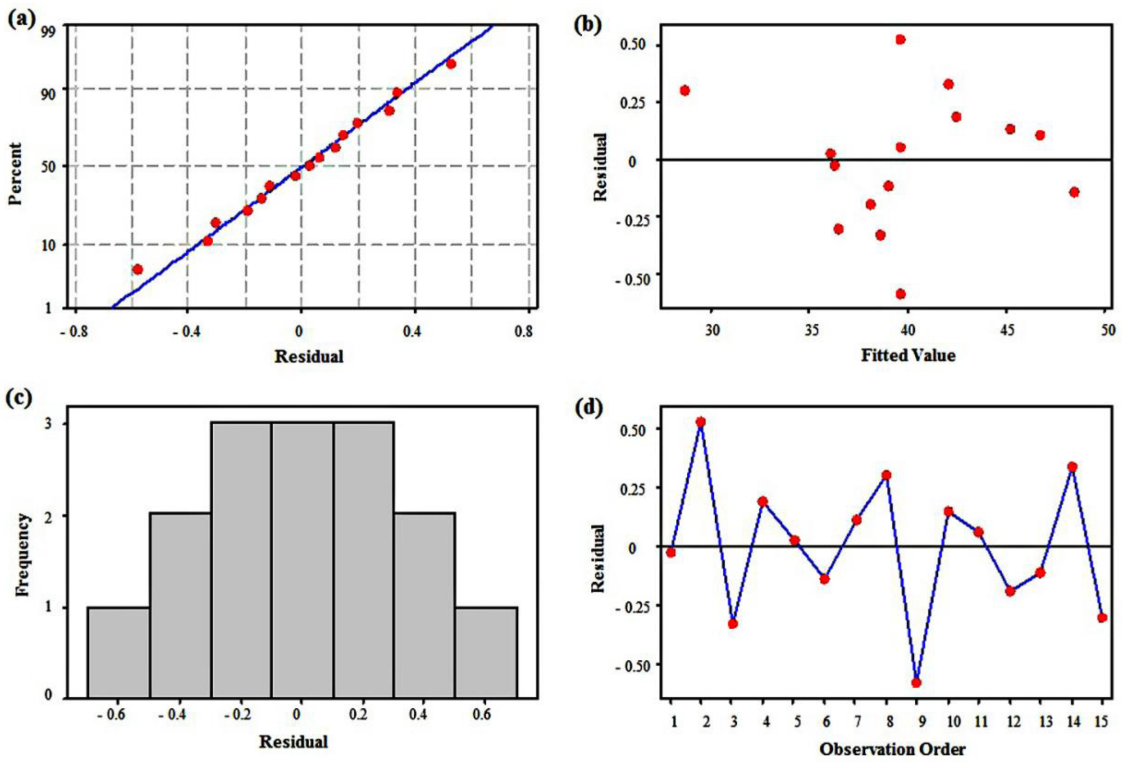

Fig.2. Residual plots of impact strength for woven flax/PLA composite.

The regression coefficients provided in Table 6 describe the impact strength value in terms of coded parameters Based on that, the full quadratic model was developed as shown in Equation 2. It was noted that insignificant effect was neglected to form the best-fitted regression equation. Thus, the proposed equation of the model were presented in the following.

$$Impact\ Strength\ (kj/m2) = 39.745 - 1.166X_1 - 2.788X_2 - 1.059X_3 - 1.647X_1^2 + 1.736X_2^2 - 7.155X_1X_2 + 2.037X_1X_3 + 1.270X_2X_3 \quad (2)$$

Where X_1, X_2 and X_3 are the coded values of the independent variables, moulding temperature (X_1), moulding time (X_2) and moulding pressure (X_3) respectively.

3.2. Contour Plot

Fig.3 (a) represents the response contour plot for the optimization for impact strength as function of mould pressure and mould temperature. It was observed that sample flax/PLA is more prominent to yield higher impact strength at lower pressure and temperature. Fig.3 (b) shows combined effect of holding time and pressure on impact properties. The trend showed that combination of shorter moulding time with lower mouldingpressure wills result in higher impact strength. Fig.3 (c) shows interaction between molding temperature and time against the impact strength. The increase in temperature with accompanying decrease in moulding time will results in increase impact strength of the composite.

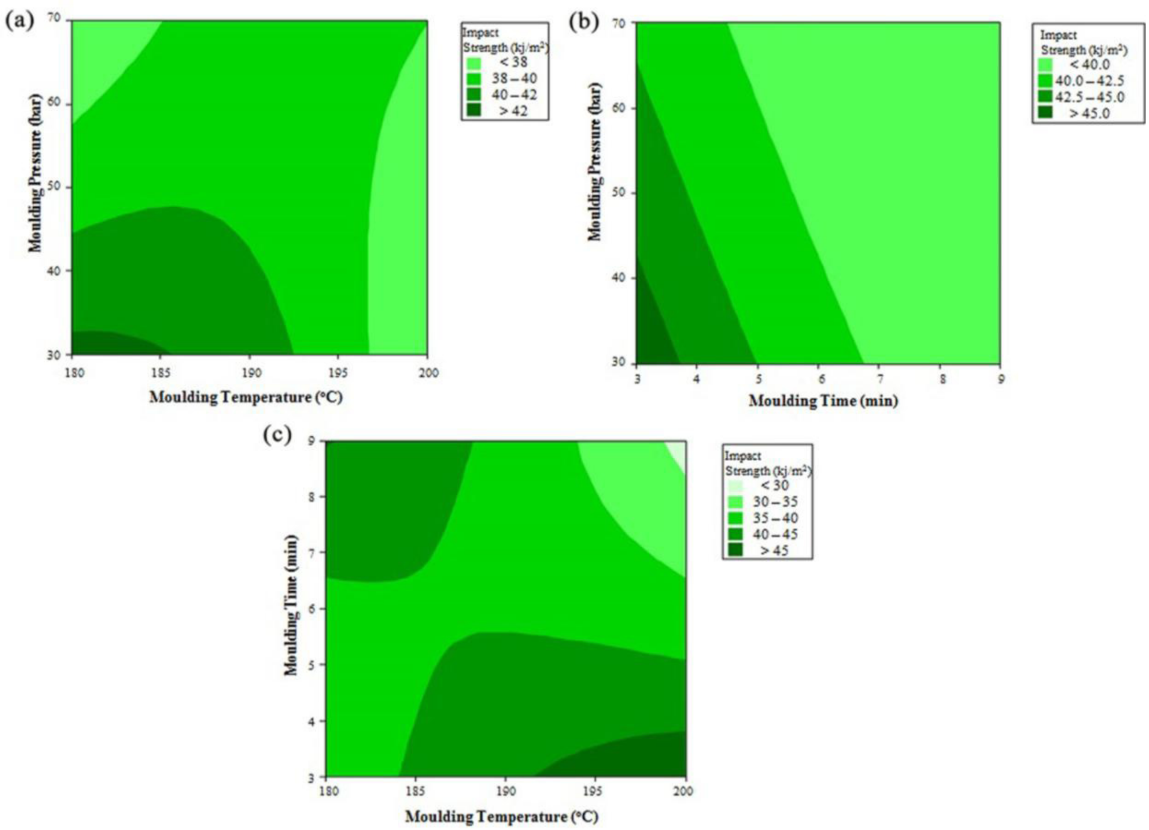

Fig.3. Contour plots of impact strength for woven flax/PLA composite.

3.3. Optimisation responses and verification the model

The optimization of compression moulding process for the woven flax fiber reinforced PLA composites, were predicted by Minitab 16 softwareusing the second order regression equation (equation 2). In this case, the predicted optimum combination variables were 200°C, 3 min and 30 bar with a desirability value near to 1 as shown in Table 7

Table 7. Optimum condition for maximum of impact strength.

Moulding Temperature X_1 (°C)	Moulding Time, X_2 (min)	Moulding Pressure, X_3 (bar)	Lower Target (kj/m^2)	Upper Target (kj/m^2)	Impact Strength (kj/m^2)	Desirebility, D
200	3	30	30.00	50.00	48.902	0.945

4. Conclusion

The investigation of this work dealt with the application of RSM using a Box Behnken design (BBD) to optimise the mechanical properties of woven flax/PLA. The ANOVA data showed that the variables are affected the impact strength significantly. The inter-actions of variables also have significant effects on impact strength. The optimal set of processing parameters for woven flax/PLA was found to be moulding temperature (200^{o}C), moulding time (3 min) and moulding pressure (30 bar), to achieve the maximum impact strength of 48.902 kJ/m^{-2}.

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