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Investigation of Polypropylene-Grass Composite Using Split-Split Plot Experimental Design

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ABSTRACT

This study investigates the effects of process parameters in the production of polypropylene-grass composite using split-split plot experimental designs. The experimental values of mechanical properties of polypropylene-grass composite obtained at barrel temperature ranging from 210^oC to 310^oC were input into the analytical design of split-split plot design to obtain its numerical design. The numerical experimental designs were evaluated for sum of squares for process parameters and their interactions. The results obtained were presented on ANOVA Table. F-test was used to compare statistical significance of the factors of the total deviation. The results of the calculated Fisher's Ratio (F_{cal})atsignificant value of 0.05 for the process parameters such as percentage by volume of material, barrel temperature, material type and their interactions ranges from -29.46 to 6.25 respectively. The results obtained shows that these process parameters contribute significantly to the production of polypropylene-grass composite.

1. Introduction

Optimal designs for variance components model have been discussed fairly in experiment that were ran in a completely random order. Most of the published work dates back to the 60's and 70's and have been restricted to specific models namely, one-way random model, the two-way crossed classification random model and the two way nested model [1]. Injection moulding is a very complex process and its process variable like barrel temperature, injection pressure, the material flow rate, mould temperature and flow pattern usually influence the properties of polymeric materials. A qualitative analysis of the influence of these factors in this case barrel temperature on the mechanical properties of a moulded part will be helpful in gaining better insight into the presently used processing methods [2]. Furthermore, inadequate investigation of process parameters and their interactions in produced composites is a major problem in polymeric industries. This may be due to a poor scientific understanding of the moulding process based on the complexities of the process containing multiple variables affecting the final part [3].

Chunping et al. [4] carried out a study aimed to model fundamental bonding characteristics and performance of wood composite. In their work, mathematical model and a computer simulation model was developed to predict the variation of inter-element contact during mat consolidation. The mathematical predictions and the computer simulations agree well with each other. Their results showed that the relationship between the inter-element contact and the mat density was highly nonlinear and was significantly affected by the wood density and the element thickness.

Goos and Vandebroek [5]; Loeza-Serrano and Donev [6] constructed D-optimal design for variance components estimation in a three stage crossed and nested classification for experiments that includes both crossed and nested factor in the same model, no assumption of a complete random model was made. Moreover, the designed experiment for variance component estimation was based on the linear mixed effect model. Ankenman et al. [1]. Aviles and Pinheiro [7] examined the experiments that have complete randomization order of runs which was not feasible or might be too expensive to use when performed. They concluded from their study that the use of split-plot designs and models are feasible, efficient and cheap. Split plot designs were initially developed by Fisher in 1925, it was used in agricultural experiments and it was basically the modified form of randomized block designs [8]. These designs were used in situations where complete randomization of runs within block is not possible. These designs were used widely in industrial experiments such as experiments where one set of factors may require a large amount of experimental materials (whole plot factors), while another set of factors might be applied to smaller experimental materials (sub plot factors). Harless et al. [9] examined mechanical properties of composite panels wet which depend on the density variations that occur through the pan thickness. They then proposed an analytical tool to predict density profile as a function of the manufacturing processes. A multi-layer description of the density and moisture gradients resulting from the felting process provided input for the mode Inputs for the pressing process included plate temperature and press closing rate. The model the developed simulated the physical and mechanical processes that occur in the press and mat system. Edelugo [10] examined the effect of reinforcement combination on the mechanical strength of glass reinforced plastic under increased temperature conditions while Ranjusha et al. [11] investigated the Talc-filled polypropylene random copolymer with different processing parameters respectively. From their result, a proof stress of 25N/mm^2 was obtained. Adeyemi and Adeyemi [12] developed empirical formulas, based on the diffusion model and the drying data (i.e moisture ratios, with drying times) of the composite from sawdust were computed and presented for various curing temperature and at different percentages of hardener resin addition. There were a number of studies on moisture transfer modeling of wood, but no systematic study for moisture transfer in board with respect to usage or storage. The physical parameters of four kinds of composite boards were determined by them in their study. The unsteady-state diffusion coefficients and surface emission coefficients of moisture in boards were separated in one experimental period by using the method of linear regression. Then the moisture transfer processes in board were analyzed by using Finite Element Method (FEM), and the moisture absorption processes of four kinds of boards were observed experimentally. By comparing the computed results with the experimental results, it showed that the error was within 10%. Therefore, they came to the conclusion that the processes of moisture transfer in composite can be described by using FEM. Olodu [3] examined the effect of process parameters such as temperature in the production of polypropylene-grass composites using split plot experimental design, his results shows that temperature contributes significantly to the production of composites in polymeric industries.

This study therefore focused on the investigation of polypropylene-grass composite using split-split plot experimental design.

2. Materials and Method

2.1. Preparation of Grass

The harvested grass was washed and soaked with dilute sodium hydroxide (NaOH) of concentration 0.10mol/dm^3 for 6 hours to ensure effective bonding between the grass and polypropylene. The grasses were first air dried in the sun and later transferred to an oven and dried at 105^{o}C. It was continuously monitored until moisture content of about 4$\pm$0.2% was obtained

[12]. The grass was grinded to granules using crushing machine. The grinded grass was screened to a particle size of 300μm diameters using vibrating sieve machine.

2.2. Mixing, Compounding and Production of Composite

Polypropylene (PP) was mixed with grinded grass in the ratio of 20:80, 30:70, 40:60, 50:50, 60:40, 70:30 and 80:20 percentages by volume respectively. The prepared Polypropylene-grass composite was blended in a cylindrical container until a homogenous mixture was obtained in the composition. The homogenous mixture of the composite was feed into the hopper of injection moulding machine and was produced at various barrel temperature ranging from 210^{o}C to 310^{o}C respectively [3]

2.3. Evaluation of Polypropylene-Grass Composite for Mechanical Strength

The produced composite was evaluated for mechanical strength (tensile strength, proof stress, percentage elongation and flexural strength) using Equations1 to 4 respectively [2].

$$\text{Tensile strength} = \frac{\text{Maximum Load}}{\text{Original Cross – Sectional Area}} \quad (1)$$

The original cross-sectional area of the specimen is 18.9mm^{2}.

$$\text{Proof stress} = \frac{\text{Force at yield}}{\text{Cross – Sectional Area}} \quad (2)$$

The Cross-sectional area of specimen =18.9 mm^{2}

Hence, proof stress $= \frac{\text{Force at yield}}{18.9} \text{N}/mm^2$

$$\text{Percentage (\%) Elongation} = \frac{\text{Extension}}{\text{Gauge Length}} \text{x } 100\% \quad (3)$$

$$\text{EI} = \frac{\text{P}L^3}{48\text{y}} \quad (4)$$

Where y is the deflection in mm, P= Load, L= Length of test specimen

2.4. The Split-Split Plot Design

The split-split plot design which is an experimental design was used to investigate the interaction between material type, percentage by volume of material, and barrel temperature on the mechanical properties of the produced composite. In simple terms, a split-split plot experiment is a blocked experiment, where the blocks themselves serve as experimental units for a subset of the factors. It involves the use of the analytical and numerical designs.

2.5. The F-test

The F-test was used for comparing the factors of the total deviation (using Equation 5). The statistical significance was tested by comparing the F test statistic.

$$\text{F} = \frac{\text{Variance between treatments}}{\text{Variance within treatments}} = \frac{\text{MS}_{\text{Treatments}}}{\text{MS}_{\text{Error}}} = \frac{\text{SS}_{\text{Treatments}/(\text{I}-1)}}{\text{SS}_{\text{Error}}/(\text{nT}-1)} \quad (5)$$

2.6. The Interactive Model Developed for PP-Grass Composite

Equation 6 shows the Interactive model developed and is depicted as:

$$X_{ijkl} = \mu + \gamma_i + \beta_j + \delta_l + y_k + \gamma\beta_{ij} + \gamma y_{ik} + \beta y_{jk} + \gamma\delta_{il} + \beta\delta_{jl} + y\delta_{lk} + \gamma\beta y_{ijk} + \gamma\beta\delta_{ijl} + \gamma y\delta_{ikl} + \beta y\delta_{jkl} + \gamma\beta\delta y_{ijkl} + \varepsilon_{ijkl} \quad (6)$$

2.7 Hypothesis

The null hypothesis with its alternative were formulated for the PP composite as follows:

Null Hypothesis(H_o): The percentage by volume of material, material type, barrel temperature and their interactions contributes significantly to the mechanical properties of the composite produced at α-value of 0.05.

Alternate Hypothesis (H_1): The percentage by volume of material, material type, barrel temperature and their interactions does not contributes significantly to the mechanical properties of the composite produced at α-value of 0.05.

3. Results and Discussion

Table 1 shows the effects of barrel temperature on mechanical Properties of PP-Grass Composite. Figure 1-4 shows the graph of Effects of Barrel Temperature on tensile strength, proof stress, Percentage Elongation and flexural strength respectively. Table 2 shows Anova results for effects of barrel temperature on PP-Grass Composite.

Table 1: Effects of Barrel Temperature on Mechanical Properties of PP-Grass Composite

			TEMPERATURE (^oC)										
MECHANICAL PROPERTY	PERCENTAGE BY VOLUME OF PP	PERCENTAGE BY VOLUME OF GRASS	210	220	230	240	250	260	270	280	290	300	310
TENSILE STRENGTH (N/mm^2)	80	20	11.20	15.90	20.41	25.64	27.90	29.10	30.37	30.77	31.15	30.90	31.07
	70	30	11.85	16.56	20.96	26.15	28.42	29.65	30.92	31.33	31.71	31.35	31.51
	60	40	15.80	20.50	24.40	29.60	31.85	33.07	34.33	34.73	35.11	34.75	34.92
	50	50	14.50	19.10	23.52	28.76	31.00	32.23	33.50	33.90	34.30	33.96	34.10
	40	60	13.80	18.52	22.93	28.10	30.36	32.59	33.82	34.23	34.62	34.28	34.45
	30	70	13.25	17.96	22.46	27.68	29.93	31.15	32.41	32.83	33.22	32.88	33.05
	20	80	12.30	17.00	21.00	26.85	29.08	30.30	31.56	31.97	32.29	31.96	32.12
PROOF STRESS (N/mm^2)	80	20	20.30	22.10	27.81	28.87	29.38	29.89	30.41	30.92	31.38	30.84	29.16
	70	30	21.45	23.23	24.84	25.92	26.41	26.91	27.42	27.93	28.39	27.84	26.16
	60	40	27.60	29.40	31.00	32.06	32.56	33.07	33.59	34.09	34.54	33.98	32.30
	50	50	26.34	28.14	29.75	30.82	31.30	31.83	32.34	32.85	33.31	32.76	31.08
	40	60	25.50	27.30	28.91	28.98	29.47	29.98	30.51	31.04	31.48	30.95	29.27
	30	70	24.60	26.40	28.00	29.10	29.63	30.12	30.65	31.15	31.61	31.05	29.82
	20	80	23.42	25.23	26.84	27.91	28.42	28.95	29.48	29.98	30.43	29.90	28.22
PERCENTAGE ELONGATION (×100%)	80	20	0.52	0.62	0.73	0.78	0.87	0.86	0.87	0.96	1.00	0.96	0.86
	70	30	0.65	0.74	0.85	0.90	0.98	0.97	0.98	1.08	1.13	1.08	0.97
	60	40	0.75	0.85	0.94	1.00	1.10	1.10	1.10	1.20	1.25	1.20	1.09
	50	50	0.68	0.78	0.88	0.94	1.04	1.04	1.03	1.14	1.18	1.14	1.00
	40	60	0.59	0.68	0.78	0.83	0.93	0.92	0.93	1.04	1.08	1.04	0.90
	30	70	0.46	0.57	0.68	0.73	0.83	0.84	0.84	0.93	0.98	0.93	0.82
	20	80	0.40	0.52	0.63	0.68	0.78	0.78	0.77	0.88	0.92	0.88	0.76

AVERAGE DEFLECTION (mm)	**80**	**20**	**5.08**	**5.12**	**5.15**	**5.17**	**5.19**	**5.21**	**5.17**	**5.16**	**5.14**	**5.11**	**5.07**
	70	**30**	**5.31**	**5.34**	**5.38**	**5.40**	**5.42**	**5.44**	**5.39**	**5.35**	**5.33**	**5.30**	**5.26**
	60	**40**	**4.87**	**4.89**	**4.93**	**4.95**	**4.96**	**5.00**	**4.96**	**4.95**	**4.94**	**4.91**	**4.87**
	50	**50**	**5.39**	**5.42**	**5.46**	**5.48**	**5.50**	**5.52**	**5.48**	**5.48**	**5.45**	**5.42**	**5.37**
	40	**60**	**5.57**	**5.61**	**5.65**	**5.67**	**5.69**	**5.71**	**5.67**	**5.66**	**5.64**	**5.61**	**5.55**
	30	**70**	**5.76**	**5.80**	**5.84**	**5.87**	**5.91**	**5.92**	**5.88**	**5.87**	**5.85**	**5.82**	**5.76**
	20	**80**	**5.93**	**5.98**	**6.02**	**6.05**	**6.07**	**6.09**	**6.04**	**6.03**	**6.00**	**5.96**	**5.90**
FLEXURAL STRENGTH X10^2(N/mm^2)	**80**	**20**	**40.20**	**39.95**	**39.70**	**39.50**	**39.38**	**39.25**	**39.54**	**39.63**	**39.80**	**39.98**	**40.30**
	70	**30**	**38.50**	**38.25**	**38.00**	**37.83**	**37.72**	**37.60**	**37.90**	**38.20**	**38.35**	**38.54**	**38.84**
	60	**40**	**42.00**	**41.76**	**41.50**	**41.32**	**41.20**	**40.91**	**41.20**	**41.25**	**41.40**	**41.60**	**41.95**
	50	**50**	**37.90**	**37.68**	**37.44**	**37.27**	**37.15**	**37.02**	**37.28**	**37.33**	**37.50**	**37.71**	**38.05**
	40	**60**	**36.70**	**36.46**	**36.20**	**36.02**	**35.90**	**35.78**	**36.05**	**36.12**	**36.26**	**36.47**	**36.80**
	30	**70**	**35.50**	**35.26**	**35.00**	**34.82**	**34.60**	**34.50**	**34.78**	**34.84**	**34.95**	**35.14**	**35.48**
	20	**80**	**34.45**	**34.20**	**33.96**	**33.80**	**33.68**	**33.56**	**33.85**	**33.92**	**34.08**	**34.30**	**34.66**

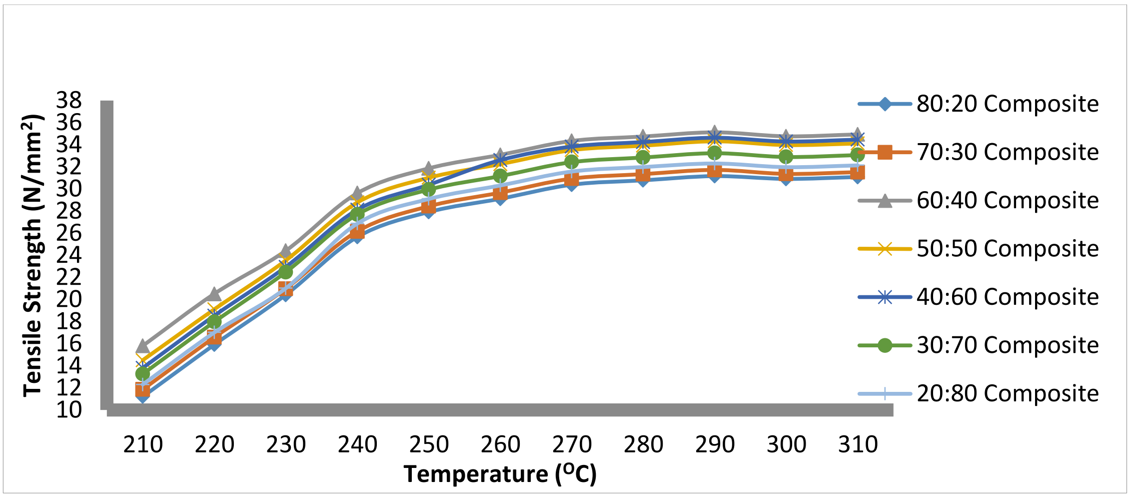

Figure 1: Effects of Barrel Temperature on Tensile Strength for PP-Grass Composite

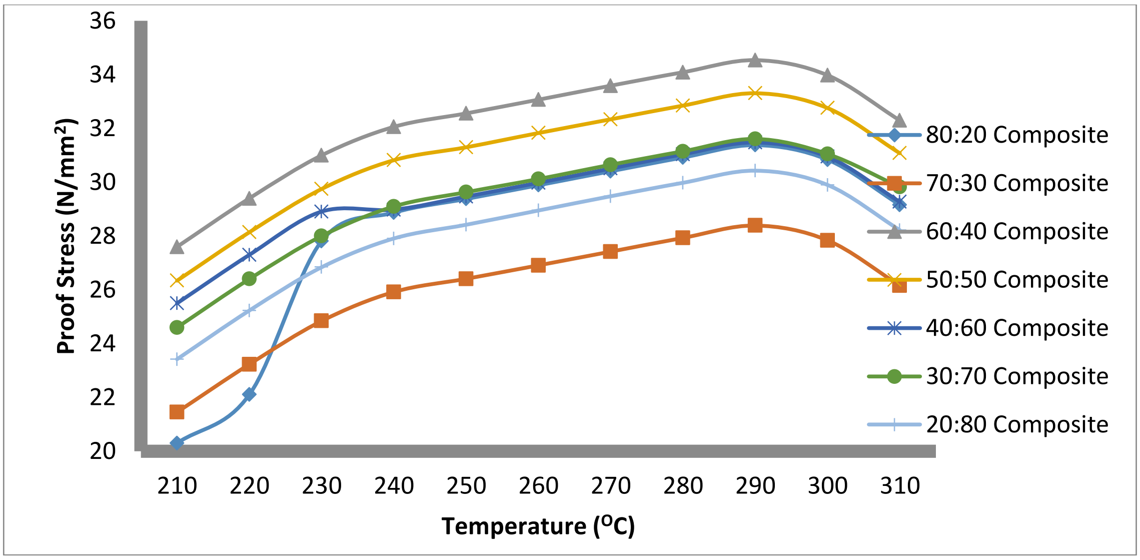

Figure 2: Effects of Barrel Temperature on Proof Stress for PP-Grass Composite

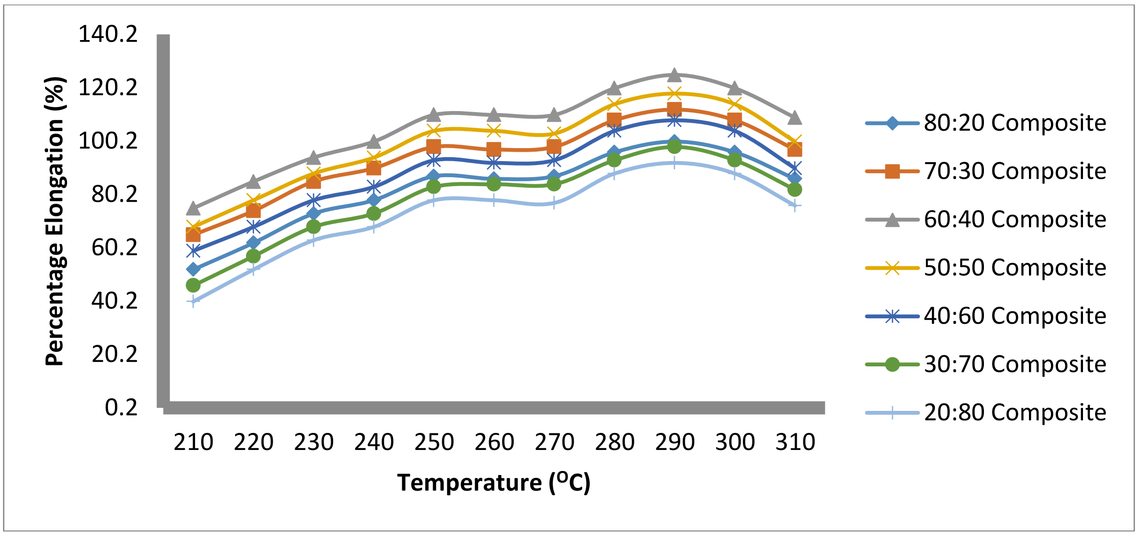

Figure 3: Effects of Barrel Temperature on Percentage Elongation for PP-Grass Composite

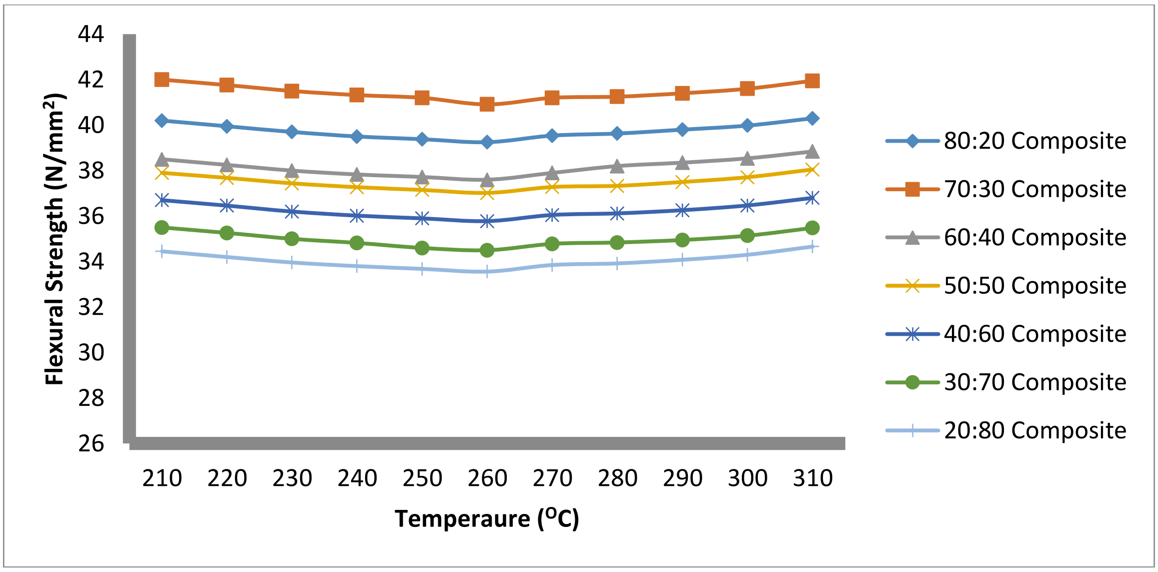

Figure 4: Effects of Barrel Temperature on Flexural Strength for PP-Grass Composite

Table 2: ANOVA Result Table for Effects of Barrel Temperature on PP-Grass Composite

Sources of Variation	Sum of Squares (SS)	Degree of freedom	Mean of Squares (MS)	Fisher's Ratio F_{cal}α=0.05	Fisher's Ratio F_{Tablel}
SS_A	0.00	K-1=1	0.00	0.00	5.99
SS_B	590.23	L-1=6	98.37	0.00	4.28
SS_C	88142.50	I-1=3	29380.83	0.00	9.28
SS_D	735.69	J-1=10	73.57	0.13	2.98
SS_{AB}	0.00	(K-1)(L-1)=6	0.00	-0.00	8.94
SS_{AC}	0.00	(K-1)(I-1)=3	0.00	0.00	3.16
SS_{AD}	5834.08	(K-1)(J-1) =10	583.41	6.25	1.99

SS_{BC}	1540.40	(L-1)(I-1) =18	85.58	1.00	2.01
SS_{BD}	5603.62	(L-1)(J-1) =60	93.39	-29.46	0.51
SS_{CD}	8090.83	(I-1)(J-1)=30	269.69	1.80	1.37
SS_{ABC}	1540.40	(K-1)(L-1)(I-1) =18	85.58	1.16	2.98
SS_{ABD}	-190.15	(K-1)(L-1)(I-1) =60	-3.17	-0.01	0.17
SS_{ACD}	4495.26	(K-1)(I-1)(J-1) =30	149.84	-13.38	1.93
SS_{BCD}	-2015.49	(L-1)(I-1)(J-1) =180	-11.20	0.21	6.57
SS_E	-9575.54	(I-1)(J-1)(K-1)(L-1) =180	-53.20		
SS_T	101203.70	IJKL-1 =615			

3.1. Interpretation of the Results

Table 1 shows the effects of barrel temperature on mechanical Properties of PP-Grass Composite. Figure 1-4 shows the graph of effects of barrel temperature on tensile strength, proof stress, Percentage Elongation and flexural strength respectively. Table 2 shows Anova results for effects of barrel temperature on PP-Grass Composite.
The investigation of treatment effect of materials (SS_A), percentage by volume of materials (SS_B), mechanical strength (SS_C) and barrel temperature (SS_D) respectively shows that the calculated Fisher's ratio values were less than the Fisher ratio values obtained from the table at α-value of 0.05 (Table 2). The results compared favourably with the results obtained by Goos, and Vandebroek [5] using D-optimal Split-Plot Designs with Given Numbers and Sizes of Whole Plots. From the results obtained, it shows that the experimental data do not furnish enough evidence to reject the null hypothesis H_o treatment at α-value of 0.05. This shows that the treatment effect and the block effect of process parameters contribute significantly to the mechanical property of the produced PP-Grass composite in industries.
Furthermore, the interaction of the process parameters obtained from treatment effect such as Material type and percentage by volume of material Interaction (SS_{AB}); Material type and Mechanical Strength Interaction (SS_{AC}); Material type, Percentage by volume of material and Mechanical Strength Interaction (SS_{ABC}); Percentage by Volume of material and Mechanical Strength Interaction (SS_{BC}); Percentage by volume of material and Temperature Interaction (SS_{BD}); Mechanical Strength and Temperature Interaction (SS_{CD}); Material type, Percentage by volume of material and Temperature Interaction (SS_{ABD}); Material type, Mechanical strength and Temperature Interaction (SS_{ACD}); Percentage by volume of material, Mechanical strength and Temperature Interaction (SS_{BCD}) respectively shows that the calculated Fisher's ratio value is less than the Fisher ratio obtained from the table at α-value of 0.05 (Table 2). The results compare favourably with the results obtained by Goos and Vandebroek [5]. The experimental data do not furnish enough evidence to reject the null hypothesis H_o treatment at α-value of 0.05. This shows that the treatment effect and the block effect interaction of these process parameters contribute significantly to the mechanical property of the produced PP-Grass composite in industries.
Moreover, the interaction of the process parameters obtained from treatment effect such as Examination of Treatment Effect of Material type and Temperature Interaction (SS_{AD}) shows that the calculated Fisher's ratio value is more than the Fisher ratio obtained from the table at α-value of 0.05 (Table 2). The results compare favourably with the results obtained by Loeza and Donev [6]. The experimental data furnish enough evidence to reject the null hypothesis (H_o) at α-value of 0.05. This shows that the treatment effect of material type and block effect (barrel temperature) interaction parameters does not contribute significantly to the strength of the composite produced in industries.

4. Conclusion

The results of the calculated Fisher's Ratio (F_{cal}) at significant value of 0.05 for the process such as percentage by volume of material, barrel temperature, material type and their interactions ranges from -29.46 to 6.25 respectively. The results obtained shows that were strong interactions between barrel temperature, type of material and percentage by volume of material on mechanical properties (Tensile Strength, Proof Stress, Percentage Elongation and Flexural Strength) for the produced PP-Grass composites. Hence, these process parameters contributes significantly to the developed injection moulded PP-Grass composite. Decisions made based on the hypothesis statements shows that there were no enough evidence to reject the null hypothesis at α-value of 0.05 for PP-Grass composite. The developed model and results obtained will be useful to researcher, industrialist and small scale manufacturer to ease the production of plastic-grass composite in polymeric industries.

Nomenclature

μ	Mean response;
γ_I	Block variable (mechanical properties);
β_j	Block variable (barrel temperature);
δ_l	Treatment Variable (percentage by volume of material);
y_k	Treatment Variable (type of material);
$\gamma\beta_{ij}$	Block interaction (mechanical properties and barrel temperature interaction);
γy_{ik}	Block and Treatment interaction (mechanical properties and type of material interaction);
βy_{jk}	Treatment Interaction (barrel temperature and type of material interaction);
$\gamma\delta_{il}$	Block and Treatment interaction (mechanical properties and percentage by volume of material interaction);
$\beta\delta_{jl}$	Block and Treatment interaction (barrel temperature and percentage by volume of material interaction);
$y\delta_{lk}$	Treatment Interaction (percentage by volume of material and type of material interaction);
$\gamma\beta y_{ijk}$	Block and Treatment interaction (mechanical properties, barrel temperature and type of material interaction);
$\gamma\beta\delta_{ijl}$	Block and Treatment interaction (mechanical properties, barrel temperature and Percentage by volume of material interaction);
$y\gamma\delta_{ikl}$	Block and Treatment interaction (mechanical properties, type of material and Percentage by volume of material interaction)
$\beta y\delta_{jkl}$	Block and Treatment interaction (barrel temperature, type of material and Percentage by volume of material interaction);
$\gamma\beta\delta y_{ijkl}$	Block and Treatment interaction (mechanical properties, barrel temperature, type of material and percentage by volume of material interaction);
X_{ijkl}	Response Variable;
ε_{ijkl}	Error term

5. Acknowledgment

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6. Conflict of Interest

There is no conflict of interest associated with this work.

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