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OPTIMIZATION OF INJECTION MOULDING PROCESS VIA DESIGN OF EXPERIMENT (DOE) METHOD BASED ON RICE HUSK (RH) REINFORCED LOW DENSITY POLYETHYLENE (LDPE) COMPOSITE PROPERTIES

Optimal parameters setting of injection moulding (IM) machine critically effects productivity, quality, and cost production of end products in manufacturing industries. Previously, trial and error method were the most common method for the production engineers to meet the optimal process injection moulding parameter setting. Inappropriate injection moulding machine parameter settings can lead to poor production and quality of a product. Therefore, this study was purposefully carried out to overcome those uncertainty. This paper presents a statistical technique on the optimization of injection moulding process parameters through central composite design (CCD). In this study, an understanding of the injection moulding process and consequently its optimization is carried out by CCD based on three parameters (melt temperature, packing pressure, and cooling time) which influence the shrinkage and tensile strength of rice husk (RH) reinforced low density polyethylene (LDPE) composites. Statistical results and analysis are used to provide better interpretation of the experiment. The models are form from analysis of variance (ANOVA) method and the model passed the tests for normality and independence assumptions.

Keywords: Injection Moulding; Design of Experiments (DOE); Central Composite Design; response surface methodology (RSM); Shrinkage; Tensile Strength

1. Introduction

In various manufacturing industries, injection moulding served as one of the most crucial processing methods for manufacturing plastic products which successfully developed into a mature technology. Injection moulding is greatly preferred in manufacturing industry as the process able to produce complex-shaped plastic parts with good dimensional accuracy at very short cycle time [1,2]. In this field, product quality is one of the essential criteria that always needed to be achieved if the required material is prone to change in geometry, color, and other related properties. Among many problems involving injection moulding processes, shrinkage listed as one of the most common defects that effect the product dimensions especially in thermoplastic materials [3,4].

Shrinkage can be explained as the bulk contraction of the polymer as it changes from the molten to the solid phase (due to relaxation of stretched polymers chains and crystallization) where this phenomenon critically influence the aesthetic properties of the end product [5]. This phenomenon causes by many factors including process parameters at filling, packing and cooling phases, cooling system design, and material properties. Particularly, semi-crystalline materials are prone to thermal shrinkage where the increasing level of crystallinity increased the shrinkage percentage. Practically, there are many ways to minimize the shrinkage including the reinforcement of material such as fiber filled thermoplastics, where the reinforced fibers able to be minimized shrinkage due to their smaller thermal contraction and higher modulus than the unfilled thermoplastics [3]. For the materials processing using injection moulding, many investigators have attempted to manufacture moulding product at an acceptable quality. However, in industry the level of product quality totally depends on the customer requirements which include mechanical, rheological, aesthetic, and thermal. The issue is how to satisfy all these requirements at the same time, where all the mentioned requirements are strongly

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influenced with the processing parameters. Therefore, optimization is needed to satisfy all the requirements by optimizing the targeted processing parameters. There are various methods possibly used to predict and optimized plastic injection moulding process accurately without consuming much time (less trial and error testing), costs, materials, and labor effort, which are artificial neural networks, genetic algorithm, and design of experiments (DOE) [6,7]. DOE offers capability to provide optimal solutions through numerical and graphical technique, which is very useful to design and analyze complicated industrial design. IM process is particularly suited to DOE due to the large number of process parameters and could be interactive to each other [5,6].

In this research, a thermoplastic polymer, low density polyethylene (LDPE) was used as the matrix polymer and the rice husk (agro-waste) flour as the reinforcing filler to develop a reinforced composite. To this date, there are only few studies available on the rice husk (*Oryza sativa*) reinforced low density polyethylene composites, specifically for the process optimization towards industrial injection moulding parts [7,8]. Thus, in this research, industrial injection moulding machine will be exploited to produce rice husk reinforced composite parts, since the implementation of rice husk composites have been extended from conventional to more crucial and complex applications (i.e., automotive, construction, and leisure). To accomplish the main goal in this research, targeted processing parameters (melt temperature, packing pressure, and cooling time) was executed through central composite design method for the rice husk reinforced composites to determine the optimum injection moulding processing parameters.

2. Experimental

2.1. Materials

LDPE (LDI300YY) was supplied by Lotte Chemical Titan in the solid pellet form. TABLE 1 shows the properties of the LDPE used in this research, according to the manufacturer's data.

TABLE 1

The properties of low-density polyethylene used in this research

Properties	Values	ASTM Method
Melt index, g/10 min	20	D1238
Density, g/cm^3	0.920	D1505
Melt temperature, ºC	160-240	—
Mold temperature, ºC	15-50	—

2.2. Rice husk (RH) filler

The RH was acquired from BERNAS rice mill factory at Utan Aji, Perlis, Malaysia in its original form. The acquired RH is specifically from the rice plant of main species of *Oryza sativa* (Asian rice) that also known as paddy which one the most widely consumed staple food in Asia. In this study, the automated hammer continuous grinder (HK-08B) was used to reduce the size of the RH. After that, the RH was sieved into preferred size (i.e., approximately 250 µm) using a vibratory sieve shaker AS 200 controls before proceeds into particle size analyzer (Malvern Sciro2000 Mastersizer) for particle size confirmation.

2.3. Injection moulding (IM) experiment

The IM experiments were conducted using a NISSEI NEX1000 injection moulding machine. The IM parameters were set according to a list of experiments generated for selected parameter such as melt temperature, packing pressure, and cooling time. The other parameters were set as a fixed value based on the recommended results from the simulation. The IM process started by preheating the plastic resin in a hoppper and then melt in a barrel. Injection process started when the material was injected into a mould by moving the barrel according to the injection speed profile that have been set. The injection process was stopped when a specified switch-over position had reached approximately 99% of the filling process. The pressure then taken over the velocity to push more material into the mould until specified filling time is reached approximately when the gate freezes. After that, the cooling process takes place until it reaches a specified cooling time. The process ended when the product is ejected out from the mould.

3. Testing and measurement of the moulded part

3.1. Tensile testing

All LDPE/RH composites were tested under tension force at constant crosshead speed of 50 mm/min using an Axial-Torsion Universal Testing Machine according to ASTM D638. The specimens were in dog-bone shape with the dimension of 150 mm × 4 mm × 1 mm. Prior to the test, the thickness of all specimens will be measured using Vernier caliper. Ten measurements were taken for each different loading to compute the average behavior of tested LDPE/RH composites.

3.2. Post-shrinkage measurement

Post-shrinkage was measured according to the ASTM D955-08 standard to determine the post-shrinkage of the moulded part. In this study, the post-shrinkage measurement (post-moulding) was measured 48 hours after moulding process. Digital caliper was used to measure the post-shrinkage of the moulded part for post-shrinkage in parallel direction of the melt flow. The accuracy of the caliper is 0.01 mm as recommend in ASTM D955-08 standard. Ten specimens for each of moulding were selected for post-shrinkage measurement. The results of the moulding shrinkage measurement can be calculated using Equation 1 for post-shrinkage in parallel direction of the melt flow, S_{Mp} (%) and Equation 2 for post-shrinkage in normal direction of the melt flow, S_{Mn} (%). Where, l_c (mm) and b_c are the length and width across the center of the cavity respectively, and l_1 (mm) and b_1 (mm) are the corresponding length and width of the test specimen respectively [12].

$$S_{Mp} = 100\frac{l_c - l_1}{l_c} \quad (1)$$

$$S_{Mn} = 100\frac{b_c - b_1}{b_c} \quad (2)$$

4. Experimental design and analysis of data

The results obtained from the experiment for post-shrinkage and tensile strength were analyzed using Design Expert software with RSM method. All the results from the experiments were arranged in a list of experiments and used to generate a relationship between input parameters setting and output responses which are post-shrinkage and tensile strength of the specimen. In this study, two levels of full factorial design were selected to analyze using three factors which are melt temperature (°C), packing pressure (MPa), and cooling time (s). Post-shrinkage in parallel and normal directions to melt flow, and tensile strength are selected as the responses. List of experiments generated in Design Expert Software based on three factors and four center points is shown in TABLE 2. Then, the full factorial design was augmented to RSM using center composite design (CCD) that consist of additional eight runs as shown in TABLE 3. Based on the results, a model was generated for each response based on the required backward model selection (alpha out = 0.05). The insignificant parameters or interaction between parameters were removed and excluded from the generated models for both post-shrinkage and tensile strength. These models were used during the optimization process.

TABLE 2

List of experiments for full factorial design

Run	Melt temperature (°C)	Packing pressure (MPa)	Cooling time (s)
1	170	15.93	23.03
2	160	12.96	26.74
3	180	18.89	19.33
4	180	12.96	26.74
5	160	18.89	19.33
6	170	15.93	23.03
7	160	12.96	19.33
8	170	15.93	23.03
9	180	12.96	19.33
10	170	15.93	23.03
11	160	18.89	26.74
12	180	18.89	26.74

TABLE 3

Additional experimental design of CCD

Run	Melt temperature (°C)	Packing pressure (MPa)	Cooling time (s)
13	170	15.93	19.33
14	170	18.89	23.03
15	170	12.96	23.03
16	170	15.93	23.03
17	160	15.93	23.03
18	180	15.93	23.03
19	170	15.93	26.74
20	170	15.93	23.03

5. Optimization process

The Design Expert software used to generate a list of the output model based on input variables that was set. These models were used to evaluate the best value of post-shrinkage and tensile strength. The most optimum values are the least value for post-shrinkage and the highest value for tensile strength. Then, the software determined the best combination of input parameters for the best result of output responses, resulted in minimizing the value of post-shrinkage and maximizing the value of tensile strength using RSM (CCD) design of experiment.

6. Verification test

Confirmation experiments were applied in this study to verify the results. The combination of input variables from the optimization result were used in the injection moulding machine to produce new parts. These parts underwent the procedure of determining the post-shrinkage and tensile strength measurement. The results were compared with the result from the model generated.

7. Results and Discussion

7.1. Experimental design and analysis of central composite design

From the full factorial analysis and results shown in TABLE 4, both post-shrinkage (parallel and normal melt flow direction) and tensile strength shows that the model and the curvature are significant (p-value < 0.05), where the second order models were the best option for further optimization by augmenting axial runs, to allow quadratic terms to be incorporated into the model. In this study, CCD with face centered was applied to optimize the model. The CCD method was popular among researchers as it is one of the most efficient design for fitting the second order model or response surface [13-15]. Based on the three factors involved, 8 axial runs with 2 replicates of center runs were required for the optimization process as recommended by the Design Expert software.

TABLE 4

Summary results of ANOVA of full factorial for both post-shrinkage (normal and parallel melt flow direction) and tensile strength

Responses	Summary	
	Model	Curvature
Post-shrinkage (normal melt flow)	Significant	Significant
Post-shrinkage (parallel melt flow)	Significant	Significant
Tensile strength	Significant	Significant

The models of post-shrinkage (both normal and parallel direction to the melt flow) and tensile strength were developed based on the results of central composite design. These models were used to predict the best combination of parameters to minimize the post-shrinkage while maximize the tensile strength at the same time towards LDPE/RH composites moulded part.

7.2. Analysis results of CCD for post-shrinkage in normal direction to the melt flow

Based on the ANOVA of post-shrinkage in normal direction to the melt flow after augmentation of full factorial experimental design, the result shows that the model was significant with several model terms. The main effects of A (melt temperature), B (packing pressure), and C (cooling time) are significant to the model, together with two terms that give quadratic effect to the model (A and B) after fitting the second-order model. A quadratic effect is an interaction term where a factor interacts with itself. This can be explained through a factor for example, A is a linear term, AB is an interaction with B, and A^2 is a quadratic effect. A positive quadratic term makes the curve convex while a negative quadratic term makes the curve concave.

Table 5 shows the summary of ANOVA for post-shrinkage in normal direction to melt flow of LDPE/RH composites. From TABLE 4, the value of R^2 (statistical measure of how close the data are to the fitted regression line) and adjusted R^2 are very high which is close to 1 (≈0.9972 and ≈0.9961) indicate that the model is desirable. The adjusted R^2 has a difference of only 0.0035 with predicted R^2 which implies that it is in reasonable agreement (<0.2). For the adequate precision, the value is 75.872 which is higher than 4 and indicates that the model is adequate [16,17].

TABLE 5

Summary of ANOVA for post-shrinkage in normal direction to the melt flow

Description	Value
Standard Deviation	0.033
Mean	1.71
R^2	0.9972
Adjusted R^2	0.9961
Predicted R^2	0.9926
Adequate Precision	75.872

Figure 1 and Figure 2 depicted the 3D surface of shrinkage in normal direction, correspondingly. Both 3D surface graphs reveal that the lowest post-shrinkage can be achieved when melt temperature is set between 165°C and 173°C with the cooling time at 26.74 s and packing pressure at 15.93 MPa, respectively.

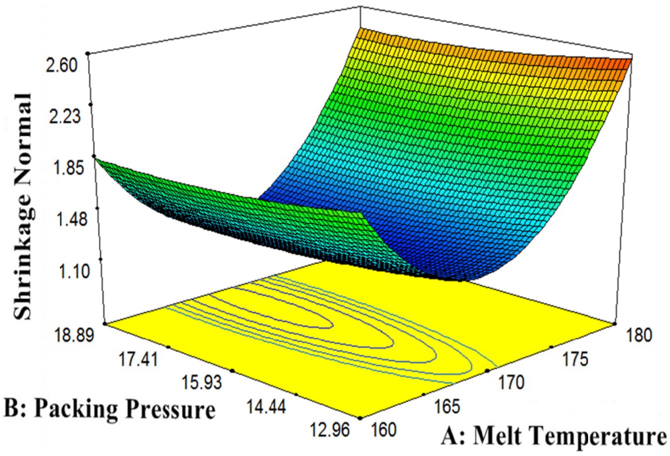

Fig. 1. 3D surface (response surface) of post-shrinkage in normal direction under the effects of melt temperature and packing pressure

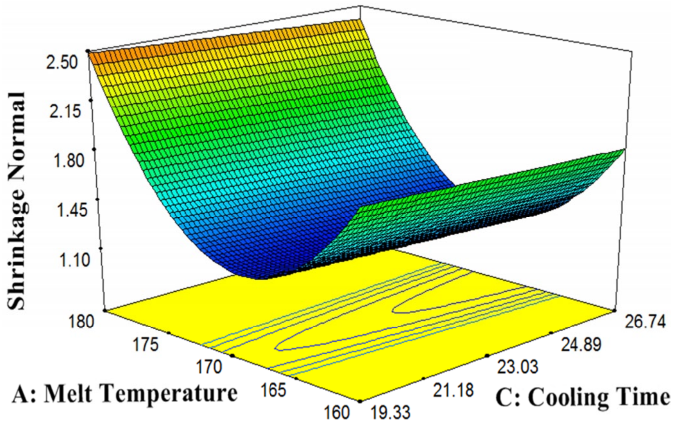

Fig. 2. 3D surface (response surface) of post-shrinkage in normal direction under the effects of melt temperature and cooling time

The empirical model generated by the Design Expert software was used to estimate the response of shrinkage in normal direction to the melt flow within the range investigated. The empirical models in term of actual factor are shown in Equation (3):

$$Post-shrinkage_{normal} = \\ = 272.43105 - 3.19286A - 0.22607B - 0.011984C \\ + 0.00947487A^2 + 0.00636849B^2 \quad (3)$$

Where A is the melt temperature (°C), B is the packing pressure (B), and C is the cooling time (s). The experimental and predicted results of post-shrinkage in the normal direction to the melt flow of LDPE/RH composites is shown in Figure 3. Overall, the experimental results are in line with the results from the empirical model. Therefore, the empirical model has a good prediction in predicting the post-shrinkage values with 8.115% error between average experimental and predicted results.

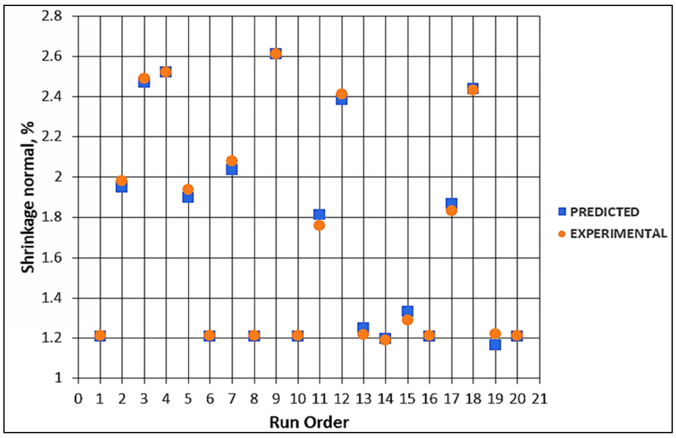

Fig. 3. Experimental and predicted results of post-shrinkage in normal direction to the melt flow using CCD

7.3. Analysis results of CCD for post-shrinkage in parallel direction to the melt flow

Based on ANOVA result of post-shrinkage in parallel direction to the melt flow of LDPE/RH composites after augmentation of full factorial experimental design, the sum of square value for block is 0.002651 which shows that the variations between blocks are not critical (≈0), where the model shows a significant result with p-value lower than 0.0001. The ANOVA results of shrinkage in parallel direction to the melt flow also indicate that melt temperature (A) and packing pressure (B) are significant parameters in this analysis where p-value is less than 0.05, with no interaction between parameters. TABLE 6 shows the summary of ANOVA result for post-shrinkage in parallel direction to the melt flow of LDPE/RH composites. From TABLE 5, the R^2 and adjusted R^2 presented high values which is 0.9300 and 0.9100, respectively, and can be conclude that model is desirable. For the difference between adjusted R^2 and predicted R^2, the result is 0.0938 which indicates that the model is in reasonable agreement as the value is below than 0.2. While for the adequate precision result, the value is 17.484 (>4), and proved that the model is adequate for future optimization.

TABLE 6

Summary of ANOVA for post-shrinkage in parallel direction to the melt flow

Description	Value
Standard Deviation	0.019
Mean	0.47
R^2	0.9300
Adjusted R^2	0.9100
Predicted R^2	0.8162
Adequate Precision	17.484

Figure 4 and Figure 5 represents the 3D surface graphs respectively, which shows that there is no interaction was found for the shrinkage in parallel direction to the melt flow of LDPE/RH composites. However, the results shows that shrinkage was reduced with increased packing pressure at low melt temperature.

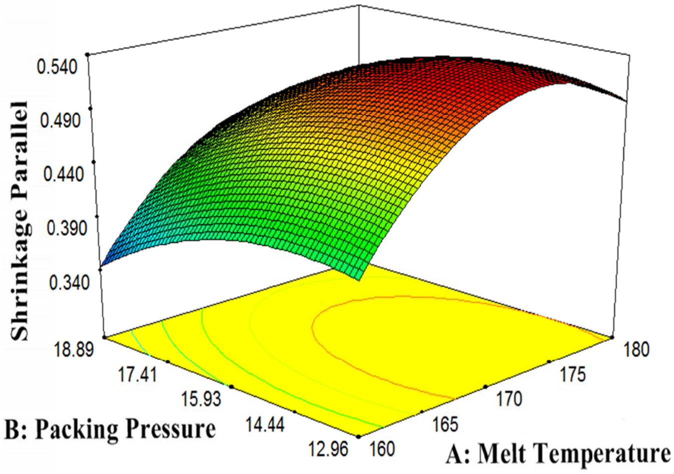

Fig. 4. 3D surface (response surface) of post-shrinkage in parallel direction under the effects of melt temperature and packing pressure

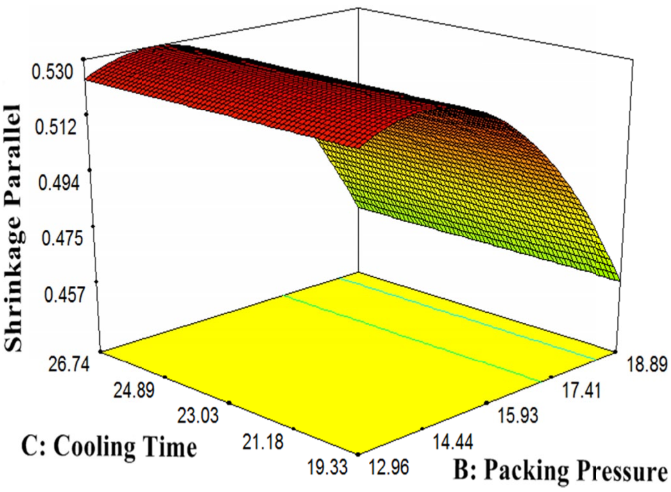

Fig. 5. 3D surface (response surface) of post-shrinkage in parallel direction under the effects of packing pressure and cooling time

The empirical models for the shrinkage in parallel direction to the melt flow in terms of actual factors is shown in Equation (4):

$$Post-shrinkage_{parallel} = -21.05322 + 0.24123A + \\ + 0.098049B - 0.000696447A^2 - 0.00342896B^2 \quad (4)$$

Where A is the melt temperature (°C) and B is the packing pressure (MPa). The experimental and predicted results of post-shrinkage in parallel direction to the melt flow of LDPE/RH composites are illustrated in Figure 6. The generated graph of experimental versus shrinkage in parallel direction to the melt flow has given a good prediction whereas the pattern shows similarity between the experimental results and empirical model. Error of average value between experiment and predicted model is 1.3% indicated the model is very good relation from experimental.

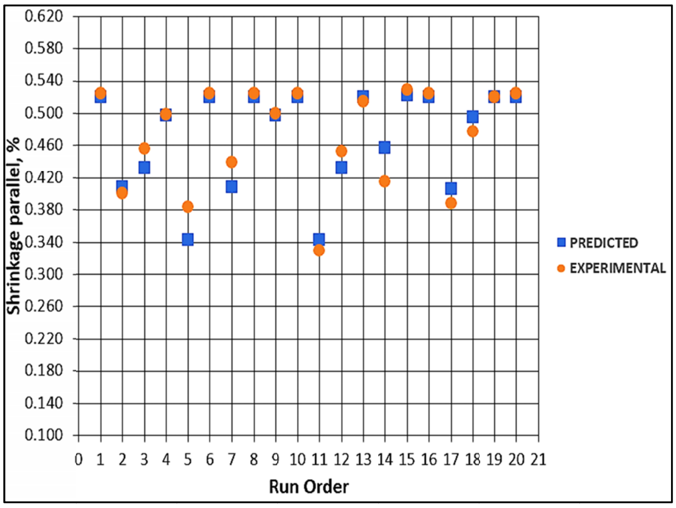

Fig. 6. Experimental and predicted results of post-shrinkage in parallel direction to the melt flow using CCD

7.4. Analysis results of CCD for tensile strength

Based on the ANOVA of CCD analysis for tensile strength of the LDPE/RH composite moulded parts, the results explained that the model was significant as the p-value is less than 0.05. Melt temperature (A) and packing pressure (B) are the significant main effects, with the terms A^2 also significant (p-value < 0.05) in establishing the polynomial model. TABLE 7 shows the summary of ANOVA results of tensile strength for LDPE/RH composites. From TABLE 6, the R^2 value is 0.9894 which acceptable for the model (≈1). The predicted R^2 is in reasonable agreement with the adjusted R^2 where the difference between them is only 0.0082 in this analysis. The adequate precision value is 54.762 which is more than 4 and indicates that the model is adequate.

TABLE 7

Summary of ANOVA for tensile strength

Description	Value
Standard Deviation	0.025
Mean	10.79
R^2	0.9894
Adjusted R^2	0.9873
Predicted R^2	0.9791
Adequate Precision	54.762

Figure 7 and Figure 8 shows the 3D surface graphs for the corresponding interaction, respectively. Both graphs explained that the maximum tensile strength of the moulded part was achieved at high melt temperature (180°C) and high packing pressure (18.89 MPa).

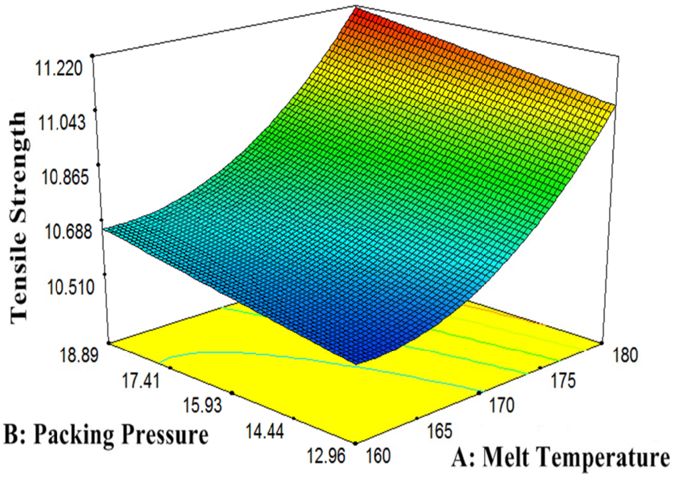

Fig. 7. 3D surface (response surface) of tensile strength under the effects of melt temperature and packing pressure

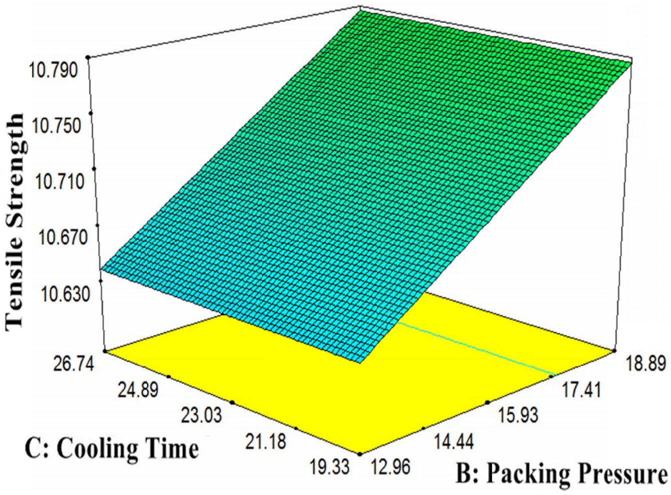

Fig. 8. 3D surface (response surface) of tensile strength under the effects of melt packing pressure and cooling time

The empirical model of the tensile strength in terms of the actual factors is shown in Equation (5):

$$Strength_{tensile\ strength} = 49.89726 - 0.49292A + \\ + 0.024789B + 0.00153A^2 \quad (5)$$

Where A is melting temperature (°C) and B is packing pressure (MPa). The experimental and predicted results of tensile strength are illustrated in Figure 9. From the generated graph, experimental versus predicted results of tensile strength shows a similar pattern. Based on this result, it can be concluded that the predicted value is in a very good agreement with the experimental results as the error between experiment and predicted values is only about 1.645%.

7.5. Optimization result of CCD

The predicted optimal solution of parameters for the post-shrinkage in parallel and normal directions to the melt flow,

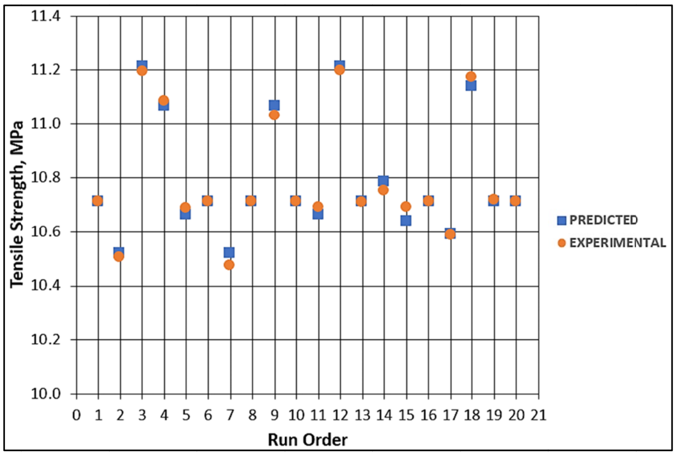

Fig. 9. Experimental and predicted results of tensile strength using CCD

and the tensile strength of the LDPE/RH composites moulded part using RSM was concluded in TABLE 8. These results were taken separately from Equations (4) to (5). The response value was a predicted value based on the optimal variable.

TABLE 8

Predictive optimal solution of the post-shrinkage and the tensile strength

Response	**Variable**			**Response value**
	Melt temp. (°C)	**Packing pressure (MPa)**	**Cooling time (s)**	
Post-shrinkage (normal)	168.36	17.18	22.44	1.175%
Post-shrinkage (parallel)	160.00	18.89	25.86	0.343%
Tensile strength	179.97	18.59	20.43	11.203 MPa

Table 9 shows the combination of variables and predicted responses in multi objectives optimization. When multi objectives optimization was applied, both predicted values of post-shrinkage and tensile strength show the increased in post-shrinkage rate and decreased in tensile strength. However, the difference between predicted optimal solution in separate objective and the predictive optimal solution in multi objectives were not significant and critical, thus this multi objectives optimization still reliable to be implement in injection moulding machine setting specifically for LDPE/RH composites.

TABLE 9

Predictive optimal solution of post-shrinkage and the tensile strength in multi objectives

	Factors/Output	**Value**
Variable	Melt temperature (°C)	175.66
	Packing pressure (MPa)	18.89
	Cooling time (s)	26.74
Response	Post-shrinkage, normal (%)	1.616
	Post-shrinkage, parallel (%)	0.460
	Tensile strength (MPa)	10.991

Besides, the experimental results between the optimal solution parameters (RSM) and the recommended values obtained from simulation was compared to attain a significant result in this study as shown in TABLE 10. The recommended values from the simulation were used in the experiment works to evaluate the post-shrinkage and tensile strength of the LDPE/RH composite moulded parts. Results show that the post-shrinkage was reduced significantly by 1.498% and 0.192% for both normal and parallel direction to the melt flow respectively, while the tensile strength was improved about 9.303% compared to the recommended setting from simulation. Meanwhile, the reduction of post-shrinkage in parallel and normal directions to the melt flow using multi objective optimization were 0.963% and 0.078% respectively, and the tensile strength of the moulded part was increased by 7.79%. These improvements according to the combination of parameters setting that were used during experimental works to improve the post-shrinkage and tensile strength on the LDPE/RH composite moulded parts. Based on these results, it can be proved that the predicted optimal solution of the selective parameters for both separate and multi objectives optimization are reliable and successfully enhanced the performance of the LDPE/RH composite moulded parts.

7.6. Verification test

A series of verification tests were conducted to validate the predicted model solutions. The verification results from experiments are represented in TABLE 11. Evaluation was made by comparing the results from prediction and confirmation tests. The predicted error in percentage (%) was calculated using Equation (6) below [18]:

TABLE 10

Comparison of the results from experimental works and recommended setting from simulation

	Response	**Recommended setting**	**Optimal solution by optimization**	**Improvement (%)**
Separate objective	Post-shrinkage, normal (%)	2.681	1.183	1.498
	Post-shrinkage, parallel (%)	0.533	0.341	0.192
	Tensile strength (MPa)	10.244	11.197	9.303
Multi objective	Post-shrinkage, normal (%)	2.681	1.718	0.963
	Post-shrinkage, parallel (%)	0.533	0.455	0.078
	Tensile strength (MPa)	10.244	11.042	7.790

$$Error = \left| \frac{Result\ of\ confirmation\ test - Predicted\ value}{Result\ of\ confirmation\ test} \right| \times 100 \quad (6)$$

From TABLE 11, predicted errors ranged from 0.054% to 5.937%. The prediction system proved that the results are in good agreement with the verification experiments standard where the errors must below than 20% [19].

8. Conclusion

In this research, a recommended set of parameters were identified using the simulation process through Autodesk Moldflow Insight. From the simulation process, a range of variable parameters were determined to generate an experimental design for full factorial analysis followed by rotatable CCD of RSM method for the selected variable parameters which are melt temperature, packing pressure, and cooling time using Design Expert software. The results of full factorial analysis and rotatable CCD were evaluated, and the optimum setting parameters were obtained purposely to minimized post-shrinkage and maximized the tensile strength through multi objectives and separate objectives optimizations. Based on the overall analyses, melt temperature was found to be the most significant factor affecting both post-shrinkage (normal and parallel) and tensile strength of the LDPE/RH composite moulded parts. It can be concluded that, the melt temperature of 175.66°C, packing pressure of 18.89 MPa and cooling time of 26.74 s are the best combination of parameters to minimize the post-shrinkage up to 0.963% and maximized the tensile strength up to 7.79% compared to the experimental results from the recommended setting simulation specifically for LDPE/RH composite moulded parts. Validation process shows that the prediction system is in good agreement with the verification experiments with error of less than 20%.

TABLE 10

Results of verification test

	Responses	Variable			Predictive value (RSM)	Actual value	Error (%)
		Melt temperature (°C)	Packing pressure (MPa)	Cooling time (s)			
Separate objective	Post-shrinkage normal (%)	168.36	17.18	22.44	1.175	1.183	0.676
	Post-shrinkage parallel (%)	160.00	18.89	25.86	0.343	0.341	0.587
	Tensile strength (MPa)	179.97	18.59	20.43	11.203	11.197	0.054
Multi objective	Post-shrinkage normal (%)	175.66	18.89	26.74	1.616	1.718	5.937
	Post-shrinkage parallel (%)				0.460	0.455	1.099
	Tensile strength (MPa)				10.991	11.042	4.619

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