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## Experimental study of influence injection moulding on shrinkage material sisal-glass fiber biocomposite using response surface methodology (RSM)

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# Experimental study of influence injection moulding on shrinkage material sisal-glass fiber biocomposite using response surface methodology (RSM)

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**Abstract.** Shrinkage is one of the properties of plastic materials, which the greater the shrinkage percentage value can result poor product quality and possible product defects. Therefore, it is important to know the shrinkage value of a material so that the geometry design of a product can be achieved properly. In this final project, shrinkage control is carried out by adjusting injection process parameters, until minimum shrinkage is obtained. The parameters set to control shrinkage values are melting temperature, holding pressure, and injection pressure. According to ASTM D955 to determine the shrinkage value of a material the specimen is made through the injection process. To obtain the minimum shrinkage value, an experimental design was carried out by using response surface method to reach the optimum injection process parameters with Box-Behnken parameter design. As the next step, the injection specimens which made from sisal fiber biocomposite material with composition of 80% Polypropylene, 10% MAPP, and 10% sisal fiber with injection process parameters according to the design. The injection result specimens were measured by calipers, Minitab 18 software was used to process the injection result. This result is used to determine the optimum value of the injection process parameters. Furthermore, with the optimum injection process parameters, the injection process is carried out again with the same material. The specimens obtained were measured to carry out a confirmation test, so that the minimum shrinkage value was obtained. From the statistical response surface analysis using Minitab 18 software, the optimum injection process parameters were obtained at 185<sup>o</sup> C, holding pressure 35 bar and injection pressure 60 bar with shrinkage values of 1.05%.

## 1. Introduction

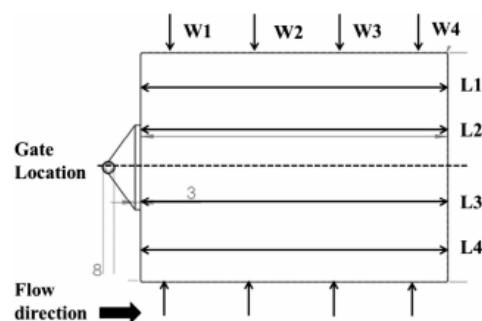
Shrinkage of products written in units of percent of products where the value of shrinkage of each plastic material varies. In the process of injection molding in the manufacture of plastic biocomposite materials many components with complex designs and require a high degree of accuracy. The shrinkage properties of a material can cause a decrease in the precision of a product. In 2017 Deni Gumelar in his assignment finally designed injection molding of shrinkage specimens that were adjusted to the ASTM D955 standard and could be used on HAITIAN MA 900 / 260e injection machines. Then Tubagus Bima <sup>[5]</sup> conducted a study and analysis of shrinkage of biocomposite material with a mixture of sisal fiber and rice husk, in which studies and analyzes of biocomposite materials were printed into shrinkage specimens that were in accordance with ASTM D955 standards.



The material used in the simulation and injection molding process is a mixture of 10% biocomposite rice husk, 5% MAPP, 85% PP and mixed biocomposite 10% sisal fiber, 5% MAPP, 85% PP. The results showed varied shrinkage values, this was caused by problems with the mold. Kazmer<sup>[2]</sup> defines parameters that have the most significant effect on shrinkage, including pressure and temperature. The holding time parameter, injection time (time) has a non-significant impact on shrinkage. This research will be carried out using a HAITIAN MA 900/260e injection machine, there are several parameters that can be used including Melt Temperature, Pressure Holding, and Injection Pressure. Determination of process parameters using the Response Surface method which is one of the concepts of statistical approaches using simple and strong experimental designs<sup>[3]</sup>. In the injection molding process, several previous studies adopted the method to determine good results in determining the parameters that affect shrinkage material. The objectives of this research include knowing the effect of melt temperature, Holding Pressure, Injection Pressure, on shrinkage that occurs in sisal fiber biocomposite material. Knowing the optimal injection process parameters to minimize shrinkage values in ASTM D955 specimens using Response Surface Methodology (RSM). Injection machines used in data collection to determine parameters that affect shrinkage on standard specimens ASTM D955 is an injection machine made in HAITIAN China with the type MA 900/260E. The experimental design based on the *box-behnken* design<sup>[3]</sup> can be shown in Table 1.

**Table 1.** Box-Behnken Experiment Design

StdOrder	RunOrder	Blocks	Parameter Proses		
			MT (°C)	HP (Bar)	IP (Bar)
7	1	1	185	30	60
4	2	1	205	35	55
5	3	1	185	30	50
14	4	1	195	30	55
15	5	1	195	30	55
10	6	1	195	35	50
11	7	1	195	25	60
8	8	1	205	30	60
2	9	1	205	25	55
3	10	1	185	35	55
1	11	1	185	25	55
13	12	1	195	30	55
6	13	1	205	30	50
9	14	1	195	25	50
12	15	1	195	35	60

**Figure 1.** Measurement location of the length and width of the specimen

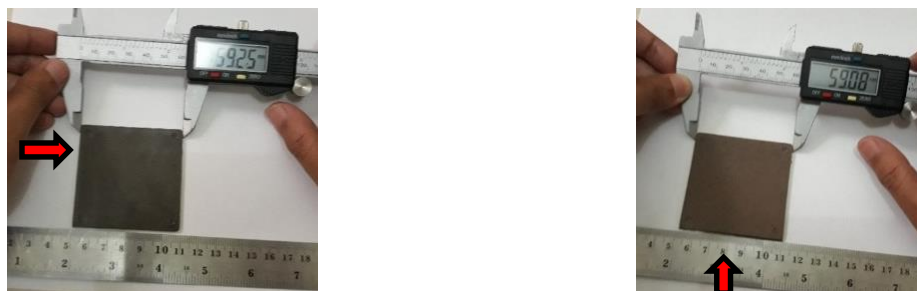
## 2. Specimen Measurement

The results of data retrieval experiments based on the *box-behnken* design are shown in Table 2 with Melt Temperature (MT), Holding Pressure (HP), and Injection Pressure (IP) shrinkage (Sh) response.

**Table 2.** Measurements in the direction of flow (L) and x-flow (W)

Percobaan Ke-	Parameter Proses			Sh L (%)	Sh W (%)
	MT (°C)	HP (Bar)	IP (Bar)		
1	185	30	60	1.17	1.48
2	205	35	55	1.26	1.50
3	185	30	50	1.18	1.49
4	195	30	55	1.26	1.53
5	195	30	55	1.26	1.53
6	195	35	50	1.20	1.46
7	195	25	60	1.23	1.50
8	205	30	60	1.25	1.52
9	205	25	55	1.27	1.53
10	185	35	55	1.09	1.40
11	185	25	55	1.24	1.58
12	195	30	55	1.23	1.51
13	205	30	50	1.28	1.58
14	195	25	50	1.18	1.55
15	195	35	60	1.09	1.38

The process of retrieving data where the shrinkage L results (in the direction of flow) in table 2, get the lowest shrinkage value on the 10<sup>th</sup> and 15<sup>th</sup> experiments at 1.09%. The injection parameters in the 10<sup>th</sup> trial included melt 185°C temperature, holding pressure 35 bar and injection pressure 55 bar. The 15<sup>th</sup> experiment used injection parameters melt temperature 195°C, holding pressure 35 bar and injection pressure 60 bar. The extraction process for shrinkage W results (x-flow) in Table 2, the lowest shrinkage value in the 15<sup>th</sup> experiment was 1.38%. The injection parameters in the 15<sup>th</sup> experiment included melt temperature of 195°C, holding pressure 35 bar and injection pressure 60 bar. Figure 2 [left] is one example of measuring specimens in sisal-glass fiber biocomposite material in the direction of the flow of plastic coming while Figure 2 [right] sisal-glass fiber biocomposite material is x-flow to the flow.

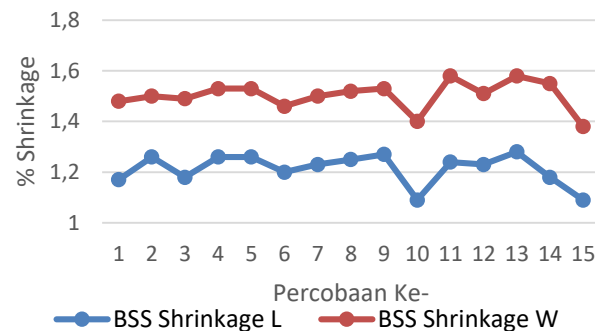


**Figure 2.** Examples of Measurement in ASTM D955 shrinkage test specimens [left] sisal-glass fiber biocomposite in the direction of flow, [right] sisal-glass fiber biocomposite x-flow

### 2.1. Comparison of Sisal-Glass Biocomposite Value

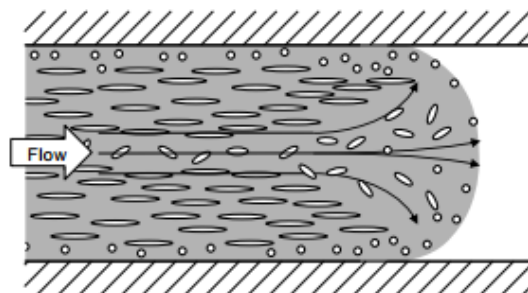
Comparison of graphs of shrinkage material values is shown in Figure 3. The flow rate of sisal-glass fiber biocomposite material (L) has a lower percentage than the x-flow (W) direction of flow of plastic arrival. Plastic and biocomposite materials have anisotropic properties which have different properties

depending on the direction of flow. Anisotropic material where the molecule or fiber becomes parallel in the flow field during the filling process and packing process as shown in Figure 4. Shear flow during the filling and packing process will tend to direct the plastic melt towards the flow due to a large part of the thickness of a specimen.



**Figure 3.** Comparison of percentage of shrinkage in terms of plastic flow

Phenomenon can be influenced by the biocomposite material properties including density and Coefficient of Thermal Expansion. The percentage of shrinkage produced is smaller than the direction of flow of plastic, this can be caused by sisal fiber material distributed in the direction of plastic flow so that it can withstand shrinkage that occurs in the direction of the flow of plastic. This can also be caused by the lower value of the coefficient of thermal expansion material which also tends to have a lower shrinkage value.

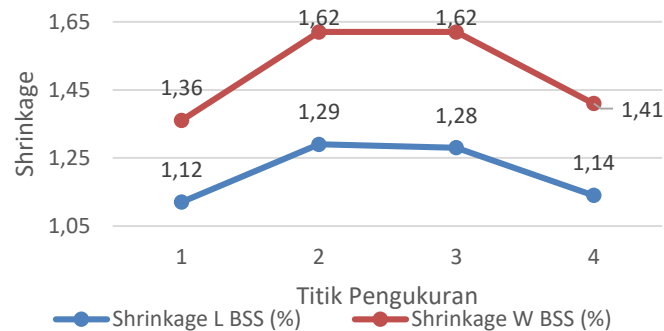


**Figure 4.** Orientation of plastic flow leads to Anisotropic[2]

## 2.2. Paired Sample-T Analysis

Figure 5. shows a graph of the average percentage of shrinkage values in each specimen point. The 1st and 4th points have a lower shrinkage percentage value than the 2nd and 3rd point. This is because the lowest plastic liquid pressure is at the center and increases from the center to the mold wall. The cooling process in plastic fluids tends to begin in the contact area with the cavity side toward the center of the specimen. This has a significant effect on the orientation of the flow of polymer molecules and a large percentage of the value of shrinkage. The percentage of shrinkage at point 1 is lower than at point 4, because the direction of the flow of plastic starts at the 1st point and ends at the 4th point table 3.

Based on data Table 3. shows  $T_{hitung} = 33.02$ , while  $T_{table}$  for two-tail  $\alpha = 0.05$  with  $v = 15 - 1 = 14$  regions received in distribution  $t$  is  $< 2.145$ .  $T_{hitung}$  value  $> T_{table}$ , then  $H_0$  is rejected. The statement that can be concluded is that the measurement of x-flow is different with the direction of flow in the same parameter.

**Figure 5.** Percentage of Each Point Shrinkage**Table 3.** Paired sample-T test results  
Descriptive Statistics

Sample	N	Mean	StDev	SE Mean
tegak lurus	15	1.5027	0.0566	0.0146
searah	15	1.2127	0.0606	0.0157

Estimation for Paired Difference

Mean	StDev	SE Mean	95% CI for $\mu_{\text{difference}}$
0.29000	0.03402	0.00878	(0.27116, 0.30884)

$\mu_{\text{difference}}$ : mean of (in flow and x-flow)

## Test

Null hypothesis	$H_0: \mu_{\text{difference}} = 0$
Alternative hypothesis	$H_1: \mu_{\text{difference}} \neq 0$
T-Value	P-Value
33.02	0.000

**2.3. Response Surface Methodology Analysis**

Analysis of estimation of second-order models of continuous Sisal-glass fiber biocomposite material the flow of plastic to shrinkage values using Minitab 18 software can be seen in Table 4. and Table 5.

**Table 4.** Second-order BSS Shrinkage in the direction of flow

## Analysis of Variance

Source	DF	Adj SS ( $10^{-4}$ )	Adj MS ( $10^{-3}$ )	F-Value	P-Value
Model	9	50.59	5.621	31.23	0.001
Linear	3	29.10	9.700	53.89	0.000
Melt Temperature	1	18.05	18.05	100.2	0.000
Holding Pressure	1	9.800	9.800	54.44	0.001
Injection Pressure	1	1.250	1.250	6.94	0.046
Square	3	10.09	3.364	18.69	0.004
Melt Temperature*Melt Temperature	1	0.092	0.092	0.51	0.506
Holding Pressure*Holding Pressure	1	5.908	5.908	32.82	0.002
Injection Pressure*Injection Pressure	1	4.523	4.523	25.13	0.004
2-Way Interaction	3	11.40	3.800	21.11	0.003
Melt Temperature*Holding Pressure	1	4.900	4.900	27.22	0.003
Melt Temperature*Injection Pressure	1	0.100	0.100	0.56	0.490

Holding Pressure*Injection Pressure	1	6.400	6.400	35.56	0.002
Error	5	0.900	0.180		
Lack-of-Fit	3	0.300	0.100	0.33	0.808
Pure Error	2	0.600	0.300		
Total	14	51.493			
Model Summary					
S	R-sq	R-sq(adj)			
0.0134164	98.25%	95.11%			

In Table 4 shows the output from Minitab for the regression analysis of variance in the shrinkage response. In determining the regression significance test there is a linear relationship of the shrinkage response variable to the process parameters using the hypothesis:

$$H_0: \beta_1 = \beta_2 = \dots = \beta_k = 0$$

$$H_1: \beta_1 \neq 0 \text{ at least one } j$$

The Analysis of Variance (ANOVA) table uses F-value because it looks for variance where several samples are owned from several populations with the same sign. Variations of all the independent variables, namely the parameters of the melt temperature injection process, holding pressure, and injection pressure both linear, square and 2-way interaction on dependent shrinkage variables using the *distribution F* table. Estimation of the second order model of shrinkage response can be known for parameters the process with the value that can be explained  $R^2 = 98.25\%$ . The output data from ANOVA can be concluded that the second order estimation model (square) is appropriate. To examine whether or not a second-order model is significant, it can be seen the p-value of regression in table 5. The p-value of 0.001 is smaller ( $<$ ) than the value of  $\alpha$  of 5%, this means that the process parameters have a significant impact on the second-order model.

**Table 5.** Estimation of Model Equation Coefficient

Coded Coefficients

Term	Coef	SE Coef	T-Value	P-Value
Constant	1.25000	0.00775	161.37	0.000
Melt Temperature	0.04750	0.00474	10.01	0.000
Holding Pressure	-0.03500	0.00474	-7.38	0.001
Injection Pressure	-0.01250	0.00474	-2.64	0.046
Melt Temperature*Melt Temperature	0.00500	0.00698	0.72	0.506
Holding Pressure*Holding Pressure	-0.04000	0.00698	-5.73	0.002
Injection Pressure*Injection Pressure	-0.03500	0.00698	-5.01	0.004
Melt Temperature*Holding Pressure	0.03500	0.00671	5.22	0.003
Melt Temperature*Injection Pressure	-0.00500	0.00671	-0.75	0.490
Holding Pressure*Injection Pressure	-0.04000	0.00671	-5.96	0.002

In Table 5. shows the testing of each regression coefficient on the resulting regression model, it can occur in adding variables or deleting one or more existing variables. Adding variables to the second-order regression model can cause the sum of square for regression to increase and the number of squared errors (error sum of square) to decrease. Adding variables that do not affect the estimation of the model can increase the mean square error, thereby reducing the usefulness of estimating the model. Hypothesis to test the significance of each individual regression coefficient:

$$H_0: \beta_j = 0$$

$$H_1: \beta_j \neq 0$$

If  $H_0: \beta_j = 0$  is failed to be rejected, then it can be indicated that  $x_j$  can be removed from the estimated model.

In estimating the second-order regression model in Table 5 the factors that did not have a significant impact included quadratic (square) *melt temperature\*melt temperature* and interaction factor (2-way interaction) *melt temperature\*injection pressure*. The p-value of the factor is  $\alpha > 0.05$ , so that these factors can be eliminated. Parameters that do not have a significant impact can be removed and then re-analyzed using Minitab 18, these results are shown in Table 6.

**Table 6.** Results Of ANOVA Eliminate the significant process parameters

Analysis of Variance

Source	DF	Adj SS (10 <sup>-4</sup> )	Adj MS (10 <sup>-3</sup> )	F-Value	P-Value
Model	7	50.40	7.200	46.14	0.000
Linear	3	29.10	9.700	62.16	0.000
Melt Temperature	1	18.05	18.05	115.6	0.000
Holding Pressure	1	9.800	9.800	62.80	0.000
Injection Pressure	1	1.250	1.250	8.01	0.025
Square	2	10.00	5.001	32.05	0.000
Holding Pressure*Holding Pressure	1	6.058	6.058	38.82	0.000
Injection Pressure*Injection Pressure	1	4.651	4.651	29.80	0.001
2-Way Interaction	2	11.30	5.650	36.21	0.000
Melt Temperature*Holding Pressure	1	4.900	4.900	31.40	0.001
Holding Pressure*Injection Pressure	1	6.400	6.400	41.01	0.000
Error	7	1.092	0.156		
Lack-of-Fit	5	0.492	0.098	0.33	0.864
Pure Error	2	0.600	0.300		
Total	14	51.493			

Model Summary

S	R-sq	R-sq(adj)
0.0124918	97.88%	95.76%

Coded Coefficients

Term	Coef	SE Coef	T-Value	P-Value
Constant	1.25308	0.00600	208.82	0.000
Melt Temperature	0.04750	0.00442	10.76	0.000
Holding Pressure	-0.03500	0.00442	-7.92	0.000
Injection Pressure	-0.01250	0.00442	-2.83	0.025
Holding Pressure*Holding Pressure	-0.04038	0.00648	-6.23	0.000
Injection Pressure*Injection Pressure	-0.03538	0.00648	-5.46	0.001
Melt Temperature*Holding Pressure	0.03500	0.00625	5.60	0.001
Holding Pressure*Injection Pressure	-0.04000	0.00625	-6.40	0.000

To prove the results in Table 6 the tests are carried out as follows:

1. Model suitability testing

To find out the suitability of the model above, then testing includes:

a.  $R^2$  Test

Table 3.5 shows that as many as 97.88% of the response variations can be explained by this estimation.

b. Lack Of Fit Test

The lack of fit test for the model obtained p-value of 86.4% or greater than the degree of significance  $\alpha = 5\%$ . This value indicates that the regression model is appropriate.

2. Testing Of Regression Coefficients

a. Individually

In testing with  $\alpha < 0.05$ , injection process parameters in table 3.5 have p-value  $< 0.05$ . this represents a process parameter that has a significant effect on shrinkage response.

b. Simultaneously



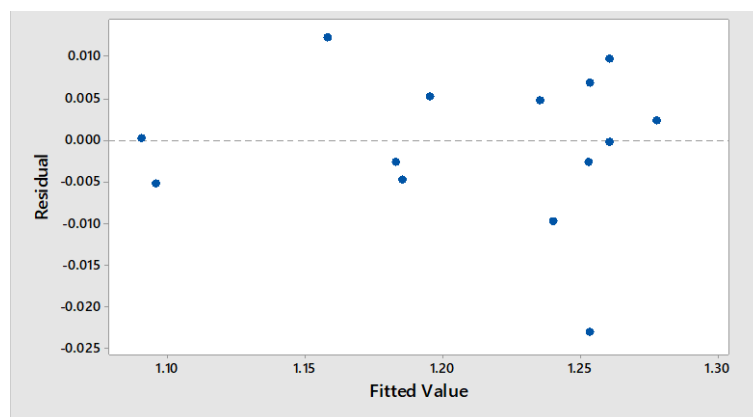
Based on data Table 3.5 shows  $F_{\text{count}} = 46.14$ , while  $F_{\text{table}} = F(7; 14; 0.05) = 3.53$ . The value of  $F_{\text{count}} > F_{\text{table}}$  can be concluded that the process parameters have a significant impact on the second-order regression model.

### 3. Testing Residual Assumptions

To check the adequacy of the model, not only do testing lack of fit, there is one test, namely residual analysis. Residual analysis is iudentik, independent, and normally distributed with mean zero on certain variations. Some tests carried out using residual analysis:

#### a. Identity

In Figure 6 below, it can be seen that the plots between residuals and fitted values appear randomly around the gold price and do not form a certain pattern. This shows that identical (homogeneous) residual variance is fulfilled.



**Figure 6.** Residual graph vs. fitted value shrinkage BSS in the direction of flow

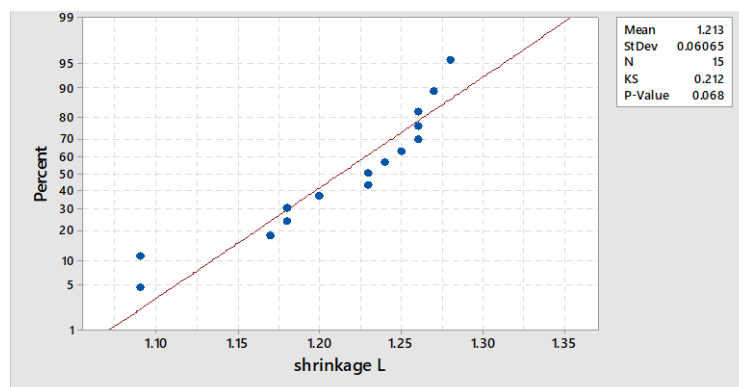
#### b. Normality

Testing of residual normal assumptions was carried out by the Kolmogorov-smirnov test. The hypothesis used for normal testing:

$H_0$  : Residual regression models are normally distributed.

$H_1$  : Residual regression models are not normally distributed.

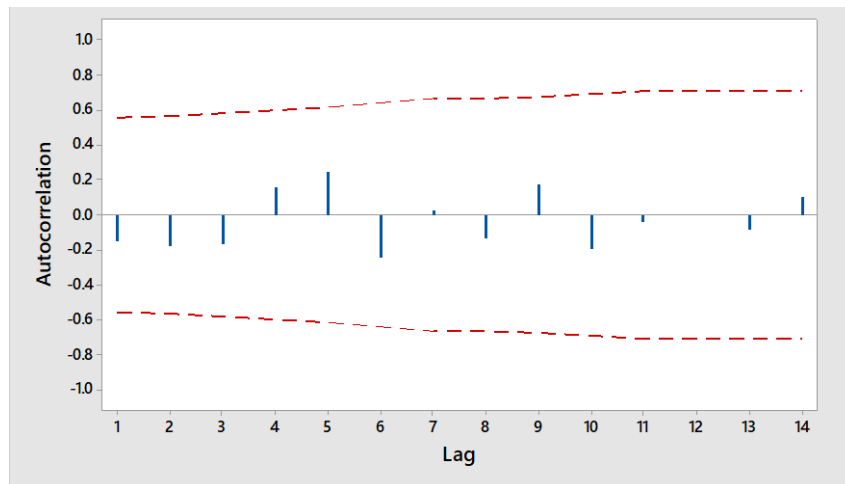
Testing of residual normal assumptions was carried out by the Kolmogorov-smirnov test. The test results with a degree of significance  $\alpha = 5\%$  are shown in Figure 7. The Kolmogorov-Smirnov ( $KS_{\text{hitung}}$ ) statistical value is 0.212, while the Kolmogorov-Smirnov value of the table ( $KS_{\text{tabel}}$ ) is  $\alpha = 0.05$  and the number of observations is 0.34. Because  $KS_{\text{hitung}} < KS_{\text{tabel}}$  then the residual of the estimation model has normal distribution.



**Figure 7.** Graph Normal Probabbility Shrinkage

c. Independence

Observations on this research have been carried out independently and there was no correlation between observations. This is evidenced by the AutoCorrelation Function (ACF) plot graph in Figure 8. the graph shows that all correlations are still in the interval.



**Figure 8.** Graph of AutoCorrelation Function for Shrinkage in the direction flow

With several tests that have been done above, it is known that the model that satisfies the relationship between the injection molding process parameters, namely melt temperature, holding pressure, injection pressure to the shrinkage response in sisal fiber biocomposite material can be formulated with the following empirical equation:

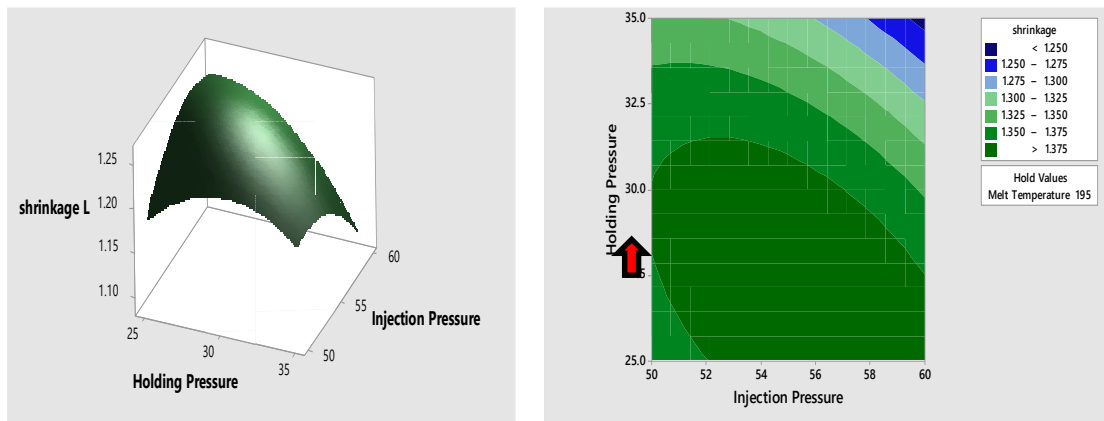
$$\hat{Y}_{shrinkage\ BSS} = 1.25308 + 0.0475x_1 - 0.035x_2 - 0.0125x_3 - 0.04038x_2^2 - 0.03538x_3^2 + 0.035x_1x_2 - 0.04x_2x_3.$$

with  $\hat{Y}$  : Estimated model value for shrinkage response  
 $x_1$  : Parameters of the process of *melt temperature*  
 $x_2$  : Parameter of the *holding pressure*  
 $x_3$  : Parameter of the *injection pressure*

#### 2.4. Surface Plot Graph

The graph of the surface plot shown in Figure 9 [left] with the x axis of the injection pressure process parameter and the y axis of the holding pressure process against the shrinkage response. Figure 9 shows the injection injection and holding pressure injection process parameters for the shrinkage response in sisal-glass fiber biocomposite material.

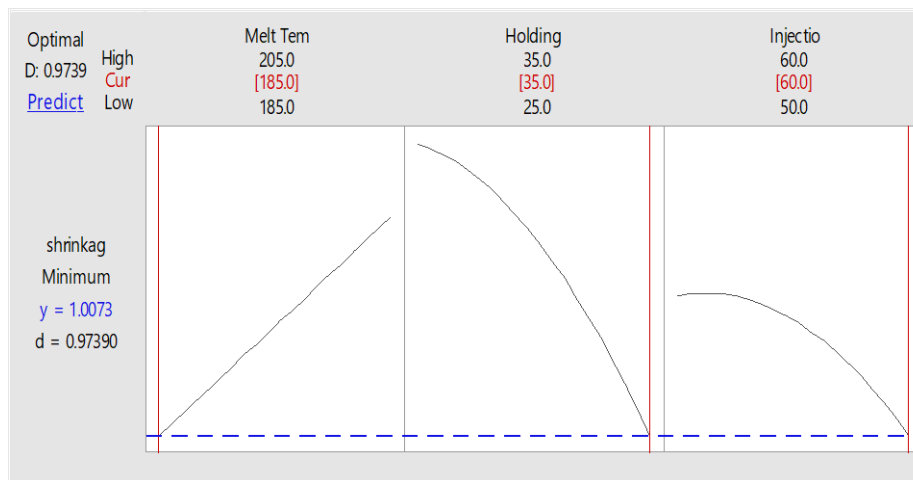
The surface plot is in the form of a hill with a process melt temperature parameter of 195°C, while a contour plot graph is shown in Figure 9 [right]. The percentage point that shows the smallest shrinkage value is in the injection pressure process parameter of 60 bar and holding pressure of 35 bar



**Figure 9.** Graph of surface [left] and contour plot [right] shrinkage vs Injection pressure, Holding pressure in the direction of flow

### 2.5. Parameter Optimazion

The desirability function approach is used for the melt temperature (0C) process parameter, holding pressure (bar), injection pressure (bar) to produce an  $\hat{Y}$ shrinkage (%) response. The desirability function uses The smaller is better with the lower sisal-glass fiber biocomposite material = 1.09%, upper = 1.28%, weight = 1. The mathematical model is processed using Minitab 18 software, so that a graph of combination parameters can produce optimal shrinkage response.



**Figure 10.** Graph of the combination of process parameters that produces the optimal response of the flow direction Sisal Biocomposite Fiber material

The analysis in Figure 10 shows the combination of process parameter settings for the flow of sisal biocomposite material. Process parameters to get the optimum percentage value, namely to melt 185°C temperature, hold pressure 35 bar, and injection pressure 60 bar. This combination is predicted to get a shrinkage response of 1.0073% with a desirability value of 1.00.

The graph in Figure 10, where the higher the pressure is given, the smaller the percentage shrinkage value will be. The holding pressure graph has a trendline that is more influential than the parameters of the melting temperature and injection pressure process. It is caused by the fact that during the freezing process in the cavity, the plastic compression properties result in high shrinkage values. This phenomenon can be prevented by providing high holding pressure so as to minimize the compressibility of plastic. Uniform and high pressure can control and produce a low shrinkage percentage value. It can be useful to determine the geometry tolerance of the specimen to be produced.

### 2.6. Confirmation Trial

Confirmation experiments were carried out by recovering data from the HAITIAN MA 900 / 260e injection molding machine. Process parameters used are based on optimization charts in BSS material Figure 11. The results of the specimens were then re-measured to determine the shrinkage percentage value of each material



**Figure 11.** Measurement of confirmation of the flow direction of Sisal Fiber Biocomposite material

In Figure 11 is one of the confirmation tests for measuring the shrinkage value percentage in the 1st experiment of sisal-glass fiber biocomposite material in the direction of the 1<sup>st</sup> replication plastic flow at the 4<sup>th</sup> point. Confirmation experiments at optimum values were carried out 3 times and replicated 3 times. The results of the confirmation experiment are shown in Table 7.

**Table 7.** Confirmation Experiment Results for optimum values

Material	Percobaan Ke-	Parameter Proses			Respon Shrinkage (%)
		MT (°C)	HP (bar)	IP (bar)	
Biocomposite	1	185	35	60	1.03
	2	185	35	60	1.06
	3	185	35	60	1.06

In Table 8 the value of the shrinkage percentage for sisal fiber biocomposite material in the optimization conditions is 1.05% smaller than the shrinkage percentage resulting in the initial condition of 1.09%. The reduction in the percentage of shrinkage value is very significant at 3.67% of the initial price. The percentage value is still greater than the value of optimization based on statistical analysis that is equal to 1.0073%. The difference between statistical analysis and optimization conditions can be caused by interference with the injection process parameters in addition to controlled process parameters but not too significant.

**Table 8.** Comparison of optimization results with initial conditions

Material		Parameter proses			Respon Shrinkage (%)
		TM(°C)	HP(bar)	IP (bar)	
Biocomposite	Kondisi Awal	195	35	60	1.09
	Analisa Statistik	185	35	60	1.0073
	Kondisi Oprimasi	185	35	60	1.05

### 3. Conclusion

From the results of the experimental studies and analyzes that have been carried out, there are some conclusions, namely:

1. The parameters of the injection molding process melt temperature, holding pressure, and injection pressure have a very significant effect on the response of sisal-glass fiber biocomposite material due to the p-value of  $0.001 < \alpha$ .
2. The effect of melt temperature process parameters on shrinkage response can be caused by density and Coefficient of Thermal Expansion, while the parameters of holding pressure and injection pressure can be attributed to the compressibility of sisal fiber biocomposite material.
3. The parameters of the injection molding process melt temperature ( $x_1$ ), holding pressure ( $x_2$ ), and injection pressure ( $x_3$ ) have the regression equation based on the following response surface:

$$\hat{Y}_{shrinkage\ BSS} = 1.25308 + 0.0475x_1 - 0.035x_2 - 0.0125x_3 - 0.04038x_2^2 - 0.03538x_3^2 + 0.035x_1x_2 - 0.04x_2x_3.$$

4. Based on the help of MINITAB 18 process parameters to obtain a minimum shrinkage value in sisal-glass fiber biocomposite material melt temperature 185°C, holding pressure 35 bar, injection pressure 60 bar. Confirmation experiments produced a shrinkage value of 1.05%

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