Characterization of Flexural Strength, Warpage and Shrinkage of Polypropylene-Nanoclay-Nanocomposites Blend with Gigantochloa Scortechinii.

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Abstract. This paper presents the characterization of flexural strength, warpage and shrinkage of reinforcement gigantochloa scortechinii fibre with the blend of polypropylene and nanoclay. The content of fiber was fixed at 0 wt.%, 3 wt.% and 6 wt.% in uniform increment. The selected injection moulding processing conditions were packing pressure, melt temperature, screw speed and filling time. The quality factors that need to be improved upon the characterization were flexural strength, warpage and shrinkage. This research started by drying the Gigantochloa Scortechinii fibres at 120°C. After that, 3 wt.% of the fibres were mixed with 81 wt.% of polypropylene, 15 wt.% of polypropylene grafted maleic anhydride (compatibilizer) and 1 wt.% of nanoclay. Samples with 6 wt.% of fibers were also prepared for comparison purpose. The mixing process was performed by using Brabender Lab-Compounder KETSE 20/40 and the pallets were produced using used Brabender® pelletizer with diameters of 1 to 4 mm. The optimisation process was accomplished by adopting the Taguchi L9 orthogonal array method. According to the results, for 0 wt.% fibre, the flexural strength is 30.0082 MPa, the warpage is 0.0030 mm and the shrinkage is 0.0004 mm at packing pressure 40%, melt temperature 165°C, filling time 2 seconds and screw speed 35%. As for the result of 3 wt.% fibre, the flexural strength is 32.2477 MPa, the warpage is 0.0067 mm and the shrinkage is 0.0004 mm at packing pressure 35%, melt temperature 165°C, filling time is 1 second and screw speed is 30%. While for the 6 wt.% fibre, the results of the flexural strength is 36.9084 MPa, the warpage is 0.0067 mm and the shrinkage is 0.0004 mm at packing pressure is 35%, melt temperature 165°C, filling time is 2 seconds and screw speed is 30%. The existence of Gigantochloa Scortechinii fibre was also proven to effect significantly towards flexural strength with 6% increasing value ordering from 0 wt.% to 6 wt.% fibre. while, the warpage value increasing from 0.0030 mm to 0.0067 mm and the shrinkage was state remain same value for 0.3 and 6 wt.% of fibre causes the result difference 0.1x10⁻² mm for each . In conclusion, the characterization of the flexural strength, the warpage and the shrinkage of the polypropylene-nanoclay-Gigantochloa Scortechinii had been achieved, and the existence of fibre obviously giving a promising manufacturing opportunity to improve the quality of the injected moulding products.
1. Introduction

Injection moulding was highly viewed in the industry due to it can produce large quantities of artefacts with reasonable time and cost savings. However, an effective control of injection moulding parameter is crucial, since each of these items is dependent on each other and any changes shall affect the outcomes [1].

These parameters can be controlled and improved via systematic approaches, such as Taguchi Optimisation Method. Some of the established researchers have obtained promising findings by using this method. Experiments have been performed towards various types of material such as polypropylene [2], acrylonitrile butadiene styrene [3], and high density polyethylene [4]. Several past researches also have been performed by the author through simulation [5], and towards different product such as snap fit samples [6] and the strength of gate weld lines [7].

Nanocomposites are compound materials in which the matrix material is strengthened with one or more distinct nanomaterials in demand to improve mechanical, tribological, physical, and thermal properties. More recently, nanocomposites have attracted remarkable attention from science as well as industry. The numbers of applications of nanocomposites have been growing at a rapid rate. Due to their unique mechanical, electrical, and thermal properties, worldwide production is estimated these applications to exceed 600,000 tones and is set to cover the following key areas in the next five to ten years, such as drug delivery systems, anti-corrosion barrier coatings, UV protection gels, lubricants and scratch free paint, new fire retardant materials and new scratch/abrasion resist materials. One of the most popular nanocomposite was polypropylene-nanoclay polymer nanocomposites. The details of preparation, properties and the application of these nanocomposites have been summarized [8]. These materials can be processed through injection moulding. A research had been made to control shrinkage and warpage towards hinges samples [9]. However, the mixing of other fillers such as natural fibres, and the selection of other parameter setting have been always the issues that affect the properties and quality of the mould products [10].

In this study, Gigantochloa Scortechinii was used as the reinforcement material in the polypropylene-nanoclay system. Gigantochloa Scortechinii or locally known as bamboo fibres can be used as reinforcement in polymer, with the aid of compatibilizer, considering mechanical properties, processing and environmental aspects. Based on previous research, the existence of bamboo fibre content had a positive effect on tensile strength, flexural modulus and strength, and negative effect on elongation at break and energy to break. Bamboo fibres can be successfully used as reinforcement in polymer, with the aid of compatibilizer, considering mechanical properties, processing, environmental aspects and products [11,12].

2. Experiment Works

The materials used in this study was divided into two different composites, which was the first mixture contained 3 wt. % of Gigantochloa Scortechinii fibre and the other one was 6 wt.%. The selected compounding materials for 3 wt. % of bamboo fibre composites were 81 wt.% of polypropylene, 15 wt.% of compatibilizer which is polypropylene-grafted-maleic anhydride (PPgMA) and 1 wt. % of nanoclay, while the mixture for 6 wt.% of bamboo fibre composites were 78wt.% of polypropylene, 15 wt.% of compatibilizer which is polypropylene-grafted-maleic anhydride (PPgMA) and 1 wt.% of nanoclay were used to determine whether there are any significant effects the content of fibre towards the quality of samples. The fibres need to be pre-heated at temperature 120°C to optimize moisture content of fibres, small value of moisture content are the best for the strength fibers13. The process of mixing was made by using a twin screw Brabender Lab-Compounder KETSE 20/40 as shown in Figure 1 and pallets were produced using used Brabender® pelletizer with diameters in range 4 mm for injection moulding process shown in Figure 2.
Cavity shape and Injection Moulding: Figure 3 displays the cavity shape chosen for this project was plate rectangular shape (flexural test) based on ISO 178 standard\(^{14}\). The mould and injection moulding machine was available in Polymer and Ceramic Laboratory, UTHM. Figure 4 shows the type of injection moulding used for this research, which is Nissei NP7-1F (Screw diameter: 19mm, Screw speed: 350rpm, clamp force: 69kN).

Measuring Flexural Strength, Warpage and Shrinkage: The characterization that’s need to improved in this research were flexural strength, warpage and shrinkage. These defects need to be analysed towards the specimens after the injection moulding process was performed. The flexural strength of the trials was measured using a universal testing machine based on ISO 178 three bending points. Figure 5(a) shows the Universal Test Machine Model AG-1 (SHIMADZU) 10kN and Figure
5(b) shows the flexural strength test of 3 point bending experiment and the screen monitoring for the graph result.

![Universal Test Machine](image1)  ![Flexural Strength Test and Monitoring Screen](image2)

Figure 5 (a) : Universal Test Machine Model AG-1 (SHIMADZU) 10kN  Figure 5 (b) : The Flexural Strength Test and Monitoring Screen of The Flexural Test.

Figure 6 shows the parameter was used in this research for flexural strength testing. The thickness, length and width evaluate from the measured of sample using vernier callipers. The Eq.1 was used to find out the value of velocity.

\[ \text{Velocity} = \frac{Z(L^2)}{6d} = \frac{0.01 (70^2)}{6(4)} = 2.0417 \text{ mm/min} \]  

(Eq.1)

To measure warpage, the specimen have to be cast out from the mould, and need to be cleared on or after flashing and runner before measuring the average thickness of the sample. After finding the specimen deflection required data, the warpage result can be calculated using the Eq. 2.

\[ Z = h - t_a \]  

(Eq. 2)

From Eq. 2, Z is the warpage of the sample, h is refer to maximum height and ta is the average of sample thickness in millimetre.

The shrinkage of the samples was measured using two equations, first find the value of \( L_c \) used (Eq.3) then to find the value of shrinkage was using (Eq. 4).

\[ L_c = L \left[ 1 + \alpha (T_{\text{mould}} - T_{\text{ambient}}) \right] \]  

(Eq.3)

\[ S = \frac{(L_c - L_m)}{L_c} \]  

(Eq.4)

From (Eq.3), \( L_c \) is the actual mould cavity length, \( \alpha \) is the coefficient of thermal expansion of tool steel with value \((6.45 \times 10^{-6}/^\circ\text{F})\), \( T_{\text{mould}} \) is the mould temperature, and \( T_{\text{ambient}} \) is the ambient temperature. In Eq.3, \( L_m \) is the average of actual sample length.
Factor selection, Level and Orthogonal Array. The Taguchi method orthogonal array that was chosen for this study was L934 (9 trials, 3 levels, and 4 factor processing conditions). This method was preferred in order to find the optimal values of the process to improve the quality characteristics. The parameter that were chosen are melt temperature, packing pressure, screw speed and filling time. These four factor processing with three different levels which was low (1), medium (2) and high (3). The factor selection and level have been stated in Table 1. The samples were injected mould from the experiment to be tested. There are 27 samples for 9 trials to be tested. The orthogonal array for this experiment, the details were stated in Table 1.

Table 1: Factor Selection and Level

| FACTOR              | LABEL | LEVEL |
|---------------------|-------|-------|
| Melt Temperature    | MT    | 165   |
|                     |       | 170   |
|                     |       | 175   |
| Packing Pressure    | PP    | 30    |
|                     |       | 35    |
|                     |       | 40    |
| Screw Speed         | SS    | 25    |
|                     |       | 30    |
|                     |       | 35    |
| Filling Time        | FT    | 1     |
|                     |       | 2     |
|                     |       | 3     |

Table 2 : Orthogonal Array

| TRIAL | MT (°C) | PP (%) | SS (%) | FT (s) |
|-------|---------|--------|--------|--------|
| 1     | 165     | 30     | 25     | 1      |
| 2     | 165     | 35     | 30     | 2      |
| 3     | 165     | 40     | 35     | 3      |
| 4     | 170     | 30     | 25     | 3      |
| 5     | 170     | 35     | 30     | 1      |
| 6     | 170     | 40     | 35     | 2      |
| 7     | 175     | 30     | 25     | 2      |
| 8     | 175     | 35     | 30     | 3      |
| 9     | 175     | 40     | 35     | 1      |

Measuring Signal to Noise Ratio and Optimisation: In this experiment, by adopting the Taguchi method, the signal to noise ratio need to be measured. The outcomes that need to be monitored in this study was the result of flexural strength, warpage and shrinkage which were influenced by the setting of four factor parameter. In addition, the optimised processing condition for the injection moulding will be the final findings. The signal to noise ratio for large the better quality characteristics was usually used for flexural strength analysis. On behalf of the flexural strength, the best result that's needed is, the more it can bend without fracture. It is to rise up the strength of the moulded measure. The equation for the larger the better as shows in (Eq. 5).

\[
\frac{S}{N} = -10 \log \left[ \frac{1}{N} \sum_{i=1}^{n} \frac{1}{y_i^2} \right]
\]  
(Eq.5)

For warpage and shrinkage as the wanted output. The formula for the signal to noise ratio for the smaller-the-better quality characteristics is also a logarithmic function. The equation for signal to noise (S/N) ratio specifically for smaller the better characteristics is stated in (Eq. 6).

\[
S/N = -10 \log \frac{1}{n} (\sum y^2)
\]  
(Eq.6)
3. Results And Discussions

Since different mixture percent content of GS were used to determine the significant effects of fibre towards the value of samples, it remained important to compare the significance of flexural strength, warpage and shrinkage towards each fibre content. Figure 6 in Figure 14 show the main effects plot for signal to noise ratio, for each fibre content respective to flexural strength, warpage and shrinkage.

![Main Effects Plot for SN ratios Flexural of 0wt% GS](image1)

Figure 6: The Flexural S/N ratio for 0 wt.% of GS

![Main Effects Plot for SN ratios Flexural 3wt% GS](image2)

Figure 7: The Flexural S/N ratio for 3 wt.% of GS
Figure 8: The Flexural S/N ratio for 6 wt.% of GS

Figure 9: The Warpage S/N ratio for 0 wt.% of GS
Figure 10: The Warpage S/N ratio for 3 wt.% of GS

Figure 11: The Warpage S/N ratio for 6 wt.% of GS
Figure 12: The Shrinkage S/N ratio for 0 wt.% of GS

Figure 13: The Shrinkage S/N ratio for 3 wt.% of GS
From Figure 6, 7 and 8, the data in Table 3 was constructed. Based from Table 3 for the optimization of flexural strength for all samples, the result shows the strength are increased according to the increased of GS. The lowest value was 30.0082 MPa for 0 wt. % GS, increased to 32.2477 MPa for 3 wt. % GS and increased to 12% for formulation 6 wt. % GS. The result also shows, the temperature to optimize the strength was same for all formulations, the packing pressure and screw speed for 0 wt. % GS are a high of 5%, comparable to 3wt. % GS and 6 wt.% GS. For 3 wt.% GS the filled times used are less than 2 seconds.

Table 3 : Optimization Flexural Strength for all wt.% GS result

| Fibre Content (wt.%) | Temperature (°C) | Pressure (%) | Speed (%) | Filling time (s) | Max Stress (MPa) |
|----------------------|------------------|--------------|-----------|-----------------|-----------------|
| 0                    | 165              | 40           | 35        | 2               | 30.0082         |
| 3                    | 165              | 35           | 30        | 1               | 32.2477         |
| 6                    | 165              | 35           | 30        | 2               | 36.9084         |

From Figure 9, 10 and 11, the data in Table 4 was constructed. Based on Table 4, the warpage for all wt.% of GS, the result shows the warpage are increased, according to the increased of 3 wt% of GS. The lowest value was 0.0030 mm for 0wt.% GS, increased to 0.0067 mm for 3 wt.% GS and 6 wt.% GS. The result of warpage for 3 wt.% GS and 6 wt.% GS are same. The result warpage for 3 wt.% and 6 wt.% of GS may effected of the surrounding during cooling processes of sample and the thermal warpage resulting from unbalanced cooling in a at plate of amorphous polymer, which discovered that the warpage phenomenon of the molded product results from the bending moment outstanding to the asymmetrical stress spreading over the thickness of the plastic parts13.
Table 4: Warpage for all wt.% GS

| Fibre Content (wt.% | Temperature (°C) | Pressure (%) | Speed (%) | Filling time (s) | Warpage (mm) |
|---------------------|------------------|--------------|-----------|-----------------|--------------|
| 0                   | 165              | 40           | 35        | 2               | 0.0030       |
| 3                   | 165              | 35           | 30        | 1               | 0.0067       |
| 6                   | 165              | 35           | 30        | 2               | 0.0067       |

From Figure 12, 13 and 14, the data for Table 5 was constructed. Based from Table 5, the shrinkage for all wt.% of GS formulations, the result shows the shrinkage are same for all formulations after optimization. The value was 0.0004 mm for 0 wt.% GS, 3 wt.% GS and 6 wt.% GS.

Table 5: Shrinkage for all wt.% GS

| Fibre Content (wt.% | Temperature (°C) | Pressure (%) | Speed (%) | Filling time (s) | Shrinkage (mm) |
|---------------------|------------------|--------------|-----------|-----------------|----------------|
| 0                   | 165              | 40           | 35        | 1               | 0.0004         |
| 3                   | 165              | 35           | 30        | 1               | 0.0004         |
| 6                   | 165              | 35           | 30        | 2               | 0.0004         |

Therefore, it can be summarized that from the data in Table 3, 4 and 5 the best temperature, pressure, speed and filling time for optimum result of flexural strength, warpage, shrinkage were achieved. The optimization by adopting the Taguchi method used orthogonal array in Table 3, 4 and 5 were obtained by analysing the S/N ratio showed in Figure 6 to figure 14, whereby the maximum value of signal to noise ratio, flexural strength and the minimum value of warpage and shrinkage can be attained by using the selected processing condition to achieve the optimization processing condition with high flexural strength result, with the lowest result of warpage and shrinkage in injection moulding. Figure 15, 16 and 17 show the comparison results before and after optimization for each flexural strength, warpage and shrinkage.

4. Conclusion

As for the conclusion, the effect of the defect and the optimum processing condition of nanocomposites blend towards fibre bamboo had been achieved, by adopting the Taguchi method, whereby the maximum flexural strength, and the minimum value of warpage and shrinkage can be attained by using the selected processing condition. Based on the results, it can be concluded that, for 0 wt.% GS, the flexural strength is 30.0082 MPa, the warpage is 0.0030 mm and the shrinkage is 0.0004 mm at packing pressure 40%, melt temperature 165°C, filled time 2 seconds and screw speed 35%. For the result 3 wt.% GS, the flexural strength is 32.2477 MPa, the warpage is 0.0067 mm and the shrinkage is 0.0004 mm at filled time is 1 seconds, packing pressure is 35%, screw speed is 30% and melt temperature 165°C. While for the 6 wt.% GS, the results of the flexural strength is 36.9084 MPa, the warpage is 0.0067 mm and the shrinkage is 0.0004 mm at screw speed is 30%, melt temperature 165°C, packing pressure is 35%, and filled time is 2 seconds. The existence of Gigantochloa Scortechinii fibre was also proven to affect significantly towards flexural strength, warpage and shrinkage. In conclusion, the characterization of the flexural strength, the warpage and the shrinkage of the polypropylene-nanoclay-Gigantochloa Scortechinii had been accomplished, and the presence of fibre obviously providing a promising manufacturing opportunity to improve the value of the injected moulding products.
Figure 15: Result Before and After Optimization for Flexural Strength

Figure 16: Result Before and After Optimization for Warpage
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References
[1] Mehat .N. M. , Kamaruddin.S : Optimisation of mechanical properties of recycled plastic products via optimal processing parameters using the Taguchi method, journal of J. Mater. Process. Technol, vol. 211, no. 12, pp. 1989–1994 (2011)
[2] Ozcelik . B, Ozbay .A, Demirbas .E : Influence of injection parameters and mold materials on mechanical properties of ABS in plastic injection molding, journal of Int. Commun. Heat Mass Transf., vol. 37, no. 9, pp. 1359–1365(2010)
[3] Ozcelik .B :Optimisation of injection parameters for mechanical properties of specimens with weld line of polypropylene using Taguchi method, journal of Int. Commun. Heat Mass Transf., vol. 38, no. 8, pp. 1067–1072 (2011)
[4] Ibrahim .M. H. I, Zainol .M. Z, Othman . M. H, Amin . A. M, Asmawi .R :Optimisation of Processing Condition Using Taguchi Method on Strength of HDPE-Natural Fibres Micro Composite, journal of in Applied Mechanics and Materials, vol. 660, pp. 33–37 (2014)
[5] Othman .M. H, Shamsudin . S, Hasan . S :The effects of parameter settings on shrinkage and warpage in injection molding through Cadmould 3D-F simulation and Taguchi method, journal of in Applied Mechanics and Materials, vol. 229, pp. 2536–2540 (2012)
[6] Othman .M. H :The Optimisation of Parameter Settings Towards Quality of Snap Fit Samples journal of Polypropylene-Nanoclay Nanocomposites, in Malaysia University Conference Engineering Technology (2014)
[7] Othman M. H, Shamsudin .S, Hasan .S, Rahman . M. N. A.:The effects of injection moulding processing parameters and mould gate size towards weld line strength, journal in Advanced Materials Research, vol. 488, pp. 801–805 (2012)
[8] Othman .M. H, Sulaiman .H, Wahab .M :A Review of Polypropylene Nanoclay Nanocomposites: Preparation, Properties and Applications, journal in Applied Mechanics and Materials, vol. 465, pp. 944–948 (2014)

[9] Muhammad .W, Azrina .W. N, Othman .M. H, Hasan .S, Ruslee .M. F :A study of injection moulding optimum parameters to control shrinkage and warpage of polypropylene-nanoclay hinges samples, (2013)

[10] Rajesh .J. J, Soulestin .J, Lacrampe .M. F, Krawczak .P :Effect of injection molding parameters on nanofillers dispersion in masterbatch based PP-clay nanocomposites, EXPRESS Polym. Lett., vol. 6, no. 3, pp. 237–248 (2012)

[11] Bonse, B. C., Mamede, M. C. S., da Costa, R. A., & Bettini, S. H. P :Effect of compatibilizer and bamboo fibre content on the mechanical properties of PP-g-MA compatibilized polypropylene/bamboo fibre composites. In Proceedings of the Polymer Processing Society 26th Annual Meeting~ PPS-26~ July (pp. 4-8). (2010)

[12] Chanda .M, Roy .S. K : Industrial polymers, specialty polymers, and their applications, vol. 74. CRC press (2008)

[13] Huang, M. C., & Tai, C. C.. Effective factors in the warpage problem of an injection-molded part with a thin shell feature. Journal of Materials Processing Technology, 110(1), 1–9. doi:10.1016/S0924-0136(00)00649-X (2001)

[14] EN ISO 178 :Plastics.Determination of flexural properties (2012)