OPTIMIZATION OF COMPRESSION MOULDING PARAMETERS FOR MULTI FILLER POLYMER COMPOSITE USING TAGUCHI METHOD

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ABSTRACT: Polymer Electrolyte Membrane Fuel Cells (PEMFC) performance depends on the properties of bipolar plates (BP). In order to produce the best performance of BP, compression moulding parameters need to be optimized. This study determined the compression moulding parameters of Graphite (G) / Carbon Black (CB) / Carbon Fiber (CF) / Polypropylene (PP) composites using Taguchi method (TM) in order to optimize the properties of BP plate. L_9 Orthogonal Array with four factors and three levels was chosen as a design of experiment for G/CB/CF/PP composition with a weight percentage of 50/25/5/20. The factors selected for this study were heating temperature, load, preheating pressing time and pressing time. The electrical conductivity value of each sample was analyzed by signal to noise ratio using TM with the larger-the-better condition in order to determine the optimum parameters. Confirmation experiment was conducted to validate the optimum parameters obtained from the TM. The electrical conductivity result of G/CB/CF/PP composites for confirmation experiment was 393.49 S/cm and it was higher than nine trials and the TM predicted value. Hence, the optimum parameters of compression moulding can be obtained using TM to improve the electrical conductivity of G/CB/CF/PP composites. TM is an effective way to get the optimal moulding parameters for G/CB/CF/PP composites and is very useful to fabricate bipolar plate for PEMFC.

KEYWORDS: *Taguchi Method; Graphite; Carbon Black; Carbon Fibre; Polypropylene*

1.0 INTRODUCTION

The fabrication method of polymer composite affects the electrical and mechanical properties of the composites through dispersion and orientation of particles during a fabrication process [1]. The most common fabrication method used to produce BP plate is compression moulding. Compression moulding parameters are vital in producing better product particularly for BP. Compression moulding parameters also affect electrical conductivity of BP plate composites[1-4]. In conductive polymer, matrix acts as a binder and fillers as reinforcement or conductive and they are mixed together to produce a composite. Conductive polymer composites (CPCs) are composites that contain electrical conductivity properties [4]. For BP application, the electrical conductivity must be higher than 100 S/cm in accordance to the U.S. Department of Energy (USDoE) [3-6]. G/CB/CF/PP composites must achieve the required targets specified by the USDoE for BP as shown in Table 1 [3-10].

Table 1: Requirement properties of the bipolar plate (USDoE target [3-10]

Property	Value
Electrical conductivity	> 100 Scm^{-1}
Thermal conductivity	> 10 $Wm^{-1}K^{-1}$
Flexural strength	> 25 MPa
Shore Hardness	> 50
Bulk Density	< 5 gcm^{-3}

Nowadays, CPC becomes one of the most attractive options especially for researchers in the semiconductor industry. CPC can be used in various applications such as sensors, batteries, bipolar plates in fuel cell systems, electromagnetic interference (EMI) and radio frequency interference (RFI) [5,11-12]. Carbon based CPC is an ideal material to produce BP as it warrants good mechanical properties with low cost. Among the carbon based materials that are often used are CB and CF [4,12]. Companies can save in terms of fabrication cost, overcome corrosion and resistance problems, mechanical properties increment and fabrication easiness through polymer composites usage [11-12]. Polymer composites can also improve fuel cell performance for transportation sector applications [13–16].

To optimize the compression moulding parameters, TM with Orthogonal Array (OA) of L9 was used and the results were then transformed into Signal to Noise (S/N) ratio. With S/N ratio, the optimum hot compression moulding parameters could be predicted. There are three quality characteristics of TM which are the larger-the-better (LTB), the smaller-the-better (STB) and the nominal-the-best (NTB). LTB was chosen as data analysis tool because the objective of this study was to obtain the highest electrical conductivity of G/CB/CF/PP composites. S/N ratio was also used to represent the quality characteristics. Signal refers to an expected output quality characteristic of the moulding process parameters whereas noise refers to an unexpected quality characteristic [4]. Then, to determine the optimum moulding parameters from the parameter design, the confirmation experiment was conducted. TM using S/N ratio is proposed as a design of experiment (DOE), which minimizes the number of experiments for optimization process and it is also proven to be an effective way to produce high quality composites with low cost and short duration of time [5]. Therefore, in this study, G, CB and CF were used as fillers to produce better composites with higher electrical conductivity and improved mechanical properties. Besides, PP was selected as a binder.

The purpose of this study was to obtain the optimum compression moulding parameters such as heating temperature, load, preheating pressing time and pressing time using TM to produce CPC BP with higher electrical conductivity and better mechanical properties according to the USDoE requirements.

2.0 METHODOLOGY

2.1 Materials

PP grade Titan 600 was used in this study as the polymer matrix which was purchased from the Polypropylene Malaysia Sdn. Bhd. The main conductive filler was G powder, the second filler was CB powder and another filler was chopped CF purchased from Zoltek Industries. A comparison of the properties among G, CF, CB and PP is shown in Table 2.

Table 2: Properties of filler materials

Material	G	CF	CB	PP
Grade	3243	Chop Flake	5303	Titan
Density (gcm^{-3})	1.74	1.72	1.7	0.91
Thermal stability (^{o}C)	350-400	2000	3000	175-220
Size (μm)	≤60	7.2	≤5	250
Resistivity	1295×10^{8} Ω m	0.00155 Ω cm	0.314 Ω cm	1× 1014 Ω m

2.2 Design of Experiment (DOE)

In this study, TM with the OA of L_9 (3^4) was used to obtain the optimum compression moulding parameters and the results were then changed into S/N ratio. With S/N ratio, the optimum hot compression moulding parameters could be predicted. Then, the confirmation experiment was conducted to determine the optimum parameters obtained from the TM design. TM analysis using S/N ratio is proposed as a DOE with the minimum number of experiments to optimize the process. This approach has been proven to be an effective way to produce high quality composites with low cost and short duration of time [20-22].

In order to obtain optimized results for G/CB/CF/PP composites, TM with OA of L9 (3^4) was carried out using Minitab software. The optimum hot compression moulding machine parameters were obtained through TM. The composites were fabricated with optimum parameters of compression moulding machine to get the optimized result. Only one composition ratio was involved in this process namely 5 wt% of CF, 25 wt% of CB, 50 wt% of G and 20 wt% of PP.

OA of L_9 consisted of four factors namely temperature (A), pressure (B), preheating time (C) and pressing time (D) with three levels as depicted in Table 3 while OA of L_9 (3^4) for TM is shown in Table 4 with the response of electrical conductivity (E).

Table 3: The compression moulding factors for OA of L_9 (3^4)

Factors	**Variables**	**Level**		
		1	**2**	**3**
A	Temperature (°C)	175	180	185
B	Pressure (ton)	5	10	15
C	Preheating time (min)	5	7	10
D	Pressing times (min)	5	10	15

Table 4: OA of L_9 (3^4) for Taguchi Method

	Factors				Response
No. of Trial	**Temperature**	**Pressure (Tons)**	**Preheat time (min)**	**Press time (min)**	**Electrical Conductivity (E)**
1	175	5	5	5	
2	175	10	7	10	
3	175	15	10	15	
4	180	5	7	15	
5	180	10	10	5	
6	180	15	5	10	
7	185	5	10	10	
8	185	10	5	15	
9	185	15	7	5	

2.3 Experimental Procedure

G, CB and PP in powdered form and chopped CF with a length of 6 mm were used to produce G/CB/CF/PP composites. The composition of G/CB/CF/PP was mixed using a ball mill machine for 3 hours and high speed mixer for 10 minutes. The mixture was poured into a square mould with the dimension of 50 mm x 50 mm, then being pressed into the hot compression moulding machine with the designed compression moulding parameters. Then, the mould was left to cool naturally until it reached room temperature before the sample being removed from the mould. Electrical conductivity (E) test was performed using Jandel Multi Height Four Point Probe combined with RM 3 Test Unit.

3.0 RESULTS AND DISCUSSION

Electrical conductivity was measured for all trial composites and the results are shown in Table 5. Based on the USDoE requirements for BP, the electrical conductivity value must be greater than 100 S/cm [1, 19]. The results indicated that the electrical conductivity for all trial composites were higher than 100 Scm^{-1}. Thus, the required values for BP were achieved. The results consisted of electrical conductivity for various moulding parameters in nine trial experiments. Three measurements were collected for each trial of experiment with various moulding parameters. The average value for the electrical conductivity was calculated based on these three measurement values. The maximum result for electrical conductivity was 370.69 Scm^{-1} whereas the minimum result for electrical conductivity was 184.28 Scm^{-1}. Table

5 also shows the decrement of electrical conductivity for G/CB/CF/PP composites from 319.24 Scm^{-1} (trial no.1) to 184.28 Scm^{-1} at the no. 9 trial.

Table 5: Results of electrical conductivity for G/CB/CF/PP composites

No. of Trial	Response: Electrical Conductivity (Scm^{-1})			
	E1	E2	E3	Average
1	309.99	312.82	334.92	319.24
2	374.79	379.44	357.84	370.69
3	316.62	324.29	338.58	326.50
4	285.20	289.58	289.36	288.05
5	256.49	277.72	241.29	258.50
6	204.04	180.08	195.69	193.27
7	273.11	263.45	256.82	264.46
8	263.13	229.74	236.71	243.19
9	194.15	159.30	199.39	184.28
Average				272.02
Maximum				370.69
Minimum				184.28

CB with complex morphology is used as one of the fillers to help in filling the holes and gaps of the produced composite [16]. The electrical conductivity value was analysed through TM using S/N ratio to determine the optimized factor. In order to obtain the highest electrical conductivity of G/CB/CF/PP composites, TM with LTB response graph approach was chosen. S/N ratio was also used to represent the quality characteristics. By choosing LTB response graph with the optimum condition, the level of parameters that contribute to the highest electrical conductivity can be determined as shown in Figure 1.

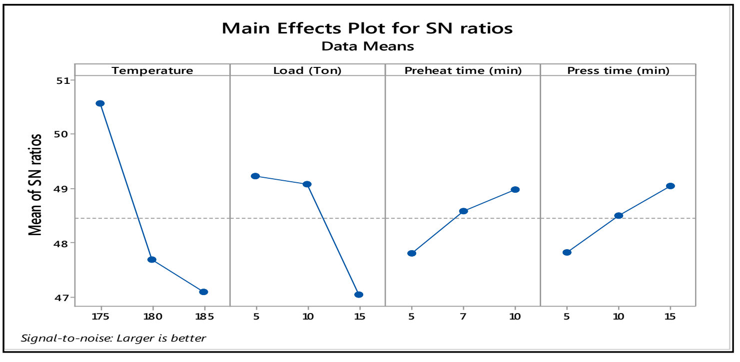

Figure 1: Response graph for S/N ratios

From the response graph (Figure 1), the highest electrical conductivity could be obtained from a combination of optimum conditions; A1 (temperature), B1 (pressure), C3 (preheating time) and D3 (pressing time). The optimized levels of parameters that contribute to the highest electrical conductivity of G/CB/CF/PP composites are shown in Table 6.

Table 6: Optimized factors/parameters for G/CB/CF/PP composites

Condition/Parameters	**Value**
Temperature (A1)	175 °C
Loads (B1)	5 tons
Preheating time (C3)	10 minutes
Pressing time (D3)	15 minutes

Moulding time is one of the most important factors in obtaining the highest electrical conductivity of G/CB/CF/PP composites because the composites require enough time to solidify [4]. This statement also supports the results in Table 6, where the optimum electrical conductivity of G/CB/CF/PP composites could be obtained from the long preheating and pressing time during the fabrication process. However, moulding temperature also gave a great impact in fabricating the highest electrical conductivity of G/CB/CF/PP BP composites. In order to minimise the viscosity of the composite for good dispersion and distribution of CPC, a suitable moulding temperature was required. In addition, to minimise the contact distance between fillers within a polymer matrix, a suitable moulding pressure was also required. Based on these statements, the factors that affected electrical conductivity of G/CB/CF/PP composites were dispersion, distribution and the contact distance between conductive fillers within a polymer matrix [4].

From the TM analysis using S/N ratio, the predicted value for electrical conductivity with the optimum condition was 382.40 Scm^{-1} and this value was higher than any other values. A confirmation experiment was conducted to validate the optimum parameters produced from the TM analysis. This experiment was also used to validate the optimum electrical conductivity of G/CB/CF/PP composites. The results of the experiment are shown in Table 7.

Table 7: Confirmation experiment of electrical conductivity with optimum condition

No. of Experiment	Electrical Conductivity (Scm^{-1})			
	E1	E2	E3	Average
1	393.78	397.45	389.23	393.49

The results depicted the new average electrical conductivity value with optimum parameters of 393.49 Scm^{-1} which was 3% higher than the predicted value from TM analysis. The results yielded that the optimum parameters obtained from the TM analysis can offer an improvement to the electrical conductivity of the composite. The fabrication procedure of the sample is also important to give better result and it is proven by the results produced from the confirmation experiment [5].

4.0 CONCLUSION

The optimum compression moulding parameters for G/CB/CF/PP composite with 25% of CB and 5% of CF have been analyzed and determined through TM using S/N ratio. The result of electrical conductivity for G/CB/CF/PP composites with optimum parameters is 393.49 Scm^{-1} which is more than the required value recommended by the USDoE for BP application. Based on the results, it can be concluded that the optimum parameters obtained by TM produce better electrical conductivity composite. Hence, these optimized results obtained from TM using S/N ratio with the quality characteristics of larger-the-better (LTB) is very important especially in the fabrication of potential G/CB/CF/PP composites as conductive polymer composites that are going to be used as bipolar plate (BP) for PEMFC.

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