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12-24	**Effect of processing parameters on electrical properties of polypropylene/graphite composite plates** Dedikarni Panuh; Radwan Dweiri; Jaafar Sahari **DOI**: 10.1504/IJMMP.2017.087658
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Effect of processing parameters on electrical properties of polypropylene/graphite composite plates

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Abstract: This paper aims at optimising the processing parameters for the production of electrically conductive polypropylene/graphite composite plates. The composites were prepared by melt compounding using internal mixer and the plates were produced by compression moulding. The effect of processing parameters on conductivity at different graphite contents were optimised using design of experiments based on a cross-mixed method. The results showed a significant dependence of the conductivity values on the processing parameters. The highest value of in-plane conductivity resulted from simulations was 86.83 S/cm and it was achieved at a mixing time of 57.95 min, a mixing speed of 105.8 rpm, a compression moulding time of 157.89 s, and a pressure of 48.24 bar. Slightly higher value of about 91 S/cm was experimentally obtained. The distribution of graphite particles and the porosity on the surfaces of plates were also examined.

Keywords: in-plane electrical conductivity; processing parameters; PP/SG composite plates; melt mixing; compression moulding; design of experiments.

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1 Introduction

The use of conductive polymer composites (CPCs) is very promising in engineering applications (Sabu et al., 2014). Their processing methods and properties of their products are an active research field. One important application where the CPCs have been intensively used is the bipolar plate in polymer electrolyte membrane (PEM) fuel cells (Allen et al., 2005). Bipolar plates have used as an alternative for metallic and traditional graphite plates in PEM fuel cells (Davies et al., 2000). The bipolar plate is used to separate each cell in the PEM stack, collect current, carry water out from each cell and humidify gas. The materials suggested for manufacturing bipolar plates must achieve high chemical and corrosive resistance, low cost and easy manufacturing processes, good electrical and thermal conductance, gas impermeabilities and good strength-to-weight performance. For bipolar plates, the in-plane electrical conductivity was restricted by the US Department of Energy to a minimum value of 100 S/cm (Xiao et al., 2005).

Generally, carbon-based CPCs have occupied a great interest in several engineering applications (Wei et al., 2007) and more specifically, in the application of bipolar plate in PEM fuel cells (Ehsan et al., 2013; Dweiri, 2015; Grundler et al., 2010; Ghosh and Verma, 2014; Xiqiang et al., 2006; Adjima and Jantrawan, 2008). Many issues have been investigated by researchers in the literature regarding to the use of carbon-based CPCs for bipolar plate. Using single and multi-conductive fillers such as graphite (G), carbon black (CB), carbon fibres (CF) were found as an effective way to improve processibility and performance of CBCs (Dweiri and Sahari, 2007; Suherman et al., 2014; Dweiri and Jaafar, 2007). Influence of graphite particle size and its shape on the performance of

carbon composite bipolar plates has also been investigated (Zhang et al., 2005; Dweiri, 2012). Intensive studies on the conventional methods such as compression and injection moulding showed that these methods are cost effective and produce net-shaped bipolar plates (Mighri et al., 2004; Müller et al., 2006; Devaraj, 2009; Cunningham and Baird, 2006; Dhakate et al., 2007).

The preparation of composites prior to compression and injection moulding and the effect of processing parameters on the mechanical and conductive properties of CPCs have attracted the attention of many researchers (Suherman et al., 2015; Carneiro et al., 2012; Raghavan et al., 2016; Dweiri and Sahari, 2010). Researchers have used melt or solution mixing techniques for preparation of CPCs. The solution mixing is environmentally harmful because of the use of solvents while the melt mixing is not and economically viable. Melt mixing was usually carried out by the use of internal mixers and many issues regarding to this technique had been highlighted by the researchers such as homogenisation of the composites and understanding the effect of processing parameters such as mixing time, rotating speed and mixing temperature on composite properties. Similar to melt mixing, compression moulding processing parameters involving compression moulding duration, pressure value and temperature are also play a main role in determining the final properties of the product. It is worth to mention that to improve the conduction properties of composites, the conductive fillers are to be dispersed uniformly throughout the polymer matrix. This can be achieved by sufficiently mixing the composites that means severe and long-time mixing parameters (Wei et al., 2007). Most studies focused on a short-time mixing within the range of 10–30 min and no studies are found for longer melt mixing duration.

This work aims at optimising the processing parameters of melt mixing and compression moulding for the production of electrically conductive PP/SG composite plates. Design of experiments based on a cross-mixed method was utilised for the purpose of optimisation. Mixing for long time of 50–120 min was carried out while there were no investigations on temperature effect. The high filling load of 60–90 wt.% SG was implemented in an attempt to meet the target of US Department of Energy of 100 S/cm in-plane electrical conductivity for bipolar plate in PEM fuel cells.

2 Experimental

2.1 Materials

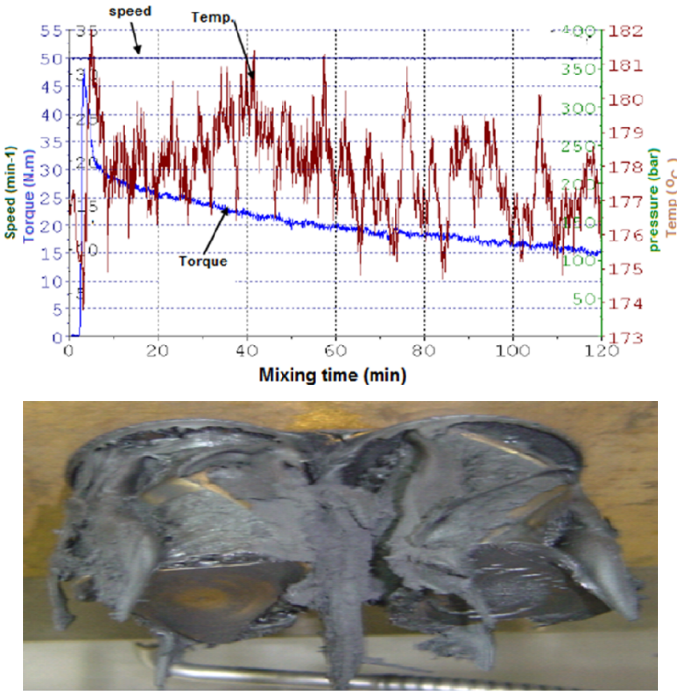
Synthetic flaky graphite particles (SG) with a density of 1.74 g/cm^3 and an electrical resistivity of 1200×10^{-6} Ω cm had been used in this study. It had a particle size distribution ranging between 10 and 200 μm and it was supplied by GME Carbon Sdn. Bhd., Malaysia. Polypropylene (PP) grade Titan (600) had a density of 910 kg/m^3, melt flow index of 10 g/10 min, a melting point of 160°C and it was supplied by Polypropylene Malaysia Sdn. Bhd.

2.2 Fabrication of the composite plates

Melt compounding of PP and SG are carried out at different compositions and processing parameters. The SG particles were mixed manually with PP at weight ratios (PP/SG) of (40/60), (25/75) and (10/90). The mixtures were then melt compounded using a Thermo

Haake mixer Roller type Rotors R600 with a 50 ml intensive batch mixer at a temperature of 175ºC. Different values of rotational speed and mixing time were applied. The melt compounded composites were crushed and placed into a mould which had been preheated in a hot press machine for 10 min without pressure. The composite plates were then hot pressed into disk samples of 100 mm diameter and 2 mm thickness at a temperature of 200ºC and different values of pressure and compression moulding time. Figure 1 shows a photo of the compounded PP/60 wt.%SG composite at the end of mixing process after opening the die. The change of torque and actual temperature during mixing time at a rotating speed of 50 rpm is also shown in Figure 1. At the beginning of mixing, the torque rapidly increases and falls down before it starts gradually decreasing. The actual temperature in mixing chamber raises up to 182ºC.

Figure 1 The photo of compounding PP/60 wt.%SG composite and the change of torque and actual temperature with mixing time during mixing process (see online version for colours)



2.3 Design of experiment

The composite material composition and processing parameters for the compression-moulded plates were optimised by design of experiment (DOE) based on cross-mixed method using computer software namely 'Design Expert® 6' (Stat-Ease). Table 1 indicates the input data for performing the simulation and it represents the factors that influencing the electrical conductivity of the composites, their symbols and the values range being selected. The DoEs based on cross-mixed method is involving components with several variables and applying an equation in the form of centroid simplex model

(equation (1)) combined with 2^n factorial model (equation (2) to produce the optimum response (equation (3) (Montgomery, 2001; Cornell, 2002).

$$y(x) = \sum_{i=1}^{q} \beta_i x_i + \sum_{i=1}^{q} \sum_{i<j}^{q} \beta_{ij} x_i x_j + \cdots + \beta_{12\ldots q} x_i x_j \ldots x_q + \varepsilon, \tag{1}$$

where x_i is the component mixture with $i = 1, 2, \ldots, q$; β is the mixing coefficient and ε is the standard error.

$$y(z) = \alpha_0 + \sum_{l=1}^{n} \alpha_l z_l + \sum_{l=1}^{n} \sum_{l<m}^{n} \alpha_{lm} z_l z_m + \cdots + \alpha_{12\ldots n} z_l z_m \ldots z_n + \varepsilon, \tag{2}$$

where z_l is the variable process with $l = 1, 2, \ldots, n$; α is the factorial denominator coefficient; and ε is the standard error.

$$\begin{aligned} y(x,z) = & \sum_{i=l}^{q} \left[\gamma_i^0 + \sum_{l=1}^{n} \gamma_i^l z_l + \cdots + \gamma_i^{12\ldots n} z_1 z_2 \ldots z_n \right] x_i \\ & + \sum_{i=l}^{q} \sum_{i<j}^{q} \left[\gamma_{ij}^0 + \sum_{l=1}^{n} \gamma_{ij}^l z_l + \cdots + \gamma_{ij}^{12\ldots n} z_1 z_2 \ldots z_n \right] x_i x_j \\ & + \cdots + \left[\gamma_{12\ldots q}^0 + \sum_{l=1}^{n} \gamma_{12\ldots q}^1 z_1 + \cdots + \gamma_{12\ldots q}^{12\ldots n} z_1 z_2 \ldots z_n \right] x_1 x_2 \ldots x_q + \varepsilon, \end{aligned} \tag{3}$$

where γ is the combination denominator coefficient. q and n were adjusted to $q = 2$ and $n = 2$ in this study.

Table 1 Factors affecting the in-plane electrical conductivity of the composites and their selected values which was used for DOE

		Symbol	*Unit*	*Selected values*
Composite components	Graphite	SG	wt.%	60, 75, 90
	Polypropylene	PP	wt.%	40, 25, 10
Processing variables	Mixing time	MT	min	50, 120
	Mixing speed	MS	rpm	50,110
	Compression moulding time	PT	sec	155, 220
	Pressure value	PV	bar	30, 50
Response	Conductivity	CON	S/cm	–

2.4 Characterisation of composite plates

In-plane electrical conductivity of the plates was measured by means of a Jandel Multi-Height Four-Point Probe combined with RM3 Test Unit which had a constant current source and a digital voltmeter designed especially for the four-point probe measurement. This technique measured sheet resistance in the range from 1 mΩ/sq up to 5×10^8 Ω/sq and volume resistivity ranges from 10^{-3} to 10^6 Ω cm. The system accuracy was within 0.3%. No electrodes used for DC measurements. The electrical resistivity, ρ, of the samples can be calculated according to the formula:

$$\rho = 2\pi s\left(\frac{V}{I}\right)F, \tag{4}$$

where s = 1 mm is the probe spacing, I is the input current through the outer two tips, V is the voltage drop across the inner two probes and F is the correction factor based on the ratio of sample thickness to probe spacing. This test met specifications of ASTM F 84-99 Standard.

The morphology of the surfaces of the plates and the distribution of SG particles on surfaces was examined using Leo 1450 Scanning Electron Microscopy Energy Depressive X-Ray Spectroscopy (SEM-EDX) instrument (High-Tech Company). The porosity measurements were performed using a Thermo Finnigan Pascal 140 series mercury porosimeter. The surfaces of the plates were polished with a fine emery paper to remove the slip layer of the binder prior tests.

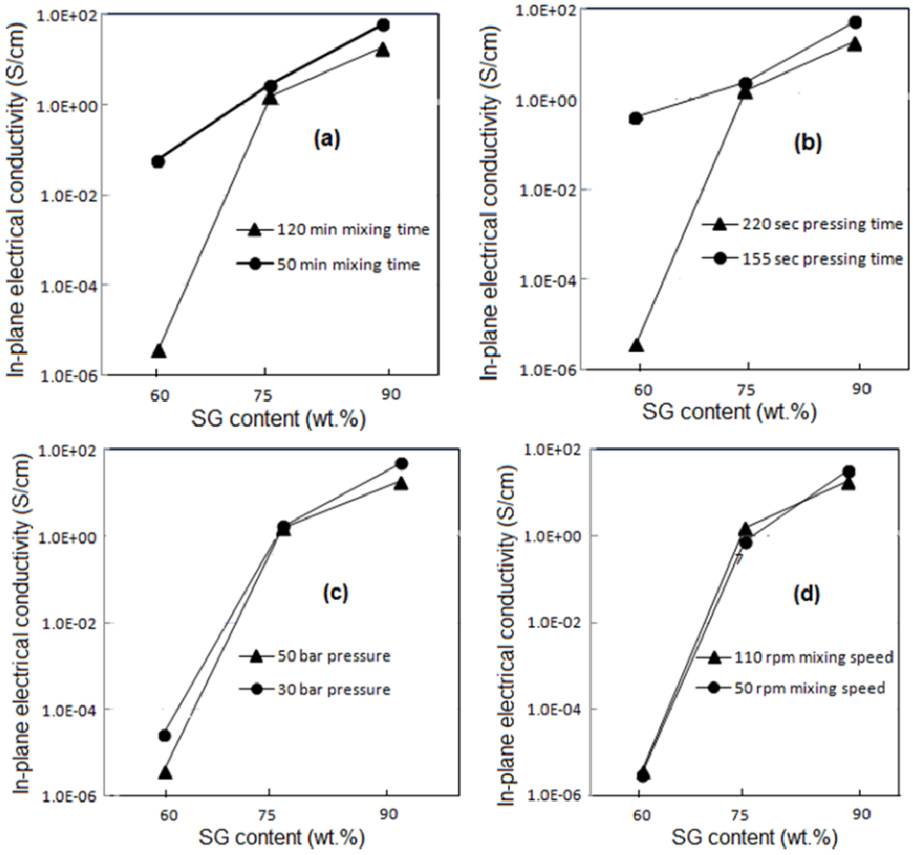
3 Results and discussion

3.1 Results of design of experiments

Initial DOE analysis using the input data shown in Table 1 was carried out and 41 different samples at different processing parameters and their resulting in-plane conductivities are shown in Table 2. The data shown in Figure 2 were explored from Table 2 to further clarify the effect of each processing parameter on the electrical conductivity of the composites. The effect of mixing time and rotating speed, compression moulding time and pressure were examined. The reference samples of 60, 75 and 90 wt.% SG were produced at MT = 120 min, MS = 110 rpm, PT = 220 s and PV = 50 bar. The results are represented in Figure 2 as the relationship of in-plane electrical conductivity vs. SG content. In general, the values of conductivity increase with the increase in filler loading regardless of the processing parameters. Figure 2(a) shows that the increase in mixing time results in a decrease in conductivity values for all types of composites. Researchers (Kalyon and Birinci, 2002) reported that the increase of mixing time increases the specific energy input generated during the mixing process which causes better coating of the binder to the SG particles, thus the formation of conductive network is hindered and electrical conductivity decreases. This effect is less pronounced as SG content increases in the composites due to the decrease of the binder amount coated the graphite particles. A reduction in the values of conductivity of the composite plates was also noticed by increasing the compression moulding time from 155 to 220 s (Figure 2(b)) and by increasing the compression pressure value from 30 to 50 bar (Figure 2(c)). It is known from the previous studies that the electrical conductivity increases as a result of the increase in compactness of filler particles in the polymer matrix with increase in moulding pressure (Das, 2002). In the pressure range selected here, opposite trend was observed and the increase of compression moulding time and pressure may elevate specific energy of a mixture and pushing the binder to the sample surface, and hence reducing conductivity. Figure 2(d) shows that the increase in rotating speed has less effective impact on electrical conductivity and literature reported that, with increasing rotating speed, the SG particles and agglomerates are subjected to more shearing action leading to appreciable breakdown of the conductive network, and hence the conductivity of the composite decreases. Furthermore, increase of rotating speed

causes a temperature rise of the matrix, so that its viscosity decreases and as a result the shearing force decreases. Finally, it was noticed from DOE that composites containing 75 wt.% SG were less affected with processing parameters compared to those having 60 and 90 wt.%.

Figure 2 Effect of: (a) mixing time; (b) compression moulding time; (c) pressure value and (d) mixing speed on the conductivity of composite plates at different SG contents obtained from DOE results



The previous simulations showed that a maximum in-plane electrical conductivity value of 72.22 S/cm was reported for PP/90 wt.%SG composites and this value was obtained at MT = 120 min, MS = 110 rpm, PT = 155 s and PV = 30 bar. For further optimising the electrical conductivity, a second running of the DOE was carried out for PP/90 wt.%SG and the mixing time, MT, was left to change from 50 min to 120 min, MS from 50 min to 120 rpm, PT ranging between 155 s and 220 s and PV from 30 bar to 50 bar. The results of simulations are shown in Table 3. It is noticed from Table 3 that the highest value of in-plane conductivity was 86.83 S/cm and it was achieved at MT = 57.95 min, MS = 105.8 rpm, PT = 157.89 s and PV = 48.24 bar. Therefore, at a high rotating speed the two conflict effects, which mentioned earlier, may counterbalance each other

(Das, 2002). By noting the change of the processing parameters all together, the judgement on increasing and decreasing of conductivity is quite different, e.g., the low mixing time, high rotating speed, low compression time and high pressure value results in high electrical conductivity value. Finally, the a composite plate of PP/90 wt.%SG was experimentally produced at the optimised parameters and its in-plane electrical conductivity value was found to be 91.27 S/cm. This value was slightly higher than that obtained using DOE but it could be concluded that there is a good agreement between the predicted and experimental value. Comparing the results obtained in this study with the previous studies (Lawrance, 1980) reported a value of in-plane conductivity about 119 S/cm for poly(vinylidene fluoride)/80 wt.% graphite. Wilson and Busick (2001) reported a value of 61 S/cm for vinyl ester/68 wt.% graphite and Dweiri and Sahari (2007) reported a value of less than 10 S/cm for PP/80 wt.% SG composites.

Table 2 Initial DOE running results and the input data are shown in Table 1

	Components		*Processing parameters*				*Response*
	SG	*PP*	*MT*	*MS*	*PT*	*PV*	*CON*
Sample	(wt.%)	(wt.%)	(Min)	(rpm)	(s)	(bar)	(S/cm)
1	90	10	120	50	155	50	27.47
2	75	25	50	50	220	30	1.5
3	75	25	50	110	220	30	1.07
4	75	25	50	110	155	30	1.481
5	90	10	50	50	220	50	41.1
6	60	40	120	50	220	50	3.07×10^{-6}
7	75	25	120	110	220	50	1.55
8	75	25	120	110	155	30	0.94
9	90	10	50	110	220	30	19.67
10	90	10	120	50	155	30	32.4
11	90	10	120	110	220	50	18.05
12	90	10	120	110	220	30	47.69
13	90	10	50	110	220	30	35.1
14	90	10	50	110	155	30	22.07
15	75	25	120	50	220	50	0.753
16	75	25	50	50	220	50	1.52
17	90	10	50	50	220	50	41.1
18	60	40	50	50	155	50	5.80×10^{-6}
19	60	40	50	110	220	50	0.06
20	90	10	50	50	220	30	48.14
21	90	10	120	50	220	50	31.99
22	90	10	50	110	220	50	61.65
23	75	25	50	50	155	50	2.53

Table 2 Initial DOE running results and the input data are shown in Table 1 (continued)

	Components		*Processing parameters*				*Response*
	SG	*PP*	*MT*	*MS*	*PT*	*PV*	*CON*
Sample	(wt.%)	(wt.%)	(Min)	(rpm)	(s)	(bar)	(S/cm)
24	60	40	50	50	220	30	0.143
25	60	40	120	110	155	50	0.39
26	90	10	120	110	155	30	72.22
27	75	25	120	50	220	30	0.86
28	60	40	120	110	220	30	2.61×10^{-5}
29	75	25	120	50	155	30	0.481
30	75	25	120	110	155	50	2.34
31	90	10	50	50	155	50	44.74
32	60	40	120	110	220	50	3.60×10^{-6}
33	75	25	50	110	155	50	2.61
34	60	40	120	50	155	30	7.02×10^{-6}
35	75	25	120	110	220	30	1.88
36	75	25	120	110	220	30	1.68
37	75	25	50	50	155	30	1.48
38	90	10	50	50	155	30	20.38
39	75	25	50	110	220	50	2.77
40	75	25	120	50	155	50	0.75
41	90	10	120	110	155	50	50.55

3.2 Porosity measurements and morphological observations

Porosity is one of the factors that affecting the electrical conductivity of the composites. Figure 3(a)–(c) shows the results of porosity measurements of three different samples. The pore size distribution of all samples was approximately ranging from 5 to 100 μm. The relative volume porosity for (40/60), (25/75) and (10/90)wt.% PP/SG was in the range of 0.15–0.75%, 0.05–0.65% and 0.0–0.65% and their total porosities were 1.037%, 1.282% and 1.77%, respectively. Figure 3(d) shows the average pore size which decreases by increasing SG content and values of 10.6, 6.01 and 5.48 μm were estimated for (40/60), (25/75) and (10/90)wt.% PP/SG composites, respectively. The decrease of the degree of porosity and the pore size causes lower distance between particles, and hence enhancing the electrical conductivity. Gautam and Kar (2015) stated that, at high nanocarbon black content in phenolic matrix, the carbon particles got agglomerated, which developed closed porosity in the composites and this causes an increase in the porosity.

The SEM micrographs of the surfaces of PP/SG composite plates consist of 60, 75 and 90 wt.% SG and their EDX mapping are illustrated in Figure 4 and the SG particle distribution is shown on the surface. In general, the SG particles are not uniformly

distributed within the polymer matrix and agglomeration of particles is found on the surfaces shown in Figure 4(a)–(c). The tendency of the SG agglomerates to form conductive paths on the polymer matrix is increased as SG content increases and these agglomerates are better distributed in PP having 90 wt.% SG. Moreover, less particle-to-particle contacts could be attributed to lower the electrical conductivity of the composite plates.

Table 3 In-plane conductivity readings and the predicted processing parameters for PP/90wt.%SG composite obtained from DOE (second running)

	Components		*Processing variables*				*Response*	
	SG (wt.%)	*PP* (wt.%)	*MT* (min)	*MS* (rpm)	*PT* (s)	*PV* (bar)	*CON* (S/cm)	
1	90	10	60.33	99.57	159.32	49.2	81.78	
2	90	10	57.95	105.8	157.89	48.24	86.83	Maximum
3	90	10	50	84.81	182.29	50	66.09	
4	90	10	120	110	193.77	31.75	52.93	
5	90	10	86.99	105.42	155	37.12	49.16	
6	90	10	82.77	66.3	155	49.99	47.94	
7	90	10	87.58	59.17	213.3	30	37.04	
8	90	10	82.32	75.64	213.94	30.01	36.74	

Figure 3 The pore size distribution of PP/SG composite plates at: (a) 60wt.% SG; (b) 75wt.% SG; (c) 90wt.% SG and (d) the average pore size vs. SG content (see online version for colours)

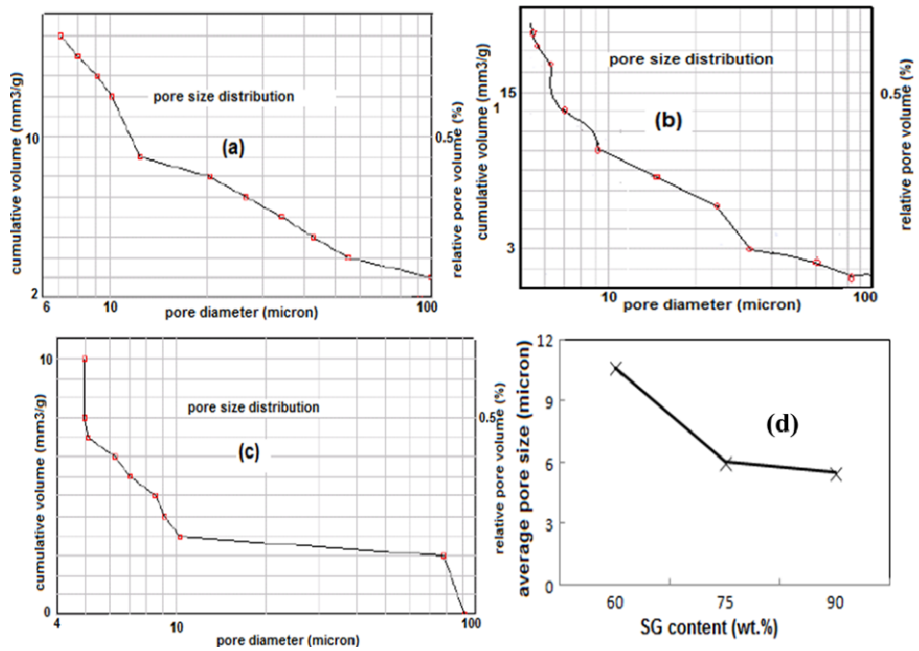
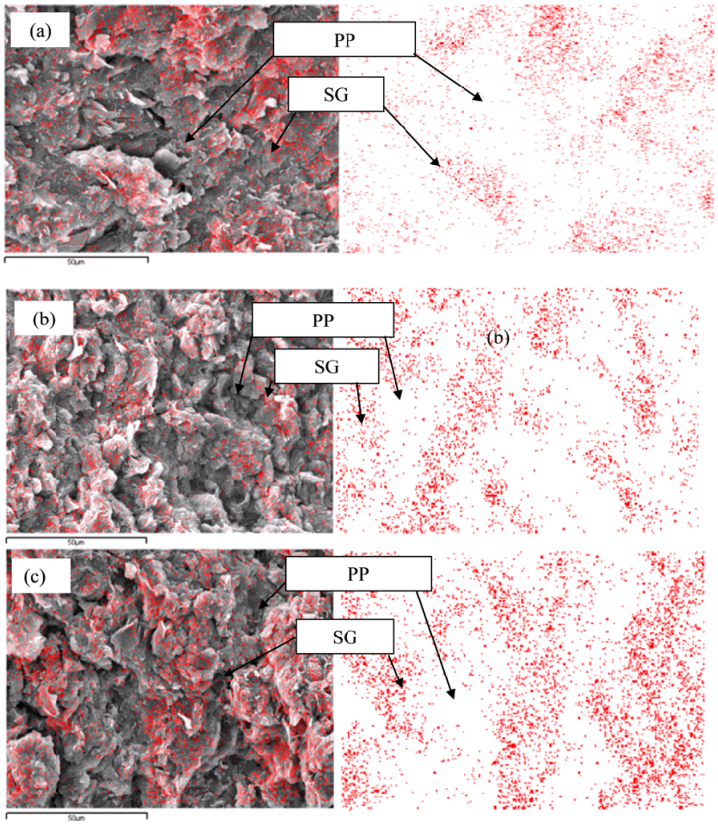


Figure 4 SEM micrographs of the surfaces of the composite plates and SG particles distribution at: (a) 60wt.% SG; (b) 75wt.% SG and (c) 90wt.% SG (see online version for colours)



4 Conclusions

A better understanding of the effect of processing parameters on the in-plane electrical conductivity of PP/SG composites was explored by using DOE and experimental investigations. The DOE results showed that the increase of mixing time and speed, compression time and pressure resulted in a decrease of the in-plane conductivity of the composites. Further DOE simulations for composites containing 90 wt.% SG showed that a combination of low mixing time and high mixing speed, and a low compression time and a high pressure produced the optimum value of conductivity, 86.83 S/cm. This value was in a good agreement with the experimental conductivity value of PP/90 wt.%SG composite which was found about 91 S/cm. Porosity of composite plates was found to increase with increasing SG content while the average pore size decreased. Morphology of the composite plates showed a tendency of the SG to agglomerate and at 90 wt.%, the agglomerates tended to form a better conductive paths in PP matrix. These values of conductivities at high filler content are still not competitive for applications of bipolar plate in fuel PEM fuel cells due to their difficulty of manufacturing and the expected degradation in mechanical properties.

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