Short Communication:

Statistical Study on the Interaction Factors of Polypropylene-Graft-Maleic Anhydride (PP-g-MA) with Graphene Nanoplatelet (GNP) at Various Poly(Lactic Acid)/Polypropylene (PLA/PP) Blends Ratio

Farah Hafidzah[1], Mohd Bijarimi[1,*], Waleed Alhadadi[1], Suriyati Salleh[1], Mohammad Norazmi[2], and Erna Normaya[2]

[1]*Faculty of Chemical and Natural Resources Engineering, Universiti Malaysia Pahang, Lebuhraya Tun Razak, 26300 Gambang, Pahang, Malaysia*

[2]*Experimental and Theoretical Research Laboratory, Department of Chemistry, Kulliyyah of Science, International Islamic University Malaysia, Kuantan, Malaysia*

* ***Corresponding author:***

tel: +609-5492918
email: bijarimi@ump.edu.my

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Abstract: *This paper reports the effects of polypropylene-graft-maleic anhydride (PP-g-MA) and graphene nanoplatelet (GNP on tensile stress of various PLA/PP weight ratio. The PLA/PP blends prepared with the ratio 70/30, 80/20, and 90/10 with the addition of PP-g-MA (1 to 5 phr) and GNP (1 to 3 phr) by using an injection molding machine. The tensile stress (MPa) was analyzed based on 11 runs of full factorial design. The results showed that the tensile stress of PLA/PP blends gradually increased after the addition of PP-g-MA and GNP. There is a relationship between PP-g-MA and GNP which causes a positive impact on the mechanical properties of PLA/PP blends. The optimum tensile stress of 50.06 MPa achieved at the ratio of 90/10 blends with 5 phr of PP-g-MA and 3 phr of GNP.*

Keywords: *poly(lactic acid)(PLA); polypropylene (PP); graphene nanoplatelets (GNP); tensile stress*

INTRODUCTION

Poly(lactic acid) (PLA) has attracted extensive studies among researchers as compared to the other polymers from petroleum [1-4]. PLA is derived from corn, sugar cane, and potato, it possesses complimenting properties in mechanical properties (high tensile strength and stiffness), biodegradability, and biocompatibility [5-6]. However, despite these outstanding properties, PLA suffered due to its ~~a~~ brittleness behavior, poor thermal stability, and weak melt strength [7-8]. On the other hand, polypropylene (PP) is a relatively inexpensive commodity, low density, sturdy, excellent in water barrier, processable, and recyclable [9].

Various approaches have been suggested in several studies for toughening PLA, including the melt blending method. The blending of PLA with PP is a well-researched due to its complementary properties, where both polymers have a similar range of temperature regions [10]. Choudary et al. [11] revealed that the mechanical properties of PLA have improved significantly with PP in the matrix blends. In terms of thermal stability, Chen et al. reported that PLA/PP blends improved as measured by thermogravimetric analysis (TGA) [12]. It is worth noting that PP is thermally stable as compared to PLA; for example, PP started to degrade at 418.8 °C, whereas PLA was 334.3 °C. The blending of 10 wt.% of PP with PLA has increased the $T_{5\%}$, $T_{50\%}$, and T_{max}, which indicated the PLA's thermal properties stabilized with the presence of PP.

It is known that PLA and PP blends are an example of incompatible polymer blends [13-14], due to the significant difference in polarities [15]. In addition, PP has an inert backbone with lack of reactivity and causes weak interfacial adhesion when blended with another polymer [16]. This blend was observed to have multiple phases through surface morphology of PLA/PP blends, which leads to poor mechanical performance. A compatibilizer is usually incorporated into an incompatible blending system to improve the properties of polymer blends. Ploypetchara et al. found that 3% of polypropylene-graft-maleic anhydride (PP-g-MA) has increased the thermal stability of PLA/PP [17]. The presence of PP-g-MA at the boundary of the PLA and PP helps to hold domains and consequently results in higher mechanical strength [18]. The improvement of the blend occurred when there was an interaction of the MA group from the compatibilizer; reacting with the hydroxyl group of PLA [19].

Apart from that, incorporation of nanofiller into the blends also serves a significant impact on the final properties of nanocomposites, such as mechanical, thermal, morphology, and interfacial properties [20-22]. In recent years, many papers reported on PLA/PP blends reinforced with clay [23-24], multi-walled carbon nanotubes [25], and sepiolite [26]. However, few studies investigated the effect of graphene nanoplatelet (GNP) reinforced on PLA/PP blends. GNP is a 2D nanofiller produced from graphite, low cost, possesses high stiffness, and good thermal stability [27]. As a nanofiller, GNPs can be embedded into a polymer matrix as an agent to enhance mechanical and other properties [28]. It has a different geometric size and atomic with a high aspect ratio [29] and classified as a polar nanofiller. Thus, only a low amount of GNP required to enhance polymer properties. Since PP is a non-polar, it is incompatible with the polymer matrix [30] due to the presence of Van der Waals forces, which lead to re-agglomeration [31] and affect the mechanical properties as well. In this study, PP-g-MA helps in improving the dispersion of GNP in PLA/PP blends. Typically, a well-dispersed system able to produce acceptable composite properties. The presence of PP-g-MA may enhance the compatibility between PLA, PP, and also GNP.

To fulfill the industrial application requirement, the compatibility of PLA/PP is required to be improved. Compatibilization is a well-known approach accomplished either through extrusion, injection, or both with PP-g-MA, which helps improve the interfacial adhesion between polymers [5]. PP-g-MA has the potential to be compatibilizer candidates for PLA/PP blends since it inherits a similar backbone structure as PP and present of the polar group, maleic anhydride (MA) [16]. On the other hand, GNP has been suggested for improving mechanical and thermal properties [4,28,32] by embedding at the polymer matrix.

Design of the experiment (DOE) such as full factorial design (FFD) has become increasingly popular in research and development. Through statistical data and analysis help to analyze the causal relationships and the actual effects of variables [33]. This approach employed in PLA/PP blends study involved the addition of compatibilizer, PP-g-MA, and nanofiller, GNP. This study is valuable since the relationship between PP-g-MA and GNP content on PLA/PP blend has not yet been investigated extensively through the FFD approach.

This paper aims to report the preparation and characterization of PLA/PP/GNP nanocomposites. The relationship between PP-g-MA and GNP loadings with various PLA/PP blends weight ratio also presented based on the designated full factorial design (FFD) experimental.

■ EXPERIMENTAL SECTION

Materials

The polylactic acid (PLA), polypropylene (PP), polypropylene grafted maleic anhydride (PP-g-MA), and graphene nanoplatelet (GNP) which obtained from commercial sources. The PLA (grade 3251D) was supplied from NaturalWorks Co., USA. The PP (Titanpro® polypropylene Copolymer 1D) was supplied from Lotte Chemical Titan. The PP-g-MA (DuPont™ Fusabond® P613) was provided by DuPont as a compatibilizer. All these polymers have a similar melting point with at the range of 160 to 170 °C. As for nanofiller, xGnP® Graphene Nanoplatelets Grade M5 was purchased from XG sciences with a carbon purity of 99.5%, the density of 2.2 $g.cm^{-3}$, an average thickness of 6 to 8nm, surface area 120 to 150 $m^2.g^{-1}$ and tensile modulus of 1000 GPa [34].

Procedure

Preparation of PLA/PP/GNP nanocomposites

PLA, PP, and PP-g-MA were dried in an oven over 12 h at 60 °C before used to reduce moisture. The materials, including PP-g-MA and GNP, were premixed and extruded in a twin-screw extruder at a temperature setting of 145 to 160 °C. The screw rotation speed fixed at 100 rpm/min. The blend composition was prepared based on Table 1. The compounded polymer was cut into a pellet and followed by injection molding based on ASTM D638 at temperature profile 140 to 150 °C. The mold temperature fixed at 30 °C.

Mechanical characterization

The yield strength (tensile stress, MPa) of samples was tested according to the ASTM D638 Type 1 standard using Universal Tensile Machine (UTM) under ambient condition. The loading speed of the machine set at 10 mm/min with a maximum 5 kN load cell. There are five specimens of each blending formulation tested, and the averaged of tensile stress obtained were recorded.

Experimental design

The factorial experimental design is an approach of experimental technique in which all factors can vary simultaneously over a set of the trial run [35]. The 2^k factorial design was employed to study the effect of factors on the mechanical properties of nanocomposites.

An optimized composition of PP-g-MA and GNP to reinforce the PLA/PP blend is very critical for nanocomposites final properties. In the present study, three manipulated factors such as PLA/PP (wt/wt.%) blending ratio, PP-g-MA, and GNP loadings were investigated in the range of 70–90 wt/wt.%, 1–5 phr and 1–3 phr. The FFD with a two-level of 23 was applied to investigate the main effects and interaction between factors concerning the tensile stress of the PLA/PP/GNP nanocomposites by conducting 11 experimental runs. Table 1 shows the design factors and factor level (where -1 = low level, 0 = center point and +1 = high level) employed in this study shown in Table 1. The statistical variance analysis (ANOVA) was employed to analyze the response data by complying 95% confidence level.

RESULTS AND DISCUSSION

Experimental Data for PLA/PP/GNP Nanocomposites Tensile stress

The results obtained from the mechanical analysis of the experimental design are the averages of five prepared specimens. A model for the mechanical properties of PLA/PP/GNP nanocomposites was designed and performed. It was found that the PLA/PP (wt/wt.%)

Table 1. Factors and levels of PLA/PP/GNP nanocomposites experimental design

Design factor	Unit	Factor levels		
		-1	0	+1
A: PLA/PP	wt/wt.%	70	80	90
B: PP-g-MA	phr[a]	1	3	5
C: GNP	phr[a]	1	2	3

[a] part per hundred of PLA/PP binary blend

Table 2. Experimental for data PLA/PP blend tensile stress (MPa) result

Run	PLA/PP (wt/wt.%)	PP-g-MA (phr[a])	GNP (phr[a])	Tensile stress (MPa)
1	-1	+1	-1	31.5353
2	+1	+1	-1	41.1994
3	0	0	0	36.1918
4	-1	-1	-1	25.3287
5	0	0	0	35.5722
6	+1	-1	-1	37.2995
7	-1	-1	+1	29.4699
8	0	0	0	36.9022
9	+1	+1	+1	50.0640
10	+1	-1	+1	38.2277
11	-1	+1	+1	37.8925

[a] part per hundred of PLA/PP binary blend

ratio, PP-g-MA (phr), and GNP (phr) design inputs, influenced the responses for tensile stress (MPa). Table 2 shows the overall results obtained based on each designed run. The results were taken based on an average of five specimens.

Variance Analysis

Pareto chart of t-value (Fig. 1) as illustrated and factors of Bonferonni limit (absolute significance) (red line) and t-value limit (significant line) (black line) on the tensile stress of the PLA/PP/GNP nanocomposites. In this chart, symbols A, B, and C denoted as PLA/PP (wt/wt.%) ratio, PP-g-MA (phr), and GNP (phr) loaded.

In the overall t-value obtained, bar chart which belongs to A, B, C, and interaction of BC, contributed significantly to the results obtained. Since the interaction of AC and AB was below the t-value limit, it was noted that both interactions not adequate to be part of the generated statistical model.

The factors affecting the PLA/PP/GNP nanocomposites determined by ANOVA, summarized in Table 3. The values of $P < 0.05$ indicate the model of the data was significant, while the statistical relationship between chosen factors with its response has a 95% confidence level when the P value less than 0.05 [36]. Based on results obtained, the P-value of the model was less than 0.05 ($P < 0.0001$), which indicates the model of the data was significant.

The individual selected factors with denoted as A, B, and C also had statistically significant for the tensile stress (MPa) of PLA/PP/GNP nanocomposites. On the other hand, the interaction between factors B and C, there was statistical evidence synergistic effect between both of them, since P-value obtained 0.0086.

The effect of selected factors and models was further confirmed based on R^2 value. In the data set, R^2 represents the gaps between the data obtained with the fitted value. The R^2 obtained was close to 100% (R^2 = 0.9873), which indicated that the experimental data were closed and almost fitted to the regression line. This fitted data has also supported by the 'Lack of Fit F-value, which not significant relative to the pure error. There is a 30.93% chance that a "Lack of Fit F-value' occurs due to noise.

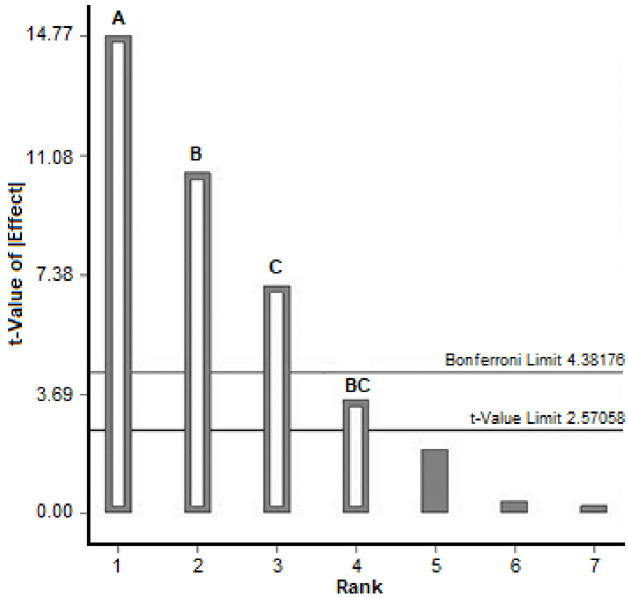

Fig 1. T-value analysis of each design factor

Table 3. Statistical analysis on design factor of PLA/PP/GNP nanocomposites

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F
Model	406.07	4	101.52	116.15	< 0.0001
A-PLA/PP	226.46	1	226.46	259.11	< 0.0001
B-PP-g-MA	115.26	1	115.26	131.87	< 0.0001
C-GNP	51.47	1	51.47	58.89	0.0003
BC	12.88	1	12.88	14.74	0.0086
Residual	5.24	6	0.87		
Lack of Fit	4.36	4	1.09	2.46	0.3093
Pure Error	0.89	2	0.44		
Cor Total	411.32	10			
R^2	0.9873				
Adj R^2	0.9788				

Main and Interaction Effects

Fig. 2 illustrated the main effects plot for the tensile stress of nanocomposites produced. These three plotted graphs explained that outcome changes in nanocomposites' tensile stress when varying PLA/PP ratio, PP-g-MA loaded, and GNP loaded into the blends. The gradient of the plot graph obtained indicated the relative tensile stress for the selected factor effects. In this study, the strength of PLA/PP blends depended on the amount of PLA content. The higher the PLA contents, the stronger the strength of blends produced. In Fig. 2(a), the maximum tensile stress (MPa) achieved at a 90/10 percentage of weight ratio.

This concentration of biodegradable PLA loading is desired in the nanocomposites to increase the use of biobased content. However, because of the immiscibility of these polymers, the strength of the PLA/PP blends obtained not promising compared to virgin PLA. Through the addition of compatibilizer, the immiscibility of polymer was able to be resolved. With the addition of PP-g-MA as compatibilizer and GNP as nanofiller, increasing trends of tensile stress were observed in the nanocomposites. The tensile stress increased starting at 1 to 5 phr of PP-g-MA loading and 1 to 3 phr of GNP. Generally, the role of PP-g-MA as compatibilizer is to improve interfacial adhesion between both polymers [37]. The presence of maleic anhydride (MA) as an initiator promotes outstanding chemical reaction with PLA [38]. While the ability of GNP which has high surface area supported in strengthening the blends. It is

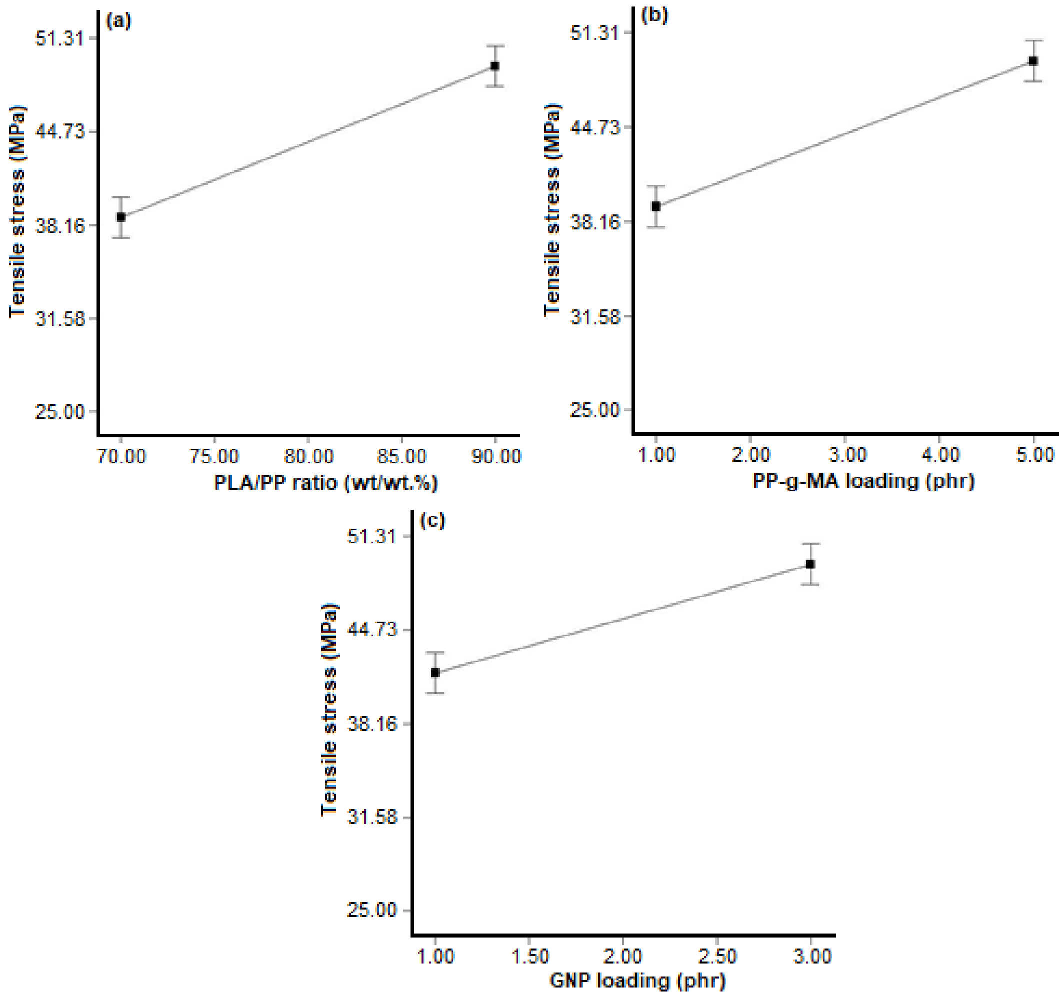

Fig 2. Main effect plot (a) PLA/PP ratio, (b) PP-g-MA loading and (c) GNP loading on the tensile stress of nanocomposites

Table 4. Statistical models in terms of code and the actual factor

Statistical model		
Coded Factor	Tensile stress (MPa) = 36.33 + 5.32 * A + 3.80 * B + 2.54 * C + 1.27 * B * C	Eq. (1)
Actual Factor	Tensile stress (MPa) = 13.14620 + 0.53205 * PLA / PP + 0.62878 * PPgMA + 0.63282 * GNP + 0.63453 * PPgMA * GNP	Eq. (2)

suggested that the lower amount of nanofiller loaded has attributed to better dispersion in the polymer matrix. A large amount of nanofiller loadings decreased the strength of nanocomposites. The results obtained in this study were similar to the previous studies i.e. by lowering the filler content, it affects the toughness of the blend [30]. Sima et al. blended a 3, 6, and 9% of GNP with PLA and PBT [28]. The SEM micrograph of PLA/GNP and PBT/GNP observed that 3% of GNP well dispersed in both polymer matrix.

Further addition of GNP, a more significant cluster of GNP was detected and leads to a decrease in the strength due to the weak binding of GNP with the polymer matrix. Inuwa et al. also found similar behavior in the blends of PET/PP with 0 to 7% of GNP [39]. The optimum increment observed through thermal and morphology observation at 3 phr GNP loading, which could be attributed to the homogenous GNP's dispersion in the matrix blends compared to other GNP loaded.

Unlike Fig. 2, this plot describes the interaction that occurs upon the effect of one factor depends on the level of the other, and this interaction unable to be detected by one factor at-a-time OFAT. Based on the interaction plot for tensile stress of PLA/PP blends illustrated in Fig. 3, it was clear the plotting lines of B (PP-g-MA loaded, phr) with C (GNP loaded, phr) were un-parallel which imply B and C was dependent to each other. The effect GNP loadings on the tensile stress of nanocomposites blend depend on the PP-g-MA content represented by the two lines on the graph. It is suggested that the nanocomposites tensile strength increases as the PP-g-MA loading was increased. This statistical significance of the interaction term indicated that there is a relationship between the role of PP-g-MA and GNP loading. At a high ratio of PLA in the blends, PP-g-MA and GNP addition causes a gradual increase in tensile stress up to 50.06 MPa. The compatibilized PP/GNP with PP-g-MA shows better in

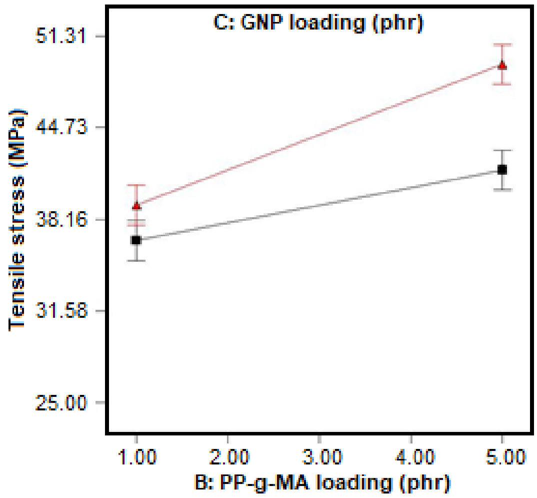

Fig 3. Interaction plot of B and C in terms of tensile stress

tensile strength than PP/GNP due to lack of compatibility between GNP and PP [40].

Developed Statistical Model

The effect of PP-g-MA and GNP with various PLA/PP blends ratio on the nanocomposites' tensile stress expressed via the developed statistical model. The regression Eq. (1) and (2) represents the appropriate description after eliminating factors with a P-value of more than 0.05. Table 4 shows the final empirical models in terms of code and the actual factor.

■ CONCLUSION

The FFD of experiments with 11 experimental formulations of PLA/PP/GNP nanocomposites was prepared and characterized in mechanical properties (tensile stress). Through this statistical analysis, it allows the generated model to describe the tensile stress of nanocomposites obtained. This model can also be used to study the main effect and interaction of PP-g-MA and GNP loaded with various PLA/PP weight ratios. Besides the amount of PLA composition in the blends, the presence of PP-g-MA as compatibilizer and GNP as nanofiller impacts on the tensile stress of the nanocomposites. The optimum tensile stress of 50.06 MPa achieved at the ratio of 90/10 blends with 5 phr of PP-g-MA and 3 phr of GNP.

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