

TENSILE PROPERTIES OF POLYPROPYLENE/LINEAR LOW-DENSITY POLYETHYLENE/NANO-TITANIUM DIOXIDE NANOCOMPOSITES USING A TWO-LEVEL FACTORIAL EXPERIMENT

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Abstract: In this paper, a 2³ factorial design analysis was used to study the parameters affecting the mechanical characteristics of polypropylene/linear low-density polyethylene/nano-titanium dioxide (PP/LLDPE/TiO₂) nanocomposites, and to optimize these factors in order to predict the maximum ultimate tensile strength (UTS), elastic modulus (EM), and yield strength (YS) simultaneously. To do this, two levels of nano-titanium dioxide (TiO₂), linear low-density polyethylene (LLDPE) and styrene-ethylene-butylene-styrene (SEBS) as the coupling agent were selected and eight experiments were conducted for every response. The most effective factors influencing the UTS, EM, and YS were found, and acceptable prediction regression models were taken. One noted that nanoparticles increased the elastic modulus. The attendance of high levels of LLDPE and SEBS resulted in a decrease in YS and UTS. Moreover, the optimum values of variables were determined by using the contour plot.

Keywords: elastic modulus; factorial design; nanocomposite; polypropylene; tensile strength

1 INTRODUCTION

Nowadays, polymeric products, especially polyethylene (PE) and polypropylene (PP), are developed for their low expense, good mechanical features, small weight, and other desirable characteristics [1]. Different kinds of polyethylene, containing high-density polyethylene (HDPE), low-density polyethylene (LDPE), and linear low-density polyethylene (LLDPE) are used to improve the mechanical and physical characteristics of PP [2]. Additionally, LLDPE has numerous applications, and the importance of LLDPE has increased due to its special characteristics [3].

To refine the particular characteristics of these polymers, several additives are mixed with them. Adding some micro- or nanoscale fillers may improve the disadvantages of neat polymers. Nanocomposites are polymers that are embedded with nanoscale fillers [4]. Polymer nanocomposites can modify the mechanical strength, heat resistance, elastic modulus, thermal degradation and viscoelasticity more than other traditional polymer composites [5]. By adding small values of nanoparticles, melt processing, polymer crystallization, and electric and thermal conductivity can be improved [6, 7].

Polymer nanocomposites have recently gained the attention of many material researchers. They usually study the effects of embedding various nanoparticles on the characteristics of polymer materials. Organic and inorganic nanoparticles can be utilized as reinforcement. Some of the most used inorganic nanoparticles are SiO₂, TiO₂, Al₂O₃, and ZrO₂ [3]. TiO₂ is a very special material due to special characteristics such as light density and thermal degradation [8]. The main problem in manufacturing the TiO₂ nanocomposite is its conflict with the polymer matrix, because TiO₂ is hydrophilic and the polymer matrix is hydrophobic. TiO₂ nanoparticles also have a large surface area ratio which makes them aggregate easily. To avoid this,

styrene ethylene-butylene-styrene (SEBS) can be applied as a coupling agent to improve the TiO₂ surface [9-11].

Many scientists have investigated the mechanical properties of polymers and polymer composites. Garcia et al. [12], for instance, added SiO₂ nanoparticles to the PP matrix and observed that the impact strength and elastic modulus were improved. Selvin et al. [13] found that TiO₂ nanoparticles significantly increased the elastic modulus of the polystyrene matrix. Moreover, Sirirat et al. [14] added small values of TiO₂ to the PP matrix and reported an improvement in some mechanical properties of the based material. Moreover, Altan [15] showed that, by embedding TiO₂ nanoparticles in the PP matrix, the elastic modulus of the structure increased, but its impact strength was reduced.

Ternary nanocomposites, including a system of the polymer matrix, elastomer, and filler, have newly been incorporated in different applications [16, 17]. Liu et al. [18] showed that, by adding TiO₂ nanoparticles to PP/LLDPE, some mechanical properties of the compounds were enhanced. Furthermore, Abu Ghalia et al. [17] reported that, by embedding calcium carbonate (CaCO₃) in PP/LLDPE compounds, some of their mechanical characteristics were improved. Altan and Yildirim [19] showed that structures including TiO₂ and SEBS present better mechanical characteristics compared to ones without SEBS.

Guo and Li [20] found that SEBS/titania nanocomposites showed good mechanical attributes. They also reported that the uniform dispersion of titania nanoparticles in the SEBS matrix increased the thermal stability of samples. Additionally, Nguyen et al. [9] concluded that some mechanical characteristics of LDPE/modified TiO₂ nanocomposites increased compared to the based LDPE/unmodified structures. Xue et al. [21] verified that the coexistence of organo-montmorillonite (OMMT) and nano-Cu in Cu/OMMT/LLDPE nanocomposites may improve the anticorrosion properties of samples.

Here, a 2^3 factorial design was used to supply a relation for the YS, UTS, and EM of PP/LLDPE/TiO₂ nanocomposites as a mathematical function of parameters (SEBS, TiO₂, and LLDPE). Moreover, the effect of every agent on mechanical characteristics was studied and the optimal range of each parameter was found in order to achieve the best tensile properties.

2 THE EXPERIMENTS

2.1 Materials

Polypropylene (PP-Z30S, MFR-25, 230 °C, 2.16 kg) and linear low-density polyethylene (LLDPE-0209, MFR-0.9, 190 °C, 2.16 kg, and density of 0.920 gr ml⁻¹) were procured from the Arak Petrochemical Company, Iran. The nano-TiO₂ rutile structure with the mean size of 30 nm and density of 4.23 g/cm was obtained from the Iranian Nanomaterials Pioneers (INP), Iran. Moreover, the KRATON polymer type G, namely styrene-ethylene/butylene-styrene (SEBS), was used as a coupling agent. The compound was prepared by using a co-rotating screw extruder (ZSK 25 P8.2E WLE) with a 170 to 190 °C temperature range. Next, they were made as granules. Granules were injected with the help of an injection molding machine (IMAN MACHINE 125g) with the temperature profiles of 190-200-210 °C, and the samples were hence prepared.

2.2 Mechanical Testing

The elastic modulus, yield, and ultimate tensile strength were specified by a Zwick/Roell-Z100 machine (Germany) due to the ASTM D638 standard with the strain rate of 50 mm/min at room temperature. Fig. 1 shows a sample before and after the tensile tests.

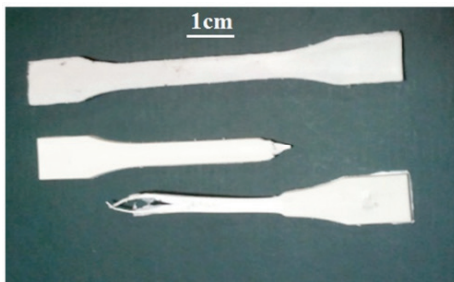


Figure 1 A sample before and after the tensile tests

2.3 Experiment Design

The factorial design of experiments (DOE) is a good and well-known procedure of testing in which all parameters are changed together in the experimental runs [22]. In this work, DOE was applied to study the effect of significant factors on the EM, YS, and UTS of PP/LLDPE/TiO₂ nanocomposites. The main aspect in DOE is the choice of control factors [23]. Here, the studied factors were LLDPE, TiO₂ nanoparticles, and SEBS. After the choice of factors, the 2^3 factorial design method was offered the levels which were coded within the

-1 and +1 range in such a way that the researcher could choose an experimental design from a list of designs.

The 2^k factorial design is one of the most widely applied designs to investigate the effects of various parameters on a particular response, where k is the number of parameters and the base 2 shows the level of treatment for each discussed parameter [22]. The performed design is shown in Tab. 1 which briefly discusses the parameters and the change of their levels. Furthermore, different modes of combining materials via the software were determined. As presented in Tab. 2, eight tests had to be prepared for each response having three replicates.

Table 1 Level of factors applied to study the effect of LLDPE, TiO₂ and SEBS

Factors		LLDPE	TiO ₂	SEBS
Level	-1 (low)	40	0	0
	+1 (high)	60	2	3

Table 2 Results of running the software

Full Factorial Design								
Sample No.	1	2	3	4	5	6	7	8
PP (Wt. %)	80	60	78	58	77	57	75	55
LLDPE (Wt. %)	20	40	20	40	20	40	20	40
TiO ₂ (Wt. %)	0	0	2	2	0	0	2	2
SEBS (Wt. %)	0	0	0	0	3	3	3	3
Random order of samples	3	2	8	1	6	4	7	5

As three parameters at two levels were assumed, the experimental design was named a 2^3 full factorial design that needed eight test runs for every response. The average of results is presented in Tab. 3.

Table 3 The experimental results for YS, UTS and EM

Sample No.	1	2	3	4	5	6	7	8
YS (MPa)	19.63	12.9	15.33	11.56	21.86	13.33	16.16	11.9
UTS (MPa)	23.63	19.5	21.86	18.3	23.93	18.03	20.33	19
EM (MPa)	223	189	226.6	207.3	212	184.6	222.6	199.3

3 RESULTS AND DISCUSSION

3.1 Analysis of Variance (ANOVA) for Tensile Strength

The p -value is described as the minimum level of importance leading to the rejection of the null hypothesis and interaction is a kind of action that occur as two or more objects have an effect upon one another [22]. F value is the measure of variation in the data about the mean. Due to the p -value described as the minimum level of importance leading to the rejection of the null hypothesis, it seems that the effect of every parameter was statistically important at the p -value of less than 0.05.

The ANOVA results for YS are presented in Tab. 4. One sees that LLDPE ($P = 0.000$), TiO₂ ($P = 0.000$), SEBS ($P = 0.001$), LLDPE×TiO₂ ($P = 0.000$), and LLDPE×SEBS ($P = 0.021$) with the p -values below or equal to 0.05 for a 95% assurance level should be statistically important for YS. In addition to that, Fisher's variance ratio (F -value) is the amount of variability from the mean. Applying the F -value, the respective significance of each parameter and its interaction would be:

LLDPE>TiO₂>LLDPE×TiO₂> SEBS>LLDPE×SEBS.

Table 4 ANOVA results for YS

Source	<i>F</i>	<i>P</i>
Main Effects	249.31	0.000
LLDPE	555.84	0.000
TiO ₂	175.14	0.000
SEBS	16.95	0.001
2-Way Interactions	21.94	0.000
LLDPE×TiO ₂	57.56	0.000
LLDPE×SEBS	6.51	0.021
TiO ₂ ×SEBS	1.76	0.203

The effects of the LLDPE×TiO₂ interaction on YS are greater than the importance effect of a single-factor (i.e. factor SEBS). The important interaction of LLDPE and TiO₂ shows that these parameters are related, i.e. if the level of one parameter varies, the effect of the other one varies, too.

The ANOVA results of UTS are presented in Tab. 5. It is clear that LLDPE (*P* = 0.000), TiO₂ (*P* = 0.000), SEBS (*P* = 0.003), and LLDPE×TiO₂ (*P* = 0.000) whose *p*-values were below or equal to 0.05 for UTS should be statistically important. Moreover, from the *F*-values in Tab. 5, the relative importance of each factor and its interactions would be:

LLDPE>TiO₂> LLDPE×TiO₂> SEBS.

Table 5 ANOVA results for UTS

Source	<i>F</i>	<i>P</i>
Main Effects	261.86	0.000
LLDPE	678.05	0.000
TiO ₂	95.35	0.000
SEBS	12.16	0.003
2-Way Interactions	27.04	0.000
LLDPE×TiO ₂	80.12	0.000
LLDPE×SEBS	0.66	0.428
TiO ₂ ×SEBS	0.34	0.569

Table 6 ANOVA results for EM

Source	<i>F</i>	<i>P</i>
Main Effects	235.29	0.000
LLDPE	553.29	0.000
TiO ₂	114.57	0.000
SEBS	38.20	0.000
2-Way Interactions	6.25	0.005
LLDPE×TiO ₂	17.82	0.001
LLDPE×SEBS	0.36	0.555
TiO ₂ ×SEBS	0.57	0.462

The EM ANOVA results are presented in Tab. 6. It is observed that LLDPE (*P* = 0.000), TiO₂ (*P* = 0.000), SEBS (*P* = 0.000), and LLDPE×TiO₂ (*P* = 0.001), whose *p*-values were below or equal to 0.05 for EM would be statistically important. Moreover, from the *F*-values in Tab. 6, the relative importance of each factor and its interactions would be:

LLDPE>TiO₂> SEBS> LLDPE×TiO₂.

The multiple regression analysis was performed on the experimentally collected data for the YS, UTS, and EM of the PP/LLDPE/TiO₂ nanocomposites. Here, the analysis was done by the Minitab® 16 software which applies the ordinary least squares technique to find the regression function.

Relying on ANOVA for YS, UTS, and EM, a fitted regression model with statistical importance was found as follows:

$$\text{Yield Strength} = 15313 - 2.888\text{LLDPE} - 1.621\text{TiO}_2 + 0.504\text{SEBS} + 0.929\text{LLDPE} \times \text{TiO}_2 - 0.312\text{LLDPE} \times \text{SEBS} \quad (1)$$

$$\text{R-sq: } 98.07\%, \text{ R-sq(Pred): } 95.67\%, \text{ R-sq(Adj): } 97.23\%$$

$$\text{Ultimate Tensile Strength} = 20.575 - 1.867\text{LLDPE} - 0.7\text{TiO}_2 - 0.25\text{SEBS} + 0.642\text{LLDPE} \times \text{TiO}_2 \quad (2)$$

$$\text{R-sq: } 98.28\% \text{ R-sq(Pred): } 96.13\%, \text{ R-sq(Adj): } 97.53\%$$

$$\text{Elastic Modulus} = 208.08 - 13\text{LLDPE} + 5.92\text{TiO}_2 - 3.42\text{SEBS} + 2.33\text{LLDPE} \times \text{TiO}_2 \quad (3)$$

$$\text{R-sq: } 97.86\%, \text{ R-sq(Pred): } 95.18\%, \text{ R-sq(Adj): } 96.92\%$$

From Eq. (2), one sees that all factors had a negative main effect on UTS. Thus, a lower factor setting (−1) would result in a higher response. In the situation of Eq. (1), an increase in LLDPE×TiO₂ and SEBS from small to high levels resulted in 2.77% and 6.76% increases in YS, whereas an increase in LLDPE and TiO₂ resulted in a decrease in YS by 31.7% and 19.14%. Thus, LLDPE had a maximum effect on YS with a 31.7% contribution. In the case of Eq. (2), by adding of LLDPE, TiO₂, and SEBS decreased UTS by 16.63%, 6.58%, and 2.40%, respectively. Therefore, LLDPE had a maximum effect on UTS with a 16.63% contribution. In Eq. (3), an increase in TiO₂ and LLDPE × TiO₂ from low to high levels resulted in 2.57% and 5.85% increases in EM, whereas an increase in LLDPE and SEBS decreased EM by 11.76% and 3.2%, respectively. Hence, LLDPE had a maximum effect on EM with a 11.76% contribution.

A verified model must predict the response with good accuracy with respect to the experimental data. Model adequacy is checked by R-Sq, R-Sq (adj), and R-Sq (pred). A R-Sq value near 100% means a reliable fit to the experimental data [22]. Based on the ANOVA results, the R-squared of the regression equations was 98.07 % for YS, 98.28% for UTS, and 97.86% for EM, which means that the model is verified. The adjusted R-square was 97.23% for YS, 97.53% for UTS, and 96.92% for EM, which accounts for the amount of predictors in the model. The prediction R-squared statistic was calculated to be 95.67% for YS, 96.13% for UTS, and 95.18% for EM. Because the predicted R-square values were near the R-square and the adjusted R-square values for every response, none of the models appeared to be overfitting and none had an adequate predictive ability [22].

3.2 Main Effects and Interaction Plot for Yield Strength

The main effects plot in Fig. 2 indicates that YS decreases as LLDPE and TiO₂ contents increase. Therefore, the maximum YS of PP/LLDPE/TiO₂ nanocomposites could be found at a lower LLDPE and TiO₂. Moreover, Fig. 2 shows that by increasing the amount of the SEBS factor increases the yield strength. The relative strength of the effect of different parameters can also be seen. The main effects

plot for YS (Fig. 2) showed that LLDPE was the most important factor.

Fig. 3 shows the interaction plot between the three discussed factors, namely LLDPE, TiO₂, and SEBS for YS, respectively. The plots, known as interaction plots, are employed to explain important interactions between process parameters. The interaction plot summarizes the interaction between the maximum and minimum amounts of each factor. From these plots, one sees that the initial interaction happened between LLDPE and TiO₂ for YS, demonstrated by non-parallel lines.

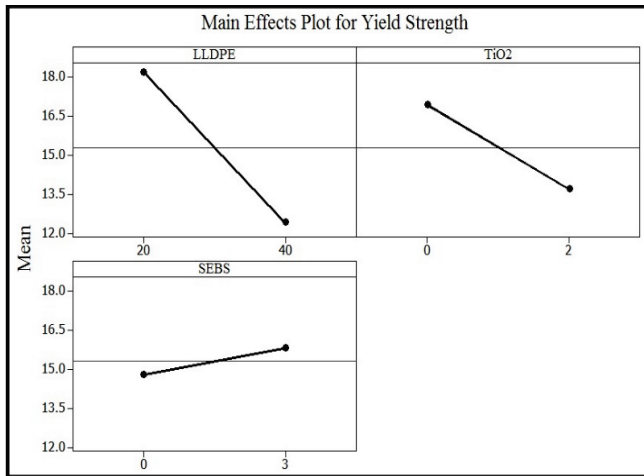


Figure 2 Main effects plot for YS

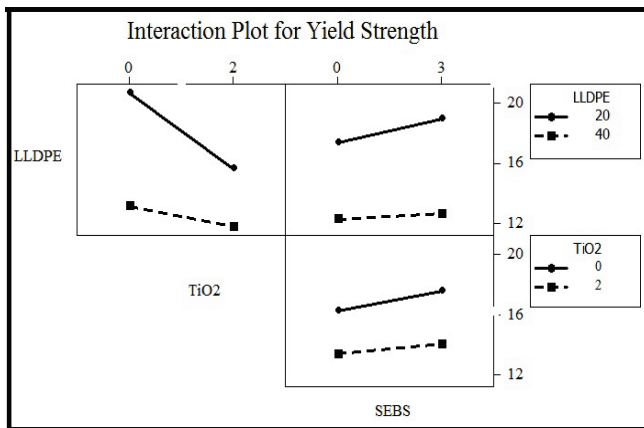


Figure 3 Interaction plot for YS

According to Fig. 3, at a low level of LLDPE (20 wt. %) and TiO₂ (0 wt. %), the interaction was very important, but adding both parameters produced a low interaction. However, the important interaction of LLDPE versus TiO₂ for YS showed that a lower TiO₂ (0 wt. %) would result in an improvement in the YS of PP/LLDPE/TiO₂ nanocomposites when factor LLDPE was under the low level (20 wt. %), while the influence of TiO₂ was reduced at the high level of LLDPE (40 wt. %). These data for YS suggested that the ideal TiO₂ of PP/LLDPE/TiO₂ nanocomposites differs from the LLDPE. The models with low TiO₂ show a large YS when LLDPE is low, and the models with great TiO₂ indicate a low YS when LLDPE is high.

3.3 Main Effects and Interaction Plot for Ultimate Tensile Strength

The main effects plot in Fig. 4 indicates that increasing the amount of all three major factors, especially polyethylene, the ultimate tensile strength is reduced. LLDPE decreased UTS for LLDPE, which was much smoother than PP. Therefore, the maximum UTS of PP/LLDPE/TiO₂ nanocomposites would be reached at a less amount of LLDPE, TiO₂, and SEBS. The relative strength of the effect of different parameters could also be seen. The main effects plot for UTS (Fig. 4) showed that LLDPE was the most important factor.

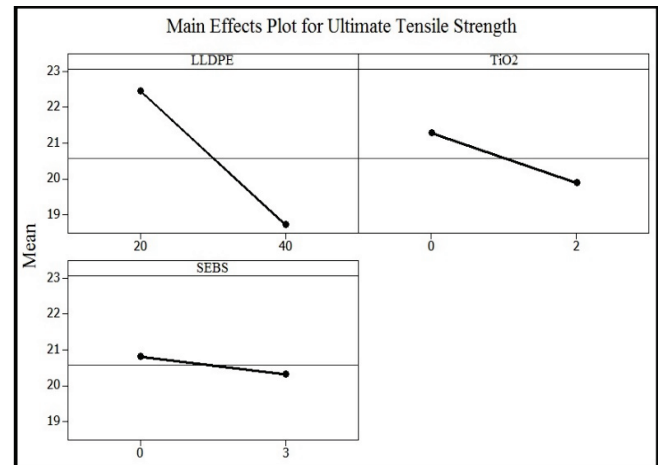


Figure 4 Main effects plot for UTS

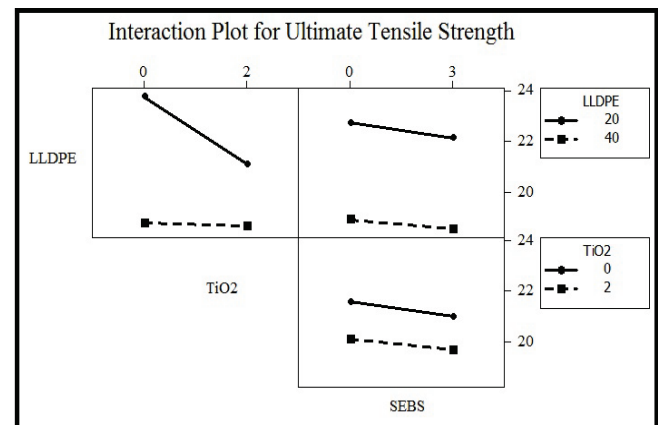


Figure 5 Interaction plot for UTS

Fig. 5 demonstrates the interaction plot between the three discussed factors, namely LLDPE, TiO₂, and SEBS, for UTS, respectively. From the plot, one sees that the initial interaction happened between LLDPE and TiO₂ for UTS, showed by non-parallel lines.

Fig. 5 shows that, at the low levels of LLDPE (20 wt. %) and TiO₂ (0 wt. %), the interaction was very important, but by adding both parameters, it produced a low interaction. However, the important interaction of LLDPE in respect to TiO₂ for UTS showed that lower TiO₂ (0 wt. %) would result in an increase in the UTS of PP/LLDPE/TiO₂ nanocomposites when LLDPE was under the low level (20

wt. %), while the effect of TiO_2 was reduced at the high level of LLDPE (40 wt. %). These data for UTS suggested that the ideal TiO_2 of PP/LLDPE/ TiO_2 nanocomposites differs with LLDPE. The models with low TiO_2 show large UTS when LLDPE is low, and the models with high TiO_2 demonstrate small UTS when LLDPE is high.

3.4 Main Effects and Interaction Plot for Elastic Modulus

The main effects plot in Fig. 6 indicates that the elastic modulus decreased as LLDPE and SEBS varied from a low to a high level. Consequently, the maximum elastic modulus of PP/LLDPE/ TiO_2 nanocomposites could be obtained at lower LLDPE and SEBS.

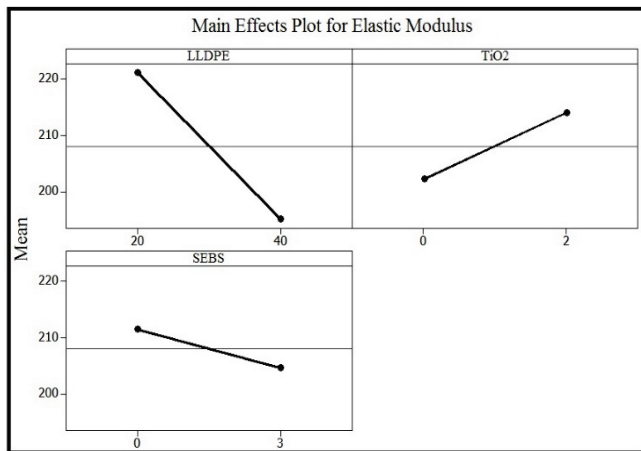
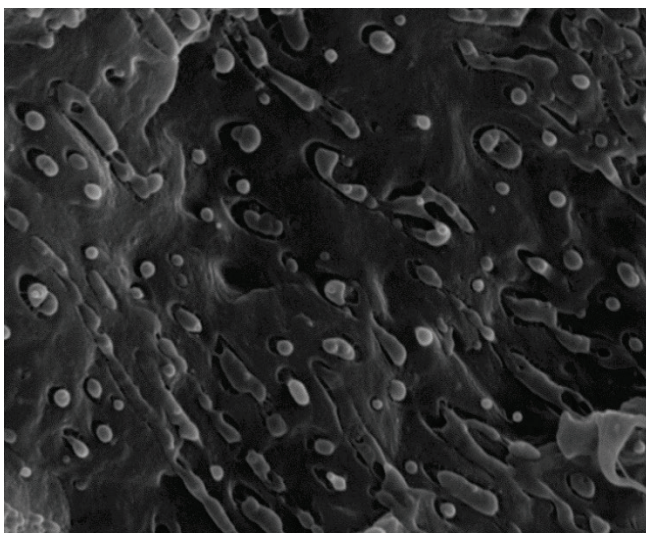


Figure 6 Main plots for EM



SEM HV: 20.00 kV WD: 12.1420 mm VEGA\\ TESCAN
SEM MAG: 15.00 kx Det: SE 2 μm
Figure 7 FESEM image taken from fractured surface sample including 2wt.% TiO_2 nanoparticles

However, regarding the mechanical properties of the variables, namely EM, a variation of TiO_2 seems to have a significantly increasing effect, similar to Selvin's [13] finding about polystyrene/ TiO_2 nanocomposites. The relative strength of the effect of different parameters may also be seen. The main effects plot for EM (Fig. 6) showed that

LLDPE was the most important factor. The elastic modulus of polymer nanocomposites largely depends on the good dispersion of nanoparticles in the matrix. Fig. 7 shows field emission scanning electron microscopy (FESEM) images taken from the samples' fractured surface. It can be observed from Fig. 7 that nanoparticles are well dispersed in the matrix. As a result, titanium oxide nanoparticles lead to an increase in the elastic modulus.

Fig. 8 illustrates the interaction plot between the three investigated parameters, namely LLDPE, TiO_2 , and SEBS, for EM, respectively. One could see that the initial interaction happened between LLDPE and TiO_2 for EM, indicated by non-parallel lines (Fig. 8). Fig. 8 shows that, at the low levels of LLDPE (20 wt. %) and TiO_2 (0 wt. %), the interaction was highly significant, and that increasing both parameters produced a good interaction and increased the EM. However, the important interaction of LLDPE versus TiO_2 for EM showed that a high TiO_2 (2 wt. %) would result in an increase in the EM of PP/LLDPE/ TiO_2 nanocomposites when LLDPE was under the high level (40 wt. %), while the effect of TiO_2 was reduced at the low level of LLDPE (20 wt. %). These data for EM suggested that the ideal TiO_2 of PP/LLDPE/ TiO_2 nanocomposites varied with LLDPE. The models with high TiO_2 show large EM when LLDPE is high, and the models with low TiO_2 indicate small EM when LLDPE is low.

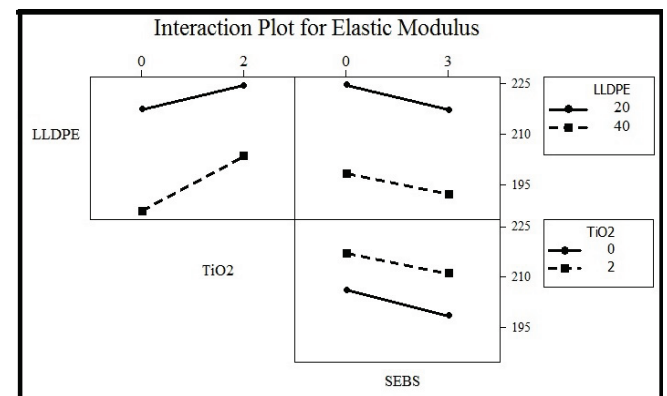


Figure 8 Interaction plot for EM

3.5 Optimal Ranges to Achieve the Best Tensile Properties

Mini-tab uses a contour plot to obtain the optimal areas of tensile properties. Contour or level plots are a method to present a three-dimensional surface on a two-dimensional plane. It graphs two predictor variables X Y on the y-axis and a response variable Z as contours. In this graphs, darker regions indicate higher responses values. They are beneficial for the creation of a favorable response. They present the contribution of two parameters simultaneously, and another parameter is retained at its middle level.

Fig. 9 presents the contour plot of the ultimate tensile strength as a function of TiO_2 *LLDPE, SEBS*LLDPE, and TiO_2 *SEBS. In any of these three modes, the third factor has been fixed in the middle level. Fig. 9 shows that to achieve the best ultimate tensile strength, low amounts of LLDPE (Less than 25 wt. %) and average amounts of titanium dioxide nanoparticles and SEBS should be used. B using this

combination, the ultimate tensile strength of more than 32 MPa can be achieved. Based on Fig. 9, the presence of high values of LLDPE (more than 35 wt.%) led to a significant reduction in the ultimate tensile strength.

Fig. 10 depicts the contour plot of the elastic modulus as a function of TiO_2 *LLDPE, SEBS*LLDPE, and TiO_2 *SEBS. In any of these three modes, the third factor has

been fixed in the middle level. It is clear that, to achieve the best elastic modulus, low amounts of LLDPE and SEBS and high amounts of titanium dioxide nanoparticles should be used. By using this combination, the elastic modulus of more than 220 MPa can be achieved. Based on Fig. 9, the presence of high values of TiO_2 led to a significant increase in the elastic modulus.

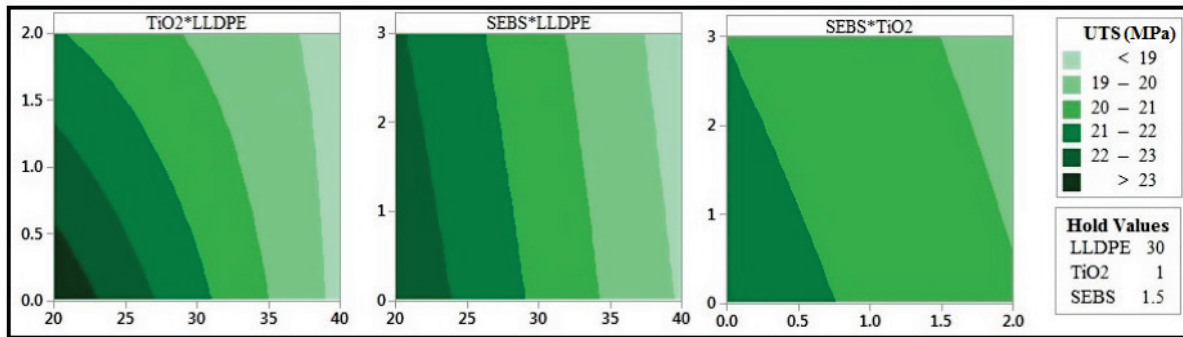


Figure 9 The contour plots of ultimate tensile strength

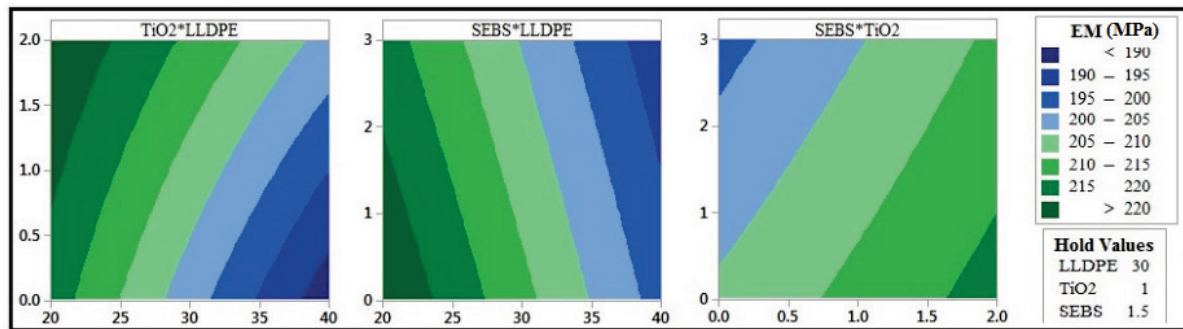


Figure 10 The contour plots of elastic modulus

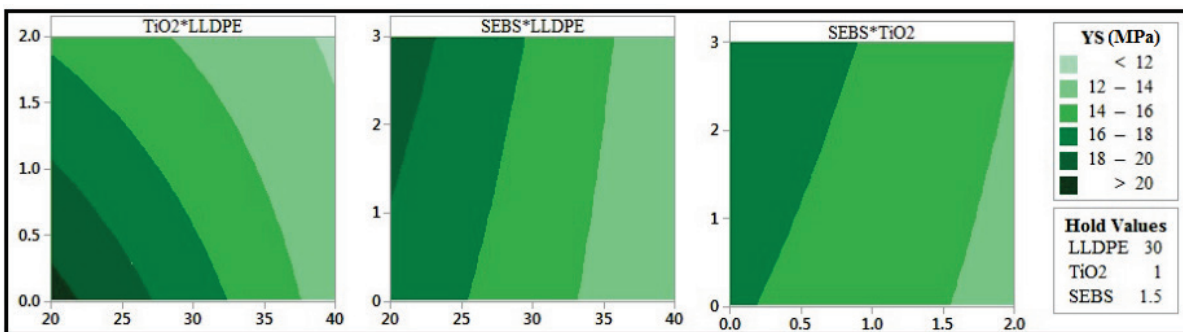


Figure 11 The contour plots of yield strength

Fig. 11 presents the contour plot of yield strength as a function of TiO_2 *LLDPE, SEBS*LLDPE, and TiO_2 *SEBS. In any of these three modes, the third factor has been fixed in the middle level. It is observed from Figure 11 that to achieve more than 20 MPa for yield strength, the smallest amount of polyethylene (about 20 wt.%) with the highest amount of nanoparticles and compatibilizer should be combined. Based on Fig. 11, the presence of high values of LLDPE (more than 25 wt.%) led to a significant reduction in yield strength.

4 CONCLUSIONS

An optimization method, in which the factorial design, mathematical modelling and contour plots were used for the prediction of the mechanical properties of PP/LLDPE/ TiO_2 nanocomposites, has been studied. The following results were obtained:

- It was observed that the most important factors were LLDPE and TiO_2 , which influenced YS, UTS, and EM, while SEBS was relatively less significant.
- From the main effects and interaction plot, one sees that by adding SEBS from low to high levels, there was a

6.76% increase in YS, whereas an increase in LLDPE led to a decrease in YS by 31.7%. An increase in LLDPE and SEBS decreased the UTS to 16.63% and 2.40%, respectively. Moreover, an increase in TiO₂ and LLDPE×TiO₂ (from a low to a high level) resulted in 2.57% and 5.85% increases in EM, respectively.

- The optimized ranges of variables on the tensile properties were found by using the contour plot. The results show that the most improved tensile properties were obtained in the low level of LLDPE and SEBS and the middle level of titanium dioxide nanoparticles.

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