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Original Article

An experimental investigation on mechanical properties of Fe_2O_3 microparticles reinforced polypropylene

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ABSTRACT

In this paper, a new polymer composite was prepared using polypropylene and Fe_2O_3 microparticles. Effect of weight fraction and microparticle size on the tensile strength and Young's modulus were studied experimentally. Also, SEM image of fracture surfaces was investigated. The experimental design of experiments was performed using a response surface methodology. Tensile test specimens consist of 5–20% Fe_2O_3 microparticles of five different sizes from 33 μm to 125 μm. The results showed that by increasing the weight fraction of the reinforcement, Young's modulus was improved compared to the pure sample, and elongation percentage was decreased. Also, as the size of the microparticles increased, the effect of the particles on the mechanical properties of the PP/Fe_2O_3 composite was reduced. For specimens containing 20wt.% of Fe_2O_3 and particle size higher than 91 μm due to the agglomeration of microparticles, the tensile strength reduced by 16%. However, if 20 wt.% of Fe_2O_3 microparticles with a particle size less than 33 μm were added, the use of these microparticles would increase Young's modulus by 300% and the tensile strength by 60%. Finally, it has been shown that dramatic improvements in the mechanical properties can be achieved by the incorporation of a suitable amount of Fe_2O_3 microparticles in polypropylene.

1. Introduction

Nowadays, polypropylene (PP) is used in a wide range of applications because of its inherent properties such as high melting temperature, relatively good mechanical properties, low density, corrosion resistance, and high chemical resistance. This thermoplastic material has many applications in various industries, such as pipe manufacturing, fiber, auto parts, and aerospace applications. But, its poor mechanical properties and low Young's modulus and tensile strength limit the applications of these materials.

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Generally, there are two ways to improve the properties of polymers: (1) combination with other polymers and (2) adding suitable types of reinforcements [1–4]. One of the most practical ways to improve and modify the mechanical properties of polymers is to add a variety of organic, inorganic, and mineral particles to them. Adding a small number of fillers, especially at the nano/microscale and forming polymeric composites, can dramatically increase their mechanical properties such and Young's modulus and tensile strength [5–8]. The use of inorganic fillers is a suitable method for improving hardness, toughness, stiffness, chemical resistance, and thermal stability of PP [9]. However, that filler agglomeration and subsequent improper dispersion in the polymer matrix lead to poor mechanical properties of the composite material [10]. Accordingly, the enhancement of mechanical properties of polymers with various nano/micro fillers such as carbon nanotube [11–14], calcium carbonate ($CaCo_3$) [15–17], zinc oxide (ZnO) [18], titanium oxide (TiO_2) [19,20] has been attracted the attention of many researchers.

Garcia et al. [21] reported a 30% increase in Young's modulus and a 68% increase in impact strength by adding SiO_2 particles to the PP polymer matrix. Cellin et al. [22] added TiO_2 particles to the polystyrene polymer matrix and observed that Young's modulus and tensile strength of the polymer increased significantly. Maiti et al. [23] reported increasing Young's modulus, tensile strength, and impact strength of PP due to adding small amounts of clay nanoparticles (about 1 wt.%) to PP. Altan and Yildirim [24] investigated morphological and mechanical properties of polypropylene and high-density polyethylene polymer composites reinforced with surface-modified TiO_2 particles. The results showed that the reinforced high-density polyethylene and PP moldings gave higher elastic modulus and tensile strength due to the rigid structure of TiO_2. Key points in enhancing the mechanical properties of reinforced polymer composites include the size, shape, properties of the fillers, interface between the fillers and the matrix, the amount of particle dispersion in the matrix, etc. [25]. Kabbani and Kadi [26] studied the effect of cooling rate conditions on the mechanical properties of glass fiber reinforced polypropylene composite. Li et al. [27] presented a new polypropylene composite filled by kaolin particles. The results of their study show that these amplifiers significantly increase the impact resistance of these composite materials.

Fe_2O_3 is one of the high-availability steel waste materials with better reinforcing properties. Environment-friendly features, low cost, high resistance to corrosion, affordability, easy production, and high load-capacity are some of the factors that can extend the use of this material as a reinforcement [28–31]. Due to the high specific surface area, high surface energy, and high surface-to-volume ratio, Fe_2O_3 particles can enhance the mechanical properties of the polymers if properly interacted with the polymer matrix and evenly distributed. Accordingly, Fe_2O_3-reinforced polymer composites have received much attention in recent years [32–34]. For example, Sun et al. [35] investigated the mechanical behavior of epoxy reinforced by Fe_2O_3 nanoparticles. The results of their study show that for 4 wt.% of Fe_2O_3 particles, tensile strength increased by 50.2%, and fracture toughness increased by 106%. Naguib et al. [29] investigated the effect of Fe_2O_3 coated particles on the mechanical properties of the epoxy matrix. Their results show that by using 3 wt.% of Fe_2O_3, Young's modulus increases from 1.45 GPa to 1.75 GPa, and the toughness increased from 300 MPa to 500 MPa compared to neat epoxy resin.

Comprehensive studies indicate that the effect of Fe_2O_3 particles on the mechanical properties of polypropylene as one of the most widely used polymers has not been studied so far. Therefore, in this study, Fe_2O_3 particles were used as the reinforcing phase of polypropylene, and by using experimental tests, the effect of these particles on the mechanical properties of this new composite was investigated. Response surface methodology was used to analyze the experimental results, and the tensile strength and Young's modulus were considered as the response. In this study, for the first time, the effect of Fe_2O_3 microparticles size and volume fraction used as polypropylene reinforcement were investigated.

2. Experimental studies

2.1. Materials

In the present study, for the composite field, propylene polymer EP440L branded by Petkim AS (Izmir, Turkey) with a melt flow index of 4.5 g/10min (230 °C/2.16 kg, ASTM D1238) and density of 905 kg/m^3 was used. Fe_2O_3 microparticle was bought from Shanghai Macklin Biochemical Co., Ltd, China, with a purity of 98%, has been used as a reinforcement. Fig. 1(a-e) shows the SEM image of the Fe_2O_3 microparticles with different sizes. Fe_2O_3 particles were used in five different sizes namely grade A ($\leq$33 μm), grade B ($33 \leq a \leq 61$ μm), grade C ($61 \leq a \leq 67$ μm), grade D ($67 \leq a \leq 91$ μm) and grade E ($91 \leq a \leq 125$ μm), where a is average grain size. The particle size and weight fractions of the samples are listed in Table 1.

Table 1 – Specifications of the samples.

No.	Fe_2O_3 wt.%	Grain size (μm)				
		$\leq$33 μm (Grade A)	$33 \leq a \leq 61$ μm (Grade B)	$61 \leq a \leq 67$ μm (Grade C)	$67 \leq a \leq 91$ μm (Grade D)	$91 \leq a \leq 125$ μm (Grade E)
1	5	PP5A	PP5B	PP5C	PP5D	PP5E
2	10	PP10A	PP10B	PP10C	PP10D	PP10E
3	15	PP15A	PP15B	PP15C	PP15D	PP15E
4	20	PP20A	PP20B	PP20C	PP20D	PP20E

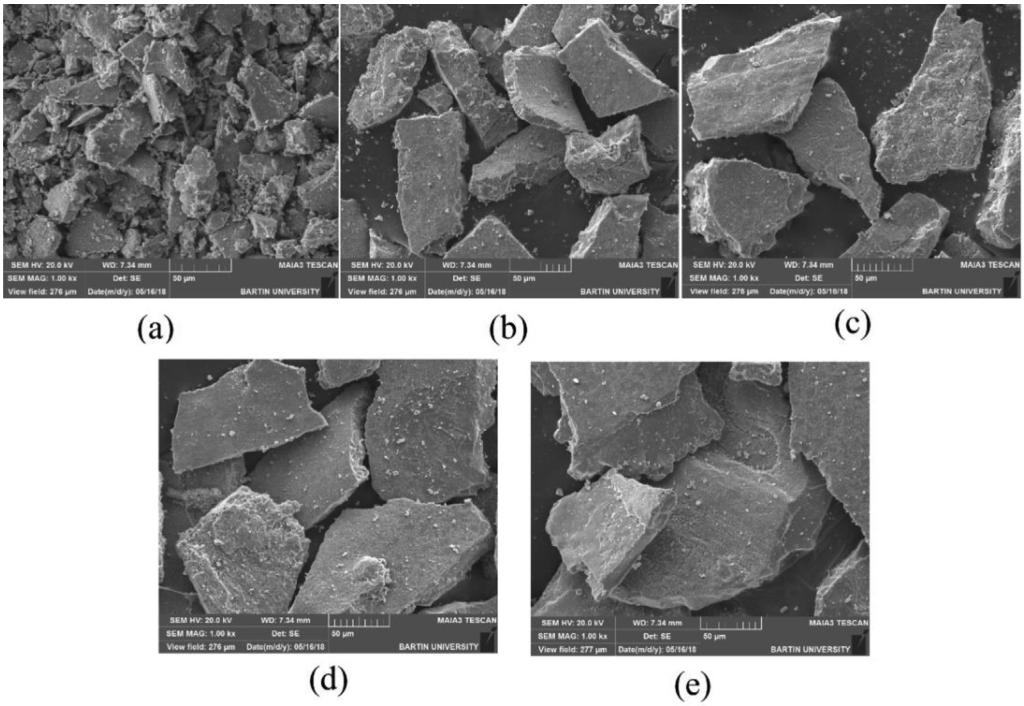

Fig. 1 – SEM image of Fe_2O_3 microparticles with different size (a) Grade A, (b) Grade B, (c) Grade C, (d) Grade D, (e) Grade E.

2.2. *Preparation of PP/Fe_2O_3 composite*

The composites were fabricated using a melt blending in a co-rotating intermeshing twin-screw extruder, with technical specifications L = 560 mm, screw diameter D = 16 and L/D = 40. The temperature distribution of the extruder consisting of six heat zones was adjusted according to the melting temperature of the polypropylene material at the material input of 50, 195, 200, 200, 195, and 190 °C, respectively. After extrusion, a plastic injection device was used to prepare different test specimens with different weight percentages and sizes of Fe_2O_3 particles under constant process conditions, according to Table 1. The device at the molding time has a temperature distribution of 160–180 °C, loading speed of 45 rpm, the injection pressure of 90 bar, and a cooling time of 65 s. The samples were made for five different volume fraction of Fe_2O_3 particles (0%, 5%, 10%, 15%, and 20% wt.) and five different sizes of these particles by standard injection molding as presented in Table 1.

The tensile test was performed according to the ASTM D638 standard. The dimensions of the desired specimens are shown in Fig. 2. For this test, the SHIMADZU AG100 kN universal testing machine was used, and the test speed was set to 5 mm/min. Force-displacement data were recorded every 1 ms until sample failure. Also, to eliminate any possible errors during the test, an average of three trials per sample was reported. Figure 3 shows some of the fabricated samples. The experiments will be analyzed using the response surface methodology, and Table 1 shows the design factors or variables selected in this research and their range of variation according to the research objectives. In this study, the Box-Behnken method was used in the response surface methodology.

Fig. 2 – Dimensions of tensile test specimens according to ASTM D638.

3. Results

In this section, the experimental test results obtained in this study are presented. The results are extracted by direct tensile tests on different PP samples containing Fe_2O_3 microparticles in different sizes and weight fractions. The elastic modulus is equal to the slope of the linear portion of the stress–strain curve, and the tensile strength is obtained from the maximum amount of stress applied to the sample. The

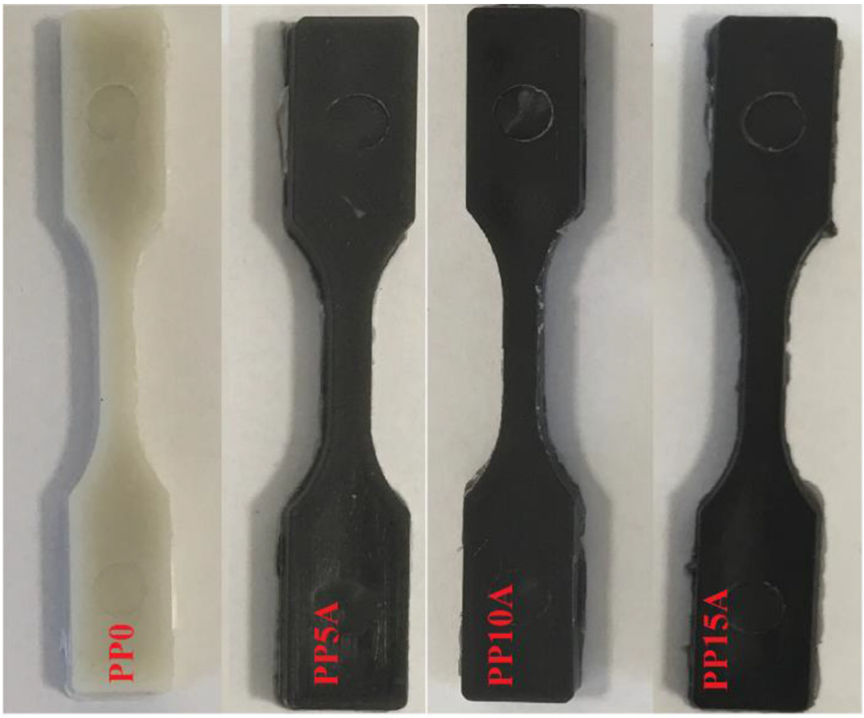

Fig. 3 – Tensile test samples produced using PP/Fe_2O_3 Composite.

stress–strain curve for the three pure PP, PP5E, and PP20E samples (see Table 1) is shown in Fig. 4(a-c). The quasi-static specimen failure mode of ASTM tensile tests for pure PP, PP20A, and PP20E samples are shown in Fig. 5(a-c), respectively. The results show that in general, the use of Fe_2O_3 microparticles increased the strain variations of the PP specimens and consequently decreases the fracture strain of the specimens, which can be seen in Fig. 5 by comparing the specimen deformation after the tensile test. The presence of Fe_2O_3 microparticles in the polymer matrix resembles pins with proper bonding to the matrix and increases the tensile strength during the tensile load. But on the other hand, with increasing tensile force, the areas around microparticles have lower strength, which are areas for crack initiation and crack growth in the matrix. The polymer composite begins to tear point in these areas.

According to the results of Fig. 4, it can be seen that Young's modulus and tensile strength for pure PP were 630 MPa and 22 MPa, respectively which by adding 20 wt.% of Fe_2O_3 microparticles with grade A particle size, these values reached 2516 MPa and 35 MPa, respectively. Therefore, the use of grade A Fe_2O_3 microparticles increased Young's modulus by 300% and the PP's tensile strength by 60%. These results illustrated that Young's modulus and tensile strength would be readily improved by adding Fe_2O_3 particles. Similarly, for PP with 20 wt.% of grade E particles, Young's modulus and tensile strength were 2280 MPa and 19.4 MPa, respectively. By comparing these values with the corresponding values of pure PP, it can be seen that grade E microparticles increase Young's modulus by about 260% but decrease the tensile strength of the sample by about 16%.

Figure 6(a) and (b) shows the SEM image of the tensile fracture surfaces of the PP20A and PP20E samples, respectively. These figures show the areas of microparticle agglomeration, voids, fracture, and pulled out of microparticles. Also, in this figure, a relatively appropriate distribution of small size

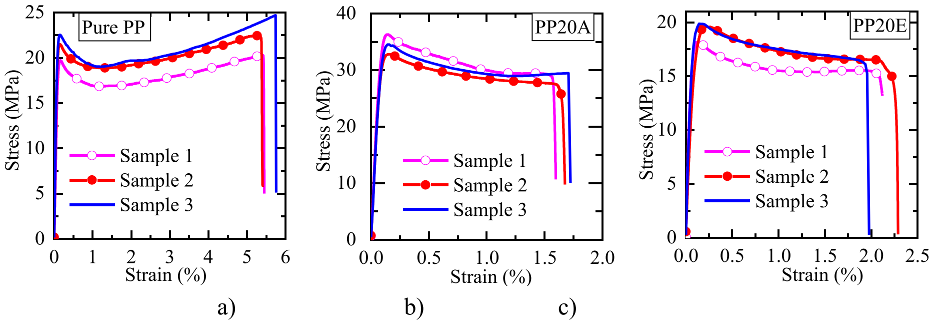

Fig. 4 – Stress–strain curves of samples (a) pure PP, (b) PP containing 20 wt.% Fe_2O_3 microparticles with grade A particle size ($\leq$33 μm) and (c) PP containing 20 wt.% Fe_2O_3 microparticles with grade E particle size (91 $\leq$ a $\leq$125 μm).

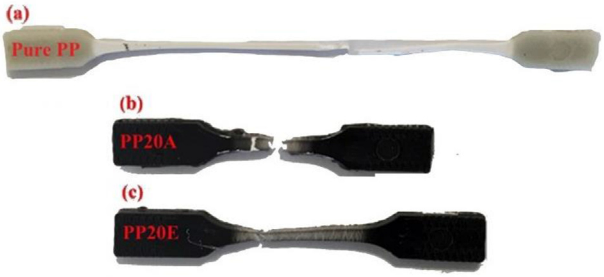

Fig. 5 – PP/Fe_2O_3 composite samples failure mode of the tensile test (a) pure PP, (b) PP contains 20 wt.% of Fe_2O_3 microparticles with grade A particle size (μ33μm) and (c) PP contains 20 wt.%. of Fe_2O_3 microparticles with grade E particle size (91 ≤ a ≤125 μm).

microparticles (Grade A) in the polymeric matrix is observed. The images show that the fracture surfaces of the samples with larger microparticles have higher non-uniformity, which by decreasing the particle size, the fracture surfaces become smooth. Also, in samples with large microparticle sizes, agglomerations were found that cause stress concentration and create areas for crack initiation that eventually lead to a brittle material. Also, weak bonding between large microparticles and matrix reduces the tensile strength. Therefore, these results show that the use of smaller microparticles has a significant effect on improving the mechanical properties of PP by increasing the cohesive level and lack of particle aggregation.

Figure 7 shows Young's modulus of 20 different investigated PP samples reinforced with Fe_2O_3 microparticles with different weight fraction and particle size, as given in Table 1. The results show that the increase in Young's modulus caused by the reinforcement particles loading seems to be depended on the amount and size of addition particles in polymer matrix which is consistent with the results presented in other research [36–38]. The addition of Fe_2O_3 microparticles, depending on the weight fraction and size of the microparticles, increased Young's modulus from 105% to 260% respect pure PP. The elastic modulus of the composite material is dependent on the ratio of the reinforcement modulus to the matrix modulus. Since Fe_2O_3 has a higher modulus than PP, so the PP/Fe_2O_3 composite modulus increases by increasing Fe_2O_3 microparticles weight fraction. The remarkable result of the experimental tests is that for samples with a Fe_2O_3 weight fraction of less than 10%, Young's modulus increases by increasing microparticle size. However, if the PP is strengthened with a weight fraction greater than 10 wt.%, the results for Young's modulus are entirely reversed, and the large microparticles have less effect on Young's modulus of the composite material. The lower modulus in higher percentages of Fe_2O_3 microparticles with large sizes (C and D grades) is because of the adverse effects of particle agglomeration and the lower effective bonding level of the microparticles and matrix.

Figure 8 shows the effect of the Fe_2O_3 microparticles on the tensile strength of PP reinforced with these microparticles. The weight fraction and size of the microparticles used have a significant effect on the tensile strength of the PP/Fe_2O_3 composite and behave differently depending on the size of the microparticles used. It is also believed that the enhanced interfacial adhesion between PP polymer and the Fe_2O_3 microparticles, results in an increase of the tensile strength and Young's modulus of the sample as compare with the pure sample. Generally, the mechanical properites of the PP/Fe_2O_3 composite reflect the nature of the interface between polymer matrix and reinforcement particles. For grade A microparticles, the tensile strength increased by increasing weight fraction, and the highest tensile strength was observed at 20 wt.%, in which the tensile strength of the composite was about 56% higher than pure PP. For B, C, and D microparticle grades, by adding Fe_2O_3 to PP, the tensile strength initially increased and then decreased. Since the tensile strength is strongly dependent on the stress transfer between the microparticles and the matrix, so when the effective surface contact area is more, proper contact between the microparticles and the matrix is established. Therefore, the applied stress is transferred from the matrix to the microparticles, and this improves the tensile strength of the composite. Based on this, it can be stated that the initial increase in strength is due to more surface contact area and desirable cohesive surface

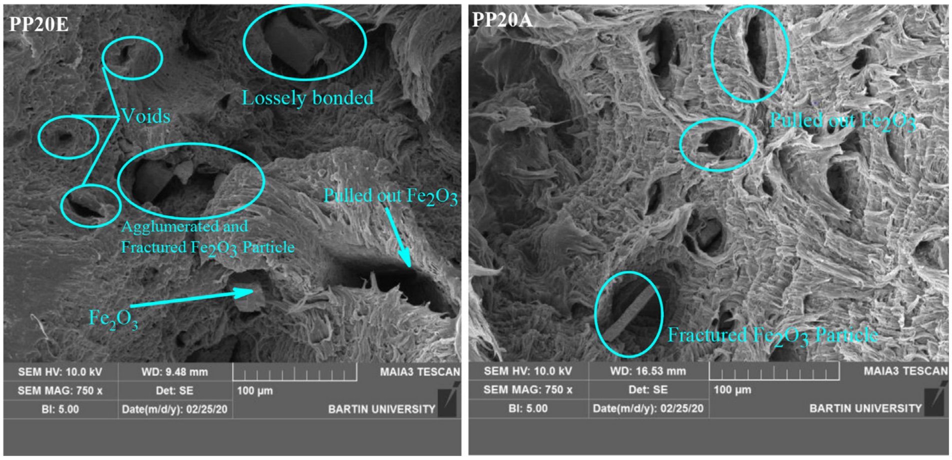

Fig. 6 – SEM image of the fracture surface of different PP samples containing 20 wt.% of Fe_2O_3 microparticles (a) Grade A particle size (≤33 μm) and (b) Grade E particle size (91 ≤ a ≤125 μm).

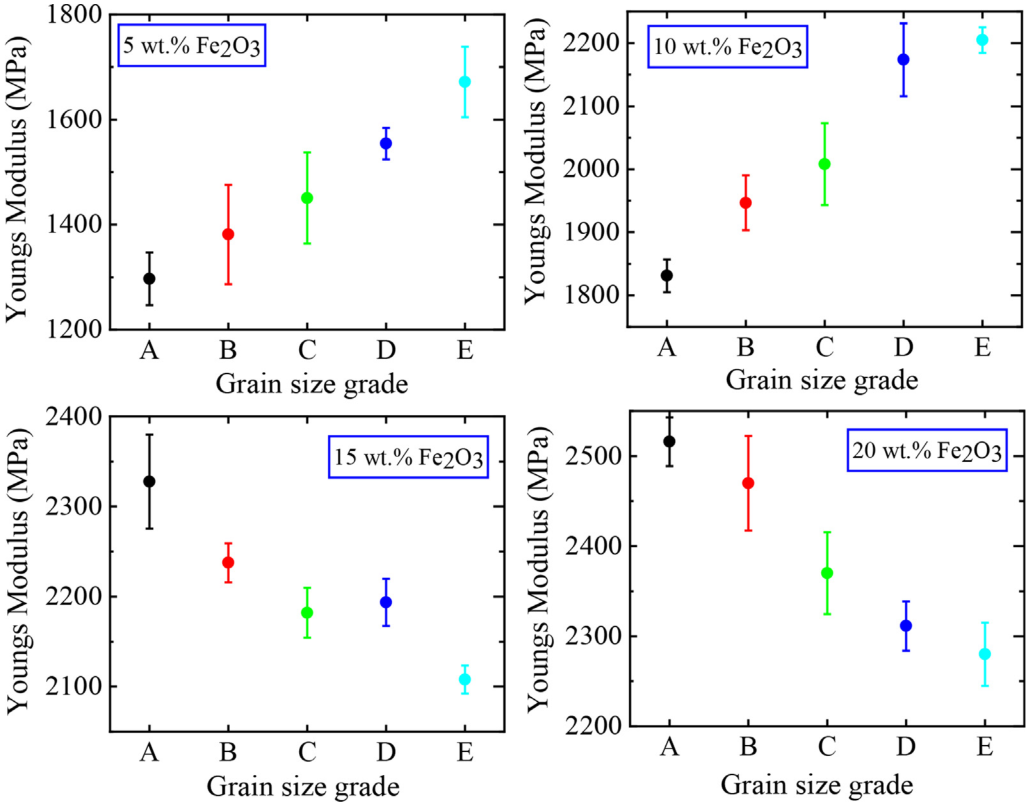

Fig. 7 – **Effect of different weight fractions and Fe_2O_3 microparticles size on Young's modulus of PP/Fe_2O_3 composite.**

between the microparticles and the matrix, and the highest increase was related to the PP/Fe_2O_3 composite containing 15 wt.% of grade B microparticles size, which the tensile strength value was obtained 37.5 MPa and was about 67% higher than the tensile strength of pure PP. This increase in strength is due to the reinforcing effects of Fe_2O_3 microparticles, which strengthens its matrix and diverts the crack growth path. The decrease in strength of PP/Fe_2O_3 composite containing more wt.% of Fe_2O_3 levels can be attributed to the weak areas. Besides, at high amounts of Fe_2O_3 with large sizes, Fe_2O_3 agglomerations are present, which causes weak interaction of the matrix and particles. Also, there is a high-stress concentration around the agglomeration regions, which provides the conditions for crack growth. All these reasons may decrease the strength of the micro composite by increasing the weight fraction and increasing the size of Fe_2O_3 microparticles. The lowest increase of tensile strength was observed for grade E microparticles. As the microparticles weight fraction increased, the tensile strength of the composite decreased due to microparticle agglomeration and decreasing the surface contact area, which even at 20 wt.% of Fe_2O_3, the tensile strength of the resulting micro composite is about 15% lower than that of pure PP. It should be mentioned that tensile strength and modulus of PP composite prepared in this work (see Figs. 7 and 8) are also simiular than those of specimens prepared in references [12,39] which reinforcement type is clay and aluminum hydroxide particles.

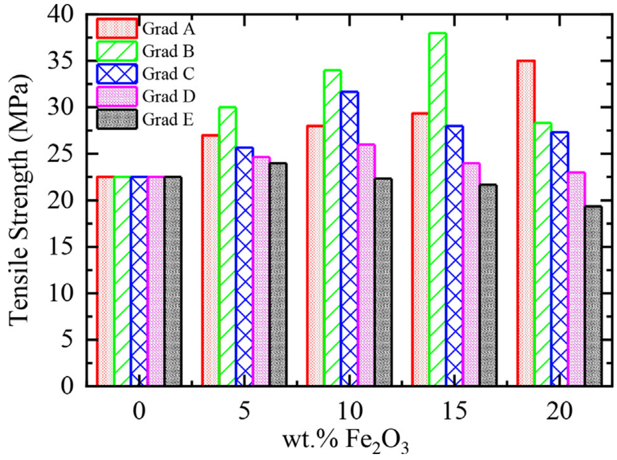

Fig. 8 – **Effect of weight fractions and grain size of Fe_2O_3 microparticles on tensile strength of PP/Fe_2O_3 composite.**

In this study, a design experiment was performed using the response surface methodology to reduce the number of experiments and to achieve a quantitative relationship

Table 2 – **Statistical analysis and accuracy of different mathematical models for the experimental data.**

Models	Standard deviation	R^2
Linear	1.78	0.87
2F1	1.53	0.88
Quadratic	1.04	0.95
Cubic	0.57	0.99

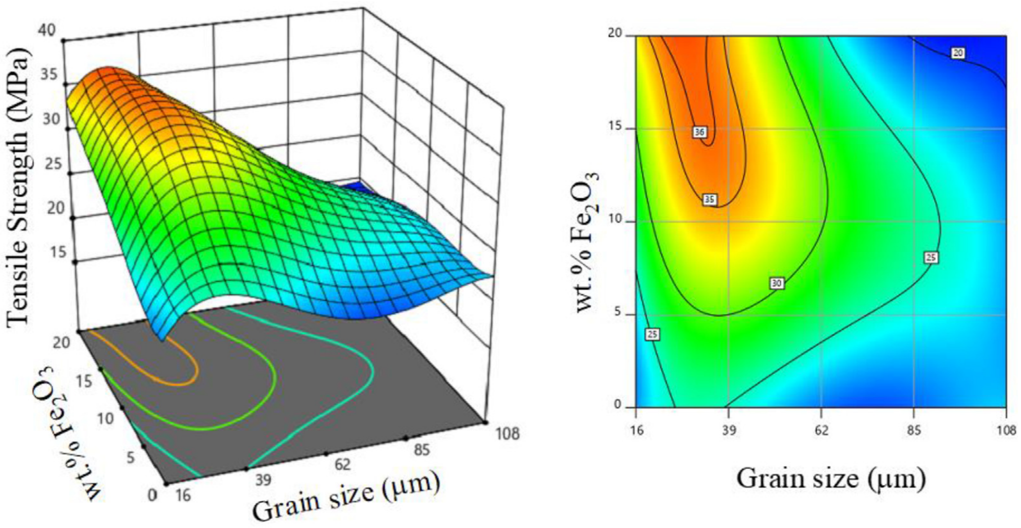

Fig. 9 – Contour plot and response surface for tensile strength of PP/Fe_2O_3 composite (standard deviation = 0.57).

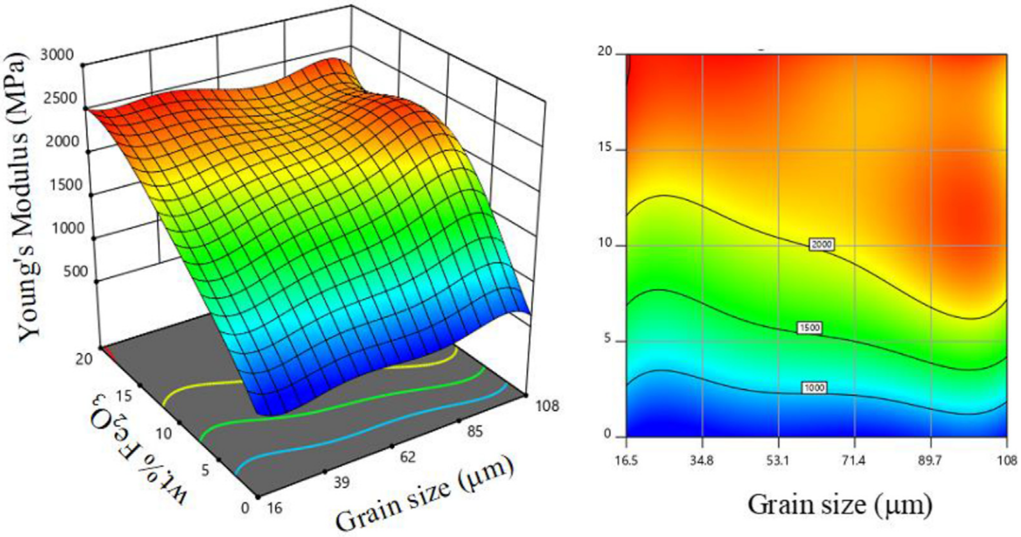

Fig. 10 – Contour plot and response surface for Young's modulus of PP/Fe_2O_3 composite (standard deviation = 0.57).

between the mechanical properties of the composite (tensile strength and Young's modulus) and the input variables (the weight fraction and size of Fe_2O_3 microparticles). Different mathematical models can be used to predict the effect of factors on the mechanical properties, in which the accuracy of some of these models for the present research data is presented in Table 2. Based on statistical analysis, the cubic model with the least error was used to analyze the data and determine the contribution of each parameter effect. Based on this cubic model with 0.57 standard deviation, the surface curves obtained by the response surface method for Young's modulus and tensile strength of PP/Fe_2O_3 composite are shown in Figs. 9 and 10. The bonding between the matrix and the microparticles is more robust in the sample contains high weight percentages of Fe_2O_3 microparticles with smaller aggregates due to a more effective surface area. Therefore, most of the applied forces to the matrix are tolerated by these microparticles, and the mechanical properties of these composites are substantially improved. The results show that the highest tensile strength corresponding to the specimens with microparticles size in the range of $20 \leq a \leq 35$ and weight fraction in the range of 15 wt.% to 20 wt.%, which is visible in Fig. 10.

Based on the statistical analysis of the data, the comprehensive mathematical model for tensile strength and Young's modulus according to the cubic model are presented in Eqs. (1) and (2), respectively.

$$\begin{aligned}\text{Tensile Strength (MPa)} = {} & 28.85 - 10.30S + 2.91W - 4.01SW \\ & - 2.56S^2 - 3.54W^2 + 3.65S^3 \\ & - 0.77W^3 - 0.67SW^2 \end{aligned} \tag{1}$$

$$\text{Young's Modulus (MPa)} = 1984.6 + 174.6S + 692.3W$$

$$- 111SW - 5.6S^2 - 488.4W^2 - 32.7S^3 + 190.8W^3 - 216.8SW^2 + 1.8S^2W \qquad (2)$$

where variables S and W represent the microparticles size and weight fraction of Fe_2O_3 microparticles, respectively. In the equation for tensile strength, the highest coefficient S is negative for the microparticle size. The SW coefficient also shows the interaction between size and weight fraction of microparticles is negative, indicating that shows the increase in size and weight of microparticles have adverse effects tensile strength. As the size and weight percentage of the microparticles simultaneous increase, the agglomeration probability of the microparticles increases. Therefore, the tensile strength decreases according to the results shown in Fig. 7, and the effect of these coefficients is physically justified.

4. Conclusion

In this paper, the effects of Fe_2O_3 microparticles on the mechanical properties of PP reinforced by these particles were investigated. For this purpose, standard PP polymer tensile specimens reinforced with Fe_2O_3 microparticles were fabricated at different weight fractions (5, 10, 15 and 20 wt.%) and five different particle sizes by melt mixing and injection. The response surface methodology was used to analyze the experimental results, and the tensile strength and Young's modulus were considered as the response. SEM images taken from the fractured surfaces of the samples showed that increasing the percentage of microparticles with larger sizes reduced the distribution uniformity and increased particle agglomeration. The presence of microparticles with smaller sizes increases the effective total surface area and uniformity in dispersion. It decreases particle agglomeration resulting in a significant increase in the tensile strength and Young's modulus. In general, the addition of Fe_2O_3 microparticles increases Young's modulus of PP/Fe_2O_3 composite compared to pure PP, and Young's modulus increased between 100 and 260% depending on the weight fraction of the microparticles used and their size. The increase of the suitable properties of PP/Fe_2O_3 composites containing a high weight percentage of microparticles with size less than 61 μm indicates the ability of this process to obtain PP/Fe_2O_3 composite with suitable mechanical properties for many engineering applications. Therefore, Fe_2O_3 microparticles become suitable transporter for their polymer matrix and effectively transfer the force from polypropylene. Another reason for this strength increase is the strong interconnection surface effect and strong adhesion between Fe_2O_3 microparticles and matrix, and the reinforcing effects of these microparticles, which strengthens their matrix and diverts crack growth. Also, the highest tensile strength increase was related to the PP/Fe_2O_3 composite containing 15 wt.% of microparticles with $20 = a \leq 35$ μm size, in which the tensile strength value was obtained 37.5 MPa and was about 70% higher than the tensile strength of pure PP.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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