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*Corresponding author

Abdulbasit Abdulqadir Hamza abdulbasit.hamza@su.edu.krd

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Fabrication of High-Density Polyethylene Matrix Surface Composite Reinforced by Various Powder Materials through Friction Stir Processing

Abdulbasit Abdulgadir Hamza*, Shawnam Rashied Jalal

Department of Mechanical and Mechatronics, College of Engineering, Salahaddin University-Erbil, Erbil, Kurdistan Region, Iraq

ABSTRACT

Friction Stir Processing (FSP) is a promising technique to improve the surface properties of materials. Compared to metals, the FSP of polymeric materials was less studied. Therefore, in the present work, high-density polyethylene (HDPE) surface composite was fabricated by the addition of reinforcements such as Graphene (C), Silicon carbide (SiC), Silicon dioxide (SiO₂), and Copper (Cu) powders. The effect of FSP parameters such as tool rotational speed (478, 679, and 925) rpm, traverse speed (22, 37, and 51) mm/min, and amount of added particles (10, 15, and 20) % on the mechanical properties of the fabricated samples were studied. The results showed that as the reinforcement powders were added to the matrix material, led to enhancements in each of the tensile strength, microhardness, and impact strength. At the optimum set of process parameters, the graphene-reinforced composites have better improvement in terms of tensile and impact strength where increased by (47.2 and 55.6) % respectively. On the other hand, in terms of microhardness, the SiC-reinforced composite exhibited greater augmentation, registering 20.16 %. While, the elongation of all fabricated composites decreased in comparison to the parent material. Based on the table of analysis of variance (ANOVA), it is noticed that the tool rotational speed has the largest effect on the quality of the products among the other parameters, followed by tool traverse speed and the amount of particles has the least contribution and effect. Considering the information provided, the friction stir process method can be used to repair cracks and other defects in polymeric materials.

1- Introduction

High-density polyethylene (HDPE) is one of the many forms of thermoplastic polymers that are accessible widely in the world (Hoseinlaghab et al., 2015). It is utilized in a range of applications, such as pipelines, water and food containers, cutting boards, and chemical storage tanks, because thermoplastic polymers are inexpensive. have high chemical resistance, recyclable, and can be reshaped (Hamza and Jalal, 2022, Karimi et al., 2021). Unfortunately, due to their poor mechanical properties and absence of special surface characteristics, polymers alone are not modern appropriate for and heavy-duty applications (Oleiwi et al., 2018). Therefore, appropriate reinforcing particles are added to the polymer matrix to create polymer matrix composites (PMCs) (Kaka, 2022). These particles can be metallic, ceramic, or polymeric. Due to their comprehensive properties of both reinforcement and polymer material such as flexibility, high strength-to-weight ratio, easy processability, and low cost, polymer matrix composites are increasingly being used in place of some metal parts in different engineering fields as in producing gears, bearings, seals, joints, food conveyors, and other components (Iftikhar et al., 2021, Butola et al., 2022).

However. the PMC has characteristics comparable to some metals, but in cases requiring high surface properties like hardness, tensile strength and resistance to wear for different applications in the industries, it is preferable to enhance the surface of the workpiece without affecting its bulk (Rostamiyan and Zaferani, 2019, Rudrapati, 2022). The mechanical properties of the reinforced polymer composites are largely determined by the type of filler material employed (Kumar et al., 2025), the and amount of reinforcements dispersion (Muhammad and Jalal, 2022), and methods by which composites are made (Salih et al., 2018, Yuan et al., 2021). Enhancing the surface properties of the polymer matrix is mostly dependent on the dispersion of powders in the polymers (Azhiri et al., 2018). Several techniques have been developed for producing polymer matrix composites such as melt mixing (Madhu et

injection molding (Battisti and 2014). Friesenbichler, 2013), vacuum arc deposition (Zhitomirsky et al., 1999), and mechanical milling (Zebarjad, 2007). These techniques often use melted polymers in the mixing procedure. The aggregation of additive particles and their uneven distribution in the produced composites are just one of the several disadvantages of the processes mentioned. Therefore, a revolutionary composite production technology known as the friction stir process (FSP) has received a lot of attention recently. friction stir process is an adaptation of the friction stir welding process, and it was initially used for the first time as a keyword in the work of Mishra et al in 1999.

The FSP was successfully developed after 2003 as a technique for producing surface composites of light materials to enhance the mechanical and surface qualities of the materials. (Khan et al., 2019, Mofakhami et al., 2024).

To improve the distribution of nano-clay particles and mechanical properties of the surface, Barmouz et al (Barmouz et al., 2011) created high-density polyethylene (HDPE)-clay nanocomposites utilizing an innovative technique based on FSP. According to the findings, the samples' microhardness increased by 62% and also, they concluded that the surface modification of polymeric materials by FSP has enormous potential. (HDPE)/copper composite was produced by Azarsa and Mostafapour (Azarsa and Mostafapour, 2013). An innovative tooling system was used with the FSP that had an internal heating system, a rotating pin, and a stationary shoulder. According to the research findings, at tool linear speed of 60 mm/min and shoulder temperature of 110 °C, ultimate tensile strength and modulus of elasticity improved by 10% and 30%, respectively. Hamed Aghajani et al (Derazkola and Simchi, 2018). Studied the effect of Alumina (Al₂O₃) nanoparticle volume fraction (up to 20%) on the wear behavior and mechanical characteristics of the (poly methyl methacrylate) matrix composite fabricated by FSP technique. The findings showed that the tensile, flexural strength, impact energy, and hardness of PMMA-based nanocomposites slowly increased with increasing nanoparticle volume percentage. While the wear rate is

reduced when the volume percentage of nanoparticles Farshbaf increases. Zinati (Farshbaf Zinati, 2015) has developed an ultrasonic-assisted friction stir process (UAFSP) for dispersion of carbon nano-tube particles in polyamide 6 base material. They demonstrated that as the traversal speed is increased, the stir also increases. This is attributed to the amplified energy that imposed by ultrasonic waves. On the other hand, ultrasonic vibration accelerates the synthesis of composite materials without changing their hardness, homogeneity, or dispersion.

The aim of this study in comparison with previous investigations, is to use FSP to create a HDPE surface composite by adding various reinforcing particles. The tensile strength, impact strength, hardness, and elongation of the samples were examined experimentally to ascertain the impact

of tool rotating speed, tool linear speed, amount of reinforcement, and type of the added powder material. The optimum set level of the selected parameters and the level of importance and percentage contribution of each parameter on the output performance were also determined.

2- Experimental Procedure

2.1- Materials

In the present study, High-density polyethylene (HDPE) as a polymer matrix which was provided by the TURKAY Plastic Company (Turkey) and Graphene (C), silicon carbide (SiC), silicon dioxide (SiO₂), and copper (Cu) as the reinforcement powder which were packaged by Pourian Chemical Company (Iran) were used. Table 1 includes information on the mechanical properties of the as-received HDPE sheet, as well as the size and purity of the reinforcing particles.

| HDPE properties | | | | | | | | | |
|---------------------------------------|-----------------------------------|-------------------------------|---------------------|-------------------|--------------------|--|--|--|--|
| Ultimate Tensile Strength (MPa) | Modulus of Elasticity (MPa) | Impact Strength (KJ/m²) | Shore D Hardness | Elongation (%) | Density (g/cm³) | | | | |
| 23.2 | 860 | 15.6 | 62 | 93 | 0.950 | | | | |
| Reinforcement Powders | | | | | | | | | |
| Powder Name | e and Symbol | Average Partic | le Size (µm) | Purity (%) | Made in | | | | |
| Graphe | ene (C) | | over 99 | India | | | | | |
| Silicon car | bide (SiC) | 10 - | 45 | over 99 | Japan | | | | |
| Silicon dioxide (SiO ₂) | | | | over 99 | China | | | | |
| Copper (Cu) | | | | over 99 | Germany | | | | |

Table 1. HDPE and Reinforcement Powders Specifications.

2.2- Friction Stir Process Machine and Accessories

The universal (Knuth UWF4 model) milling machine was utilized to produce a surface composite of the HDPE polymer. Some specific accessories are required to be installed on the bed of the machine. The accessory which is shown in Figure 1 aid in securing the workpiece firmly and preventing it from sliding or moving as a result of the force produced during the FSP. The produced accessory comprises of two special strips and a flat backing plate. The flat backing plate is fastened to the machine bed by four bolts and also each strip is fastened against

the backing plate by three bolts. The backing plate and the strips are made from 20 mm-thick carbon steel.



Figure 1. Workpiece Clamping Tool

2.3 - Friction Stir Processing Tool System

Although conventional FSP tools are very effective for metals like aluminum alloys, but when used to process plastic materials, they do not reach the desired results. Therefore, the tooling system has been improved. Figure 2 illustrates the used tool system in this study which consists of a stationary shoe and a rotating tool. Due to the utilization of a thrust bearing, the shoe remains stationary in relation to the rotating tool. The tool's shoulder and pin both enter the workpiece during friction stir process to produce enough heat for the processed area. Because

the friction between the tool and the treated material generates heat during the FSP process. The tool shoulder is designed concavely to keep soften polymer material from escaping underneath it and, on the other hand, forces it back into the treated area. Additionally, the conical pin shape with two grooves that improve friction at the tool and material interface also helps to decrease pileup and polymer sticking on the tool pin (Raza et al., 2018). Preventing the reinforcing particles from pushing out of the groove is the purpose of the stationary shoe.

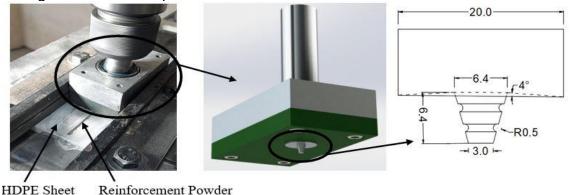


Figure 2. Stationary Shoe Tool System with Shoulder and Pin Geometry (dimensions are in millimeter)

2.4 – Experimental Setup

Experimental work was divided into four groups based on the reinforcing materials. The friction stir process parameters and their levels were identified by going through a series of experiments during this investigation and they are presented in Table 2. Due to the large number of FSP parameters and their levels, it was decided to use the L9 orthogonal array from

the Taguchi method. Because the Taguchi method is a statistical tool developed by Genier Taguchi (1940's) a Japanese engineer, suggested a model for design of experiment. Instead of having all possible experiments like full factorial design, Taguchi model provides a minimum number of experiments. Because of this, it reduces time, waste materials and costs. According to the Taguchi method, in this

investigation nine runs are required for each reinforced powder. Αt the beginning of experimental work, the provided HDPE plates were cut into sheets with dimensions of (250, 80, 10) mm and used as the polymer matrix. The sliced polymer sheet was clamped and a groove of 2 mm in width was machined into the middle of each HDPE plate in the longitudinal direction to pre-place strengthening particles in the polymer (Fig. 1). Due to the variation in the volume fraction, the groove depth varies, and it is equal to (1.4, 2.1, and 2.8) mm, which was calculated by using the fractional volume percentage expression given below (Sharma and Tripathi, 2022).

Fractional Vol. % = $\frac{\text{Groove area}}{\text{Tool pin area (projected)}} *$ 100..... (1)

Groove area = Width of groove *

Depth of groove (2)

The projected area of the tool pin is equal to 28.5 mm² which was determined by using the SketchUp program. The channels were filled with strengthening powder and compressed to fill

completely and then FSP was carried out on the attended polymer surface.

Table 2. Processing Parameters and their Levels

| parameters | Symbol | Unit | Levels | | |
|------------------|--------|--------|--------|-----|-----|
| Rotational speed | ω | rpm | 478 | 679 | 925 |
| Travers speed | f | mm/min | 22 | 37 | 51 |
| Volume fraction | V | % | 10 | 15 | 20 |

In the FSP operation, the tool was rotated clockwise and the tool probe was lowered gently until the stationary shoe contacted the matrix surface then the rotated tool stayed in its place for 30 seconds to soften the specific area of the matrix and then the tool started to linear movement along the filled channel to produce a surface composite. Following the completion of the FSP, the sheets were let to cool to room temperature for nearly ten minutes and then taken off from the fixture. The fabricated samples are shown in Figure 3 and then the prepared samples are cut according to the dimensions of each test.

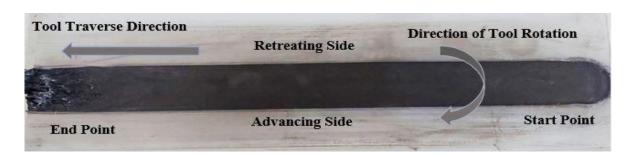


Figure 3. Prepared Sample by FSP

2.5- Mechanical Testing

Samples were cut longitudinally from the middle of the composite for the tensile, impact, and hardness testing. The tensile test specimens were prepared according to ISO 527-1 (ISO, 2019) and 527-2 (ISO, 2012) standards and the test was performed on a testometeric tensile test machine by using constant head speed of 50 mm/min. To evaluate the composite region's unique strength, the specimens' thickness was lowered to 6 mm. Average ultimate tensile strength, and percentage of elongation were measured from the load elongation data. The impact strength of the manufactured composites was assessed using the Charpy impact test.

Impact samples and tests were prepared and performed according to ISO 179 (ISO, 2010) and the Gunt Hamburg pendulum impact tester was used for testing the samples. The Durometer hardness tester type (shore D), which has a scale graduation (0 - 100) hardness number, was utilized for the hardness test. The test was carried out under ASTM D2240-15, (ASTM, 2015) and an average value was taken after measuring the values at five distinct points on the center of the composite material. For all mentioned tests and each processing condition, three samples were tested at temperature $(23 \pm 2 \, ^{\circ}\text{C})$ and an average of these three sample results was used for analyzing.

3.Results and Discussion

3.1-**Prepared** Composite Surface **Evaluation**

The surface of the produced polymer composites provides important information on the flow of the materials, distribution of the reinforcement, and the mechanical properties. All of these depend on the generation of heat. During the FSP process, heat is produced by friction between the tool and the matrix. Figure 4 displays Cu-reinforced samples as an example for the surface and macrostructure analysis which produced at different processing conditions. From Figure 4(a) It is clear that the composites formed with low tool rotational speed and high tool traverse speed (i.e. low heat input) did not distribute the reinforcement powders evenly over the FSP region. In such condition (low heat input), due to less stirring action and insufficient material flow, the treated surface becomes scratched and shallow. On the other hand, the composite qualities are not as good as intended because sufficient heat and time were not given for mixing the powders with the matrix. Figure 4(b) shows that by increasing the rotating speed and decreasing the traverse speed, excellent stirring action, particle distribution, and material flow are achieved. This is attributed to the proper quantity of heat and enough time provided for the particles to mix with the matrix (Rostamiyan and Zaferani, 2019). Because of this, the strength of produced composites and their surface characteristics are improved. Furthermore, although a uniform distribution of particles is observed, an excessive rotation speed weakens the composite material due to burning and producing defects on the surface of the products as seen in Figure 4(c), this decreases the strength of the produced composite.

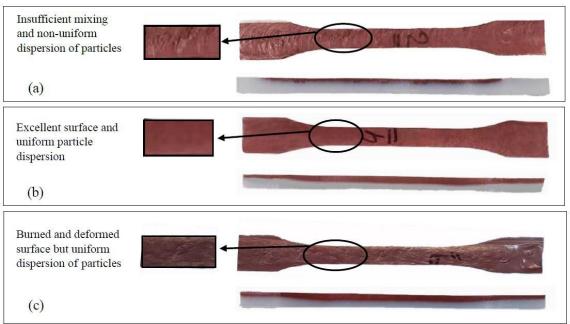


Figure 4. Composite Surfaces at (a) Low rotational speed, (b) Mediocre rotational speed, and (c) High rotational speed.

3.2- Analyzing Tensile Strength

Tensile test is a very important test in mechanical especially in the production of the composites in order to evaluate and to measure the ability of composite to withstand stress in tension. In this study, samples that have been produced were tested. However. The Taguchi approach highly recommends analyzing data by using signal-to-noise (S/N) ratio. S/N ratio, as defined by the Taguchi technique, is the ratio of using the greater is better category, and Table 3

"noise," which represents the unwanted value, such as the squared deviation of the output characteristics, to "signal," which represents the desirable value, such as the mean of the output characteristics. (Ahmadi et al., 2014). There are three status of performance characteristic in the analysis of the S/N ratio: smaller is better, larger is better, and nominal is better. The preferred quality of the composite is a higher tensile strength. Therefore, the S/N ratio was calculated

software.

where y is the observed data and n is the No. of

observations. The response of the S/N ratio for

each level of the FSP parameters was acquired

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presents the outcomes of the tensile strength and related S/N ratio for each reinforcement and experimental run. The following formula was used to determine the S/N ratio for the larger is better quality (Bozkurt, 2012).

 $S/N \ ratio = -10 \log \left(\frac{1}{n} \sum_{i=1}^{n} \frac{1}{yi^2}\right)...$ (3)

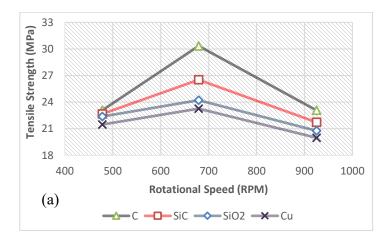
Table 3. displays the tensile strength and associated signal-to-noise ratio for every powder and experimental run.

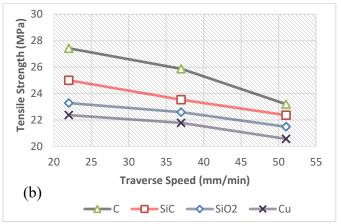
| Браус | 110 1011 | one eneriga | rana | Graphene (C) | | Silicon Carbide | | Silicon Dioxide | | Copper | |
|-------|----------|-------------|------|-------------------|-------|-------------------|-------|----------------------------|-------|-------------------|-------|
| Ехр. | ω | f | ٧ | Powder | | (SiC) Powder | | (SiO ₂) Powder | | (Cu) Powder | |
| No | (rpm) | (mm/min) | (%) | T-S | S/N | T-S | S/N | T-S | S/N | T-S | S/N |
| | | | | N/mm ² | ratio | N/mm ² | ratio | N/mm ² | ratio | N/mm ² | ratio |
| 1 | 478 | 22 | 10 | 23.78 | 27.52 | 23.16 | 27.29 | 22.89 | 27.19 | 22.10 | 26.89 |
| 2 | 478 | 37 | 15 | 23.59 | 27.45 | 22.81 | 27.16 | 22.45 | 27.02 | 21.79 | 26.76 |
| 3 | 478 | 51 | 20 | 21.87 | 26.80 | 22.02 | 26.86 | 21.78 | 26.76 | 20.53 | 26.25 |
| 4 | 679 | 22 | 15 | 34.16 | 30.67 | 29.32 | 29.34 | 25.87 | 28.26 | 24.79 | 27.89 |
| 5 | 679 | 37 | 20 | 31.21 | 29.89 | 26.23 | 28.38 | 24.54 | 27.80 | 23.50 | 27.42 |
| 6 | 679 | 51 | 10 | 25.66 | 28.19 | 24.02 | 27.61 | 22.25 | 26.95 | 21.52 | 26.66 |
| 7 | 925 | 22 | 20 | 24.34 | 27.73 | 22.54 | 27.06 | 21.09 | 26.48 | 20.23 | 26.12 |
| 8 | 925 | 37 | 10 | 22.82 | 27.17 | 21.59 | 26.69 | 20.78 | 26.35 | 20.05 | 26.04 |
| 9 | 925 | 51 | 15 | 22.07 | 26.88 | 21.06 | 26.47 | 20.44 | 26.21 | 19.66 | 25.87 |

According to the Taguchi method, the optimum level of FSP parameters is the level that represents the highest value of the signal-tonoise ratio. Based on Table 3, the rotational speed of 679 rpm, traverse speed of 22 mm/min, and volume fraction of 15% are the optimal set level of the FSP parameters in this research for all reinforcements, because the maximum S/N ratio and tensile strength were achieved at the mentioned levels. The influence of FSP parameters on the ultimate tensile strength is displayed in Figure 5. Low heat input and inadequate mixing result in poor mechanical properties of composites, as seen by the observed decrease in tensile strength at low rotational speeds (Figure 5a). But for graphene reinforcement, a small improvement occurred this may be due to the degree of compatibility between Graphene with the HDPE polymer. Increasing the rotational speed increases the heat input due to the friction and leads to better mixing and dispersion of particles resulting in producing good mechanical properties composites for all added powders. Whereas further increment causes the processed zone to burn because the higher amount of heat is concentrated on the stirring zone due to the polymers' restricted capacity to transfer heat and also ejects the molten polymer from the stir zone, which leads to degradation of the processed zone and declines the tensile strength (Hajideh et al., 2018) Figure 5b makes evident how mechanical qualities are changed when the traverse speed is increased. Low traverse speeds provide more time for stirring action, which increases the distribution of powder particles in the base material. It also permits the polymer to crystallize and results in a low cooling rate and slow crystal development. This could improve the spherulite structure, a polymeric composite with slower crystal formation shows improved mechanical properties (Azarsa and Mostafapour, 2013). However, at the higher traverse speeds, the aggregation of the added particles increased. due to the insufficient time for mixing and may produce some voids inside the composite and lead to a reduction in the composite quality. Furthermore, the type and quantity of particles added to the matrix influence

the composites' performance. Adding up to 15% reinforcing particles to the samples boosted their tensile strength (Figure 5c). It was also noticed that when the reinforcement level was increased from 15% to 20%, the composite's properties dropped. Particles may have clumped together and formed an agglomeration due to the high concentration of particles in the matrix and

this agglomeration weakens the surface adhesion between the filler particles and the polymer matrix and makes it a stress-concentrated region (Gao et al., 2015). At the optimum level set of parameters, ultimate tensile strength increased for all fabricated composites when compared to the base polymer samples.





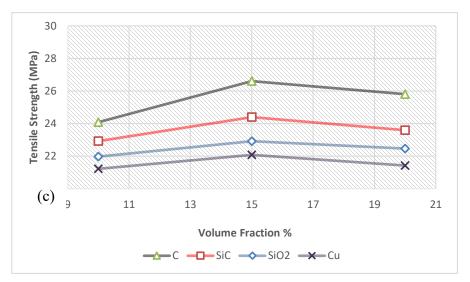


Figure 5. Main Effects Plot of Tensile Strength with FSP Parameters: (a) Rotational speed, (b) Traverse speed, and (c) Volume fraction

The decomposition of variances, also known as analysis of variance (ANOVA), gave a greater sense of the relative influence of the various FSP factors on the tensile strength (Eşme, 2009). ANOVA is used to show which friction stir process parameter has a substantial impact on the response. Additionally, ANOVA might be used to calculate the percentage contribution of

each parameter to the output performance. Results of the analysis of variance for the output response are displayed in Table 4. At a 95% confidence level, the F-test determines whether these estimates differ considerably. F values were used to verify that the process parameters are significant.

Table 4. Presents ANOVA Table for Tensile Strength

| Reinforcing Powder | Source | DF | Seq SS | MS | F | Р | Contribution (%) |
|-------------------------|------------------|----|---------|--------|-------|-------|------------------|
| | Rotational speed | 2 | 105.560 | 52.780 | 22.14 | 0.043 | 71.47 |
| Cranhana | Traverse speed | 2 | 27.424 | 13.712 | 5.75 | 0.148 | 18.57 |
| Graphene Powder | Volume fraction | 2 | 9.949 | 4.974 | 2.09 | 0.324 | 6.74 |
| Powder | Error | 2 | 4.768 | 2.384 | | | |
| | Total | 8 | 147.702 | | | | |
| | Rotational speed | 2 | 38.747 | 19.373 | 17.44 | 0.054 | 70.80 |
| | Traverse speed | 2 | 10.495 | 5.248 | 4.73 | 0.175 | 19.17 |
| SiC Powder | Volume fraction | 2 | 3.265 | 1.632 | 1.47 | 0.405 | 5.96 |
| | Error | 2 | 2.221 | 1.111 | | | |
| | Total | 8 | 54.727 | | | | |
| | Rotational speed | 2 | 17.8834 | 8.9417 | 13.86 | 0.067 | 70.33 |
| | Traverse speed | 2 | 4.9068 | 2.4534 | 3.80 | 0.208 | 19.30 |
| SiO ₂ Powder | Volume fraction | 2 | 1.3454 | 0.6727 | 1.04 | 0.489 | 5.29 |
| | Error | 2 | 1.2900 | 0.6450 | | | |
| | Total | 8 | 25.4254 | | | | |
| Cu Powder | Rotational speed | 2 | 16.3245 | 8.1623 | 23.07 | 0.042 | 70.02 |
| | Traverse speed | 2 | 5.0656 | 2.5328 | 7.16 | 0.123 | 21.73 |
| | Volume fraction | 2 | 1.2133 | 0.6066 | 1.71 | 0.368 | 5.20 |
| | Error | 2 | 0.7077 | 0.3539 | | | |
| | Total | 8 | 23.3112 | | | | |

A parameter's high F-value suggests that the parameter has a substantial influence on the product quality. The relative ability of each selected factor to lower variation is shown by its percentage contribution. A slight change in a parameter that contributes a large proportion will have a significant impact on the composite performance.

3.3- Analyzing of Elongation Percentages at Break

By examining the material's ductility, one can have a better understanding of the strain at the breakdown point. In many industrial applications that call for flexible materials, a material's capacity to deform plastically and adjust to the given load can be quite valuable. The pure HDPE sample which is used in this study has a ductility of 93%. However, after producing the

composites, a decrease in the elongation was observed in all samples when compared to the pure HDPE because reinforcing powders make the HDPE harder and this leads to a decrease in elongation. Figure 6a-c represents the influence of individual process parameters elongation of all the prepared composite samples. Figure 6a shows that increasing the rotational speed causes the composite's percent elongation to rise. Figure 6 (b-c) show the effect of traverse speed and volume fraction on elongation. It is seen that when traverse speed and volume fraction rise, the percent elongation decreases. The development of aggregate particles and the creation of structural cavities are connected to this deteriorating behavior, and the particle dispersion between the molecular chains which reduce the mobility of the chains and lead to reduced ductility (Khan et al., 2018).

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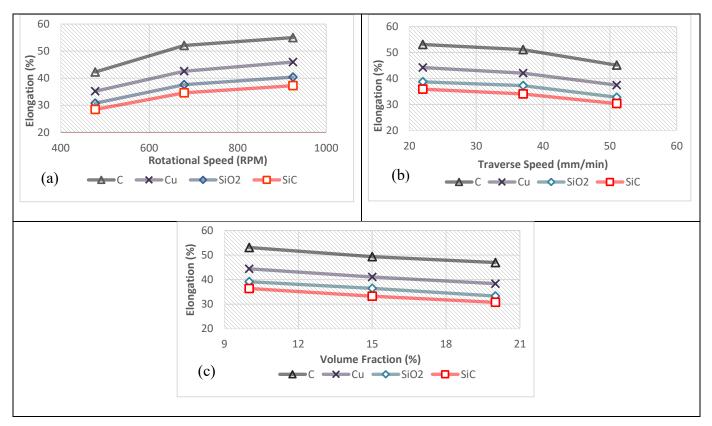


Figure 6. Main Effects Plot of Elongation Percentage with FSP Parameters: (a) Rotational speed, (b) Traverse speed, and (c) Volume fraction.

3.4 - Microhardness Behavior

Figure 7-a demonstrates how rotational speed affects microhardness levels. Since the low rotational speeds generate low heat which is insufficient for heating the base polymer and well distribution of powder particles in the base material it leads to a reduction in microhardness. Increasing the rotational speed up to 679 RPM results in a noticeable improvement in microhardness. The powder split apart during the FSP due to shear stresses exerted on the polymer macromolecules, which facilitates easier dispersion and penetrates powders into the polymer chains. Elevated rotating speeds cause more shear stresses on polymer chains, which in turn cause higher powder layer delamination in the HDPE matrix and ultimately raise the microhardness values. Further increment of rotational speed causes the stir zone to burn and throw out the molten polymer due to the centrifugal force produced during the FSP which generates defects in the processed zone and reduces the hardness of the processed zone. The role of traverse speed on the microhardness of the composites is shown in Figure 7-b. It is seen that the high microhardness value was obtained at the lower traverse speed. This could be attributed to the multiple reasons that might be in charge of this behavior. While it is true that stirring activity occurs in the stirring zone for a longer period of time at lower traversal speeds, this results in a better state of powder dispersion and microhardness of the fabricated composite. On the other hand, a low traversal speed permits the polymer to crystallize by causing a low cooling rate and slower crystal formation. Consequently, a polymeric composite with slower crystal growth exhibits enhanced microhardness (Barmouz et al., 2011). The relation between strengthening powders and the microhardness values is displayed in (Figure 7c). It is seen that the microhardness value increases by increasing the amount of particles. This could be attributed to a lot of amount of powders distributed in the same area of the produced composites.

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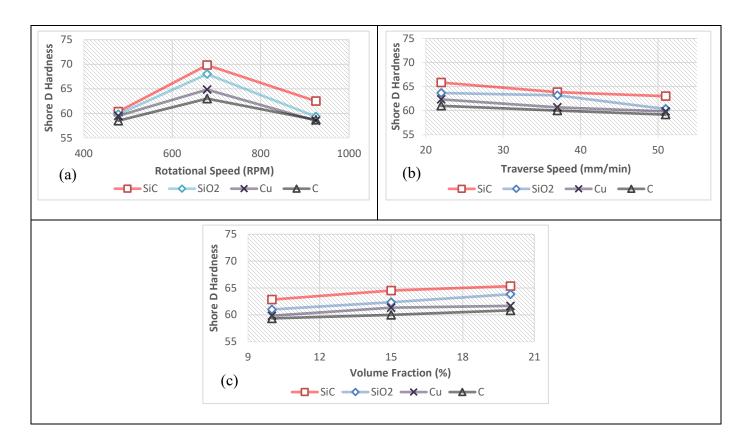


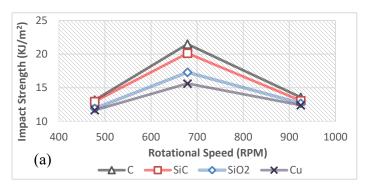
Figure 7. Main Effects Plot of Microhardness with FSP Parameters: (a) Rotational speed, (b) Traverse speed, and (c) Volume fraction.

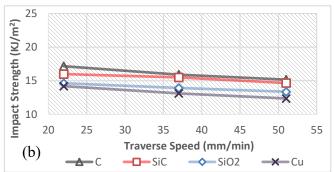
3.5- Impact Strength

Figure 8a represents the effect of rotational speed on the impact strength and observed that the highest value of the above-mentioned property has been obtained at the medium level of rotational speed for all kinds of added powders. Impact strength decreases at low rotational speeds because insufficient heat is generated to soften the matrix and combine it with the particles in the processed zone. While an extra high rotational speed (925 RPM) degrades the processed area and damages the mechanical properties that are noted the same outcome by (Abdel-Gwad et al., 2015). But

impact strength decreases with an increase in traverse speed as displayed in Figure 8b. This could be because the tool has less time to mix the ingredients efficiently because the traverse speed is responsible for giving sufficient time to the process of mixing. Lastly, the impact strength increases with increasing the amount of powder added to the matrix as shown in Figure 8c. This effect may be attributed to the reinforcing powders' adsorption into the polymeric chains, which alter the direction of can development, and the same outcome was confirmed by Derazkola and Simchi (Derazkola and Simchi, 2018).

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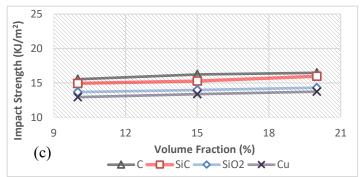


Figure 8. Main Effects Plot of impact strength with FSP Parameters: (a) Rotational speed, (b) Traverse speed, and (c) Volume fraction.

4.Confirmation Test

Confirmation tests for tensile strength were made for the Taguchi method at optimum and random levels. For the tensile strength, the rotational speed of 679 rpm, traverse speed of 22 mm/min, and volume fraction of 15% are the optimal set level of the FSP parameters. While for the random level, the rotational speed 679 r.p.m, traverse speed 37 mm/min, and volume fraction 10 % selected and three samples were

fabricated and tested. The test results and the predicted values derived from the Taguchi approach were compared in Table 5. The experimental values and predicted values are really near to one another. Error values must be less than 10% for statistical analysis to be considered reliable (Saurabh et al., 2022). Consequently, successful optimization is shown in the confirmation test results.

Table 5. Predicted Values and Confirmation Test Results for Tensile Strength.

| | | Tensile Strer | Error | | |
|-----------------------------------|------------------|---------------|--------------|------|--|
| Fabricated Composites | Parameter Levels | Predicted | Experimental | (%) | |
| HDPE / Graphene | Optimum | 33.38 | 34.16 | 2.28 | |
| composite | Random | 29.30 | 30.78 | 4.81 | |
| HDDE / SiC composite | Optimum | 28.65 | 29.32 | 2.29 | |
| HDPE / SiC composite | Random | 25.71 | 26.50 | 2.98 | |
| HDPE / SiO ₂ composite | Optimum | 25.51 | 25.87 | 1.39 | |
| | Random | 23.87 | 24.5 | 2.57 | |
| HDDE / Cu composito | Optimum | 24.58 | 24.79 | 0.85 | |
| HDPE / Cu composite | Random | 23.12 | 23.67 | 2.32 | |

5.Conclusion

In this study, polymer surface composites were effectively fabricated by combining the friction stir process with a newly developed tool system that includes a stationary shoe-shaped shoulder and a tool with a concave shoulder and grooved conical pin geometry. From the analysis of variance (ANOVA) table related to tensile strength shows that the contribution of rotational speed is between (70 - 72) % among the FSP parameters and followed by traverse speed (18.5 - 22) % and volume fractions of added powders (5 - 7) %. Therefore, rotational speed in FSP is a key factor that significantly influences the quality of the fabricated composites.

Among the percentage contribution of reinforced powders, noticed that the contribution of Graphene is higher than others in terms of tensile strength, this means that the graphene has more effect on the quality of the fabricated samples.

The optimum set level of parameters (rotational speed, traverse speed, and volume fraction) for ultimate tensile strength is 679 rpm, 22 mm/min, and 15%, respectively. However, the higher impact strength and microhardness are achieved at 679 rpm, 22 mm/min, and 20%.

Graphene-reinforced composites showed better results in measured properties except in terms of microhardness which has the minimum hardness when compared to the other composites. This may be attributed to the type of powder material, particle shape morphology, compatibility, and so on.

In comparison between reinforced composites, Graphene reinforced composites exhibit more elongation. This attributed to the flexibility and stretching ability of the Graphene.

The elongation of all fabricated composites decreased in comparison to the parent material. This is because the polymer chains' mobility was reduced by adding reinforcing particles.

According to the confirmation test results, the error between Taguchi predicted and experimental values of FSP parameters were smaller than 5 % this indicating that the measured values were within the 95% confidence interval.

Declaration of Competing Interest

The authors state that none of their personal ties or known conflicting financial interests might have appeared to have influenced the work described in this study.

Data availability

The study presented in the paper did not use any data.

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