Investigation on the effects of the processing parameters and the number of passes on the flexural properties of polymer nanocomposite fabricated through FSP method

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Abstract

Flexural properties calculation helps in designing structural elements like beam, cantilever and shafts. Moreover, the flexural properties are of vital importance in engineering and industrial applications such as joints replacements. The purpose of this investigation is to study for the first time, how the friction stir processing (FSP) parameters affects the flexural properties of UHMW-PE composites reinforced with nano particles. The tool rotational speed (\(\psi\)), tool feed rate (\(f\)), volume percentage (\(\nu\)) of nano powder and tool shoulder temperature (\(\tau\)) are selected as the process parameters. The ultimate flexural strength (UFS) and flexural yield strength (FYS) are calculated from the flexural test stress-strain diagrams. The analysis of variance is conducted which reveals that the selected parameters are significant for both UFS and FYS. Macroscopic and microscopic study shows that the FSP parameters affects the mixing of the strengthening particles and hence the flexural properties of the composite. The combinations of low level of \(\nu\) with medium level values of other parameters results in the highest flexural properties. Moreover, the combinations of higher levels of \(\tau\) and \(\psi\) results in material degradation. At the end, optimum conditions for the highest flexural properties are sorted out and the effect of increasing the number of passes has been investigated which significantly improve the flexural properties of the composite material.

1. Introduction

Polymer nanocomposites (PNCs) are a type of polymer composites in which reinforcing agents are nanoparticles. PNCs have tremendous applications in biomedical and fuel cells industries [1, 2]. Bio-polymeric composites like Poly L-lactic acid (PLLA) and hydroxyapatite (HA) are used for bone repair and regeneration applications [3]. Fuel cells employ polymer composites for matrix of bipolar plates, electrode binders and proton membrane exchangers. Field effect transistors photodiodes, conductors, super capacitors, light emitting diode sensors uses CNT reinforced polymer composites [4].

Composites based on Ultra-high molecular weight polyethylene (UHMW-PE) have extensive applications in industries because of its superior biocompatibility and high impact strength. UHMW-PE has been used in conveyor bands, cradles, cores of golf balls, ski and snow board surfaces, as noise reducing materials and in truck and dump truck bed liners. UHMW-PE has also found applications in automotive, aerospace and other industrial applications and the major use is found in the biomedical engineering applications, especially in joint
replacement and implants [5–7]. When UHMW-PE is reinforced with biocompatible and bioactive material such as nano-Hydroxyapatite (nHA), the fabricated composites offer improved properties with special characteristics such as anti-bacterial behavior and biocompatibility [8–10].

There are a number of methods employed for PNCs production such as melt mixing [11], solution blending [12], friction stir processing (FSP) [7] etc. Few of these methods have serious shortcomings such as solution blending technique is inapplicable for polymer materials which are insoluble in solvents [13]. On the other hand, melt mixing does not yield the same level of dispersion relatively and is uneconomical [14, 15]. FSP is a novel technique for the fabrication of PNCs. It is a novel variant of Friction Stir Welding (FSW), which is a solid state joining techniques first developed by The Welding Institute of Cambridge UK in 1991 for joining aluminum alloys. Later, this process was employed for welding of some other metals, polymers and composites [16, 17].

Polymers consist of chains of various lengths. Stirring may cause some chains to melt but other to be in solid form which will cause various defects [7]. Therefore, a special tool (Hot Shoe shaped shoulder tool) is being used nowadays to counter the defects production in FSW/FSP of polymers and polymer composites. FSP works on the same principal as FSW. In FSW a non-consumable rotating tool is plunged in the adjoining part of same or different material and then longitudinal motion is given to the rotating tool along the joint, in FSP a groove is made in a polymer material and the reinforcing component is added in the groove after that the tool is plunged in the groove and longitudinal motion is given to the rotating tool along the groove. Figure 1 shows the basic process of FSP in case of polymer matrix composites. FSP is more suitable than other methods because it is simple, cheap and flexible [18, 19].

Flexural strength is the maximum bending stress in the stress-strain diagram before the sample breaks. Flexural strength calculation helps in designing structural elements like beam, cantilever and shafts. It helps in selection of most suitable material for specific purpose. Flexural strength can also help engineers in evaluating the effects of various external loads on buried pipelines. Furthermore, UHMW-PE based composites are used in joint replacement and most of joints are under flexural loading. Therefore, higher flexural strength will results in less chance of joints fracture [20].

Based on the above discussion, the aim of this research was to study the effects of processing parameters on flexural properties of polymer nanocomposite fabricated through friction stir processing. The feed rate, tool speed, temperature of shoulder and volume fraction of nHA were chosen as processing parameters. The levels of these parameters were selected on the basis of literature review. Variation in these parameters resulted in variation of flexural properties of the composite which were investigated. Moreover, the conditions of the parameters for achieving the composite with best flexural properties were also investigated.

Figure 1. Step-wise procedure of FSP technique (a) Groove in UHMW-PE sheet, (b) nHA powder of specific volume fraction (ν) Compressed into Groove and (c) Processing at specific Rotational Speed ψ, Feed Rate f, Volume fraction ν and Shoulder Temperature τ.
2. Material and methodology

In this research the nano polymer matrix composite was formed from two materials; (1) nano Hydroxyapatite (nHA) powder (as strengthening material) and (2) Ultra High molecular Weight Polyethylene (UHMW-PE) (as matrix material). UHMW-PE was supplied by the Ningjin Hongbao Chemical Company Ltd China. These sheet have a melting temperature of (130–138) °C and tensile yield strength of 12.5 MPa. Properties of the as-received base material are given in table 1. The nHA powder was provided by Xian-Lyphar Biotech Company ltd China. The nHA powder was needle like structure with 96% purity and 60 nm average size. Table 2 shows the composition of the nHA.

Friction stir processing is a simple technique. A three axis vertical milling machine with a shoe shaped tool with closed loop temperature controller is utilized to fabricate the polymeric composite. Moreover, a specific fixture is also utilized which contains the mild steel base and two bars to support and grip the material firmly, acrylic sheet was placed in between polymer material and base to protect the tool from reaching the fixture base. Figure 2 shows the CAD schematic of fixture and actual fixture employed in the fabrication process.

The tooling system introduced by Strand [21] was modified. In this process, there is no need to cover the nano powdered material to prevent out flow of particles hence, reducing production time and cost. The thrust bearing was used due to which the shoe shaped tool is fixed compared to rotating pin. The shoulder of the tool is formed of 7075 Al and the tool pin is formed from steel. The threaded pin is basically used to enhance the thermal conductivity and better material blending [18]. Figure 3 shows the tool schematic and the actual tool. Mostly the sticking occurs between heated aluminum shoulder and polymer material so to prevent this phenomena, shoulder is coated with Polytetrafluoroethylene (PTFE). The basic objective of pin is to to mix the reinforced material with that polymer after producing adequate heat due to stirring effect. The shoulder of the tool has a basic role that it firmly presses the melted material until cooling. The closed loop temperature controller is also attached to maintain the temperature during the process. Figure 4 shows the vertical milling machine and figure 5 shows the temperature controller used in the fabrication process.

In this study, the tool rotational speed (\(\psi\)), tool feed rate (\(f\)), volume percentage (\(\nu\)) of nano powder and tool shoulder temperature (\(\tau\)) are selected as the process parameters. The levels of these parameters were selected based on the literature [14, 18 and 22] as well as from the preliminary experiments. It was observed that 20% nHA was not mixing with the polymer matrix in this FSP technique. Hence, 5, 10 and 15% were selected as levels of \(\nu\) based on the preliminary experiments. Similarly, in this FSP technique, shoulder temperature of 115 was resulting in material burning so 30, 65 and 100 were selected as levels of \(\tau\). Moreover, from the literature review and the facilities at hand the levels of feed rate and rotational speed were defined. 660, 1200 and 1700 rpm were selected as levels of \(\psi\), whereas, 30, 48 and 85 were selected as levels of \(f\).

Design Expert V11 package was practiced in developing the test plan. It provides options of employing different approaches for the formulation of test plan such as Full Factorial and Response Surface Method (RSM).

Table 1. Properties of as-recieved polymer sheets.

| Properties       | Units      | Value  |
|------------------|------------|--------|
| Density          | Kg m\(^{-3}\) | 0.958  |
| Tensile Strength | MPa        | 18.6   |
| Impact Strength  | KJ m\(^{-1}\) | 23     |
| Color            | —          | Blue   |
| Coefficient of friction \(\mu\) | —         | 0.1–0.15 |

Table 2. Composition of as-received nano Calcium Hydroxyapatite.

| Component                        | Percent composition |
|----------------------------------|---------------------|
| Phosphorous pentoxide(P\(_2\)O\(_5\)) | >39%                |
| Calcium oxide(CaO)               | >56%                |
| Mg                               | <0.6%               |
| Na                               | <0.15%              |
| Fe                               | <0.06%              |
| Al                               | <0.05%              |
| Loss on Drying                   | <1%                 |
| Sulphate, Chloride, Heavy Metals | <0.0999%            |
RSM requires less number of tests as compared to full factorial design and it also takes into account the combined effects as well as the individual effects of process parameters. Moreover, I-Optimal designs minimize the average variance of prediction over the region of experimentation [23]. Hence, this approach (I-Optimal design based RSM) was opted for the current investigation. The complete test plan as shown in table 3. The processing of the composite was conducted according to the formulated test plan.

After processing, the top and bottom faces of the processed sheet were face milled to 0.5 mm for the removal of the irregularities. Samples from the processed zone were taken out from the sheet according to the standard of testing for the flexural testing of the polymer. The samples were extracted with the help of a CNC milling machine. The cutting parameters of the end mill cutter practiced during the extraction of the samples includes 2000 rpm (spindle speed), 450 mm min\(^{-1}\) (feed rate) and 1 mm (depth of cut). A wooden fixture was used to clamp the friction stir processed (FSPed) UHMW-PE sheet for safety of the samples.

Flexural testing was carried out in order to study the performance of the composite material under bending conditions in accordance with ASTM-D790 standard. Flexural Tests were performed on Instron universal Testing Machine (30 KN) at cross head speed of 4 mm/min. The stress-strain data was logged and important results like flexural yield strength (FYS), ultimate flexural strength (UFS) were calculated from the flexural tests.

Figure 6 shows the samples extracted from the processed sheet for flexural testing and microscopic analysis. TESCAN scanning electron microscope was utilized to perform microscopic analysis of the samples. These samples were mounted using cylindrical die. The resin and hardener were mixed and poured into the die and left for 20–30 min for solidification. The samples were ground on 220, 320, 500, 1200, 2400, 4000 Abrasive grinding papers until the required grinding of the sample was completed. Coolant water was used during grinding and the samples were washed between each grinding step. Velvet cloth ws utilized for polishing the samples.

3. Results and discussion

3.1. Flexural testing results and stress-strain curves of composite and parent material

Table 4 shows the ultimate flexural strength (UFS), relative UFS, flexural yield strength (FYS) and relative FYS of the FSPed composite and parent material after testing on Instron universal Testing Machine (30 KN). It can be observed that experiment number 11 represent the highest values of FYS and UFS which shows that the medium condition of \(\psi, \tau, f\) and lowest value of \(\nu\) has the highest UFS as well as FYS.

Figure 7 shows the stress-strain graphs of selected processed composites and parent material. It is obvious from the graph that most of the sample behaved in the similar way as that of the parent material. Test 18 showed very low flexural properties (9.5% relative UFS and 10.6% relative FYS) whereas Test 11 showed the highest flexural properties (78.3% relative UFS and 110% relative FYS) amongst the FSPed composite.

3.2. ANNOVA results

Analysis of variance (ANNOVA) was performed to know which parameters were significantly affecting the flexural properties of the FSPed composite as show in table 5. We can notice that all the parameters are
significant parameters with 95% confidence level. The order of significance is same for UFS and FYS i.e. $(\psi > \tau > \nu > f)$.

3.3. Effects of process parameters on the Flexural properties of the composite

From figures 8(a) and 8(d) it is shown that FYS increases when $f$ decreases in all the cases but it negligibly changes at high $\psi$, low $\nu$ and high $\tau$. From figures 8(a) and 8(b) it can be observed that FYS increases with increase in $\psi$ in all cases but it decreases beyond 1200 rpm at low $f$ and at high $\tau$ combinations. From figure 8(c) it is shown that FYS decreases significantly with increase in $\nu$ but there is very negligible change in FYS with increase in $\nu$ at low $f$. As From figures 8(b) and 8(d) it can be observed that FYS decreases as $\tau$ decreases in all the combinations but FYS negligibly changes with increase in $\tau$ at high $\psi$.

From figures 9(a) and 9(b), it can be observed that UFS increases with increase in $\psi$ in all the cases but it decreases above 1200 rpm at low $\nu$ and at high $\tau$ combinations. Similarly, figures 9(b) and 9(d) shows that UFS increases with increase in $\tau$. However, increase in $\tau$ causes slight decrease in UFS at higher $\psi$ (greater than 1200 rpm). figures 9(c) and 9(d) shows that UFS increases as $f$ decreases. However, decrease in $f$ negligibly effects UFS at high $\tau$ and low $\nu$. The same trend is shown by $\nu$ whereas the negligible effects of $\nu$ are at low $f$ and high $\psi$ figures 9(a) and 9(c).
3.4. Empirical model and validation

The overall model equation of hyper surface which shows the output response of a selected processing parameter, the output response prediction can be done through this equation. The empirical modeling equation is developed through Design Expert Software (Statistical Software) for FYS and UFS.

The R² value for each model is above 96% which shows that data point follows the model curves. The output response of entire design space and processing parameters can be predicted from that model. Moreover, for additional validation the model output response was compared with experimental results. Table 6 shows the various set of processing parameters (other than the test plan) which shows that the experimental values are closer to the predicted model values. The predicted error ranges from 3.08% to 0.97%. This assure that the models are reasonably correct.

\[
\begin{align*}
\ln(\text{UFS}) &= (-4.89222 + 0.005178 \psi - 0.003106 f - 0.123939 \nu
\ + 0.051692 \tau + 0.000012 \psi f + 0.000089 \psi \nu - 0.000036 \psi \tau
\ - 0.002609 f \nu + 0.000362 f \tau + 0.000961 \nu \tau - 1.55E-06 \psi^2
\ - 0.000153 f^2 + 0.002802 \nu^2 - 0.000201 \tau^2)
\end{align*}
\]
$\text{Table 3. Experimental test plan.}$

| Run | $\psi$ (rpm) | $f$ (mm/min) | $\nu$ (%) | $\tau$ (°C) |
|-----|--------------|--------------|------------|-------------|
| 1   |  660         |  30          |  15        |  65         |
| 2   |  1700        |  30          |  5         |  30         |
| 3   |  660         |  48          |  5         |  65         |
| 4   |  1700        |  85          |  15        |  100        |
| 5   |  660         |  85          |  15        |  30         |
| 6   |  1700        |  30          |  10        |  100        |
| 7   |  660         |  48          |  15        |  100        |
| 8   |  1700        |  85          |  10        |  30         |
| 9   |  660         |  30          |  10        |  100        |
| 10  |  1700        |  48          |  15        |  30         |
| 11  |  1200        |  48          |  5         |  65         |
| 12  |  660         |  30          |  5         |  100        |
| 13  |  660         |  85          |  10        |  100        |
| 14  |  1700        |  85          |  5         |  100        |
| 15  |  1200        |  48          |  5         |  65         |
| 16  |  1700        |  48          |  10        |  65         |
| 17  |  660         |  85          |  5         |  30         |
| 18  |  660         |  48          |  10        |  30         |
| 19  |  1200        |  48          |  10        |  100        |
| 20  |  1700        |  85          |  15        |  100        |
| 21  |  1200        |  30          |  15        |  30         |
| 22  |  1700        |  48          |  10        |  65         |
| 23  |  1200        |  48          |  10        |  100        |

$\text{Ln}(\text{FYS}) = (-4.78776 + 0.005728 \psi - 0.01689 f - 0.197192 \nu + 0.044214 \tau + 0.000031 \psi f + 0.000044 \psi \nu - 0.000036 \psi \tau - 0.002418 f \nu + 0.000285 f \tau + 0.001246 \nu \tau - 2.00E - 06 \psi^2 - 0.000191 f^2 + 0.00697 \nu^2 - 0.000147 \tau^2)$

$\nu = \text{Volume Fraction of Nano Powder}$

$f = \text{Tool Feed Rate}$

$\psi = \text{Tool Rotational Speed}$

$\tau = \text{Shoulder Temperature}$

### 3.5. Microscopic and macroscopic analysis

Table 7 shows the SEM and optical images of the composite. It can be observed from the table that poor surface finish and defects (voids, agglomeration and cracks) are formed in FSP technique due to either low $\tau$, high $f$, low $\psi$, high $\nu$ or an improper combination of these conditions. Low $\psi$ means low rotational speed which will induce
low frictional heat and thus poor mixing of the reinforcing material due to low heat. Low $\tau$ means low shoulder temperature and hence lower tendency of the polymer material to be melted properly and hence resulting in poor mixing of the materials. Very low $\psi$ and $\tau$ will cause agglomeration of particles and poor mixing whereas, very high $\psi$ and $\tau$ will cause material burning and degradation. Low $f$ means more time for the matrix and reinforcing material to be mixed which result in excessive mixing of the materials. Very high $f$ will result in voids and channels. Low $\nu$ means lower reinforcing material to be mixed. Lower the reinforcing material, lower the tendency of the agglomeration.

Low $\psi$ with low $\tau$, high $\nu$ and high $f$ resulted in very low heat generation and ineffective mixing of the two materials and hence caused agglomeration, cracks and voids (Experiment No. 5, 17 and 18). The above mentioned experiments resulted in low flexural properties because of these defects. Whereas, high $\tau$ along with high $\psi$ and low $f$ resulted in excessive heat generation and mixing and hence caused material burning and
Table 5. ANOVA analysis.

| Source | Ultimate flexural strength | | Flexural yield strength |
|--------|----------------------------|------------------|-------------------------|
|        | p-value | Significant | Source | p-value | Significant |
| Model  | Quadratic<0.0001 | Y | Model | Quadratic<0.0003 | Y |
| $\psi$ | 0.0007 | Y | $\psi$ | 0.0009 | Y |
| $f$ | 0.0152 | Y | $f$ | 0.0767 | N |
| $\nu$ | 0.0118 | Y | $\nu$ | 0.0108 | Y |
| $\tau$ | 0.0003 | Y | $\tau$ | 0.0038 | Y |
| $\psi f$ | 0.0910 | N | $\psi f$ | 0.0034 | Y |
| $\psi \nu$ | 0.0268 | Y | $\psi \nu$ | 0.3143 | N |
| $\psi \tau$ | <0.0001 | Y | $\psi \tau$ | 0.0002 | Y |
| $f \nu$ | 0.0049 | Y | $f \nu$ | 0.0215 | Y |
| $f \tau$ | 0.0042 | Y | $f \tau$ | 0.0376 | Y |
| $\nu \tau$ | 0.0992 | N | $\nu \tau$ | 0.0892 | N |
| $\psi^2$ | 0.0277 | Y | $\psi^2$ | 0.0241 | Y |
| $f^2$ | 0.5068 | N | $f^2$ | 0.5067 | N |
| $\nu^2$ | 0.6158 | N | $\nu^2$ | 0.3294 | N |
| $\tau^2$ | 0.1563 | N | $\tau^2$ | 0.3887 | N |

Figure 8. Significant interactions for FYS.
degradation (Experiment No. 4). This degradation may affect the biocompatibility properties. Therefore, these combinations of process parameters should also be avoided in the production of UHMW-PE composite through FSP. Moreover, medium conditions of two or three parameters with low condition of the remaining parameters resulted in better surface finish, low or negligible cracks and voids and no degradation or material burning (Experiment No. 11 and 23). These combinations hence resulted in better flexural properties as compared to other combinations because of better mixing of the two materials and adequate heat generation.

### 3.6. Comparison of present study with previous studies

Table 8 shows the comparison between previous studies on flexural properties of polymers joined by FSW/FSP [22, 24] and this present study which shows that the present study agrees with the previous studies in defects formation. However, the present study doesn’t agree with the best results of previous studies. This is due to the

![Figure 9. Significant interactions for UFS.](image)

### Table 6. Comparison between the experimental and predicted flexural properties of test parameters combinations other than test plan combinations.

| S. No. | ψ  | f  | ν  | τ  | Ultimate flexural strength (MPa) | Flexural yield strength (MPa) |
|-------|----|----|----|----|-------------------------------|-------------------------------|
|       | Pred | Exp | Error (%) | Pred | Exp | Error (%) |
| 1     | 660 | 85 | 14 | 35 | 0.0177 | 0.018 | 1.80 |
| 2     | 660 | 85 | 10 | 30 | 0.0283 | 0.029 | 2.70 |
| 3     | 1200 | 48 | 7.5 | 45 | 0.5770 | 0.59 | 2.24 |

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Table 7. Macroscopic and microscopic analysis of FSPed composite.

| Exp. No. | Sample image | Comments |
|----------|--------------|----------|
| 18       | ![Image](image1.png) | **Defects:** Agglomeration, voids and poor surface finish.  
**Causes:** Low τ and low ψ. |
| Exp. No. | Sample image | Comments |
|---------|--------------|----------|
| 17      | ![Sample Image](image.jpg) | **Defects:** Large channel, peeling and poor surface finish.  
**Causes:** low $\tau$, high $f$ and low $\psi$. |
| Exp. No. | Sample image | Comments |
|---------|--------------|----------|
| 5       | ![Sample Image](image1.png) | **Defects:** Peeling, agglomeration and voids  
**Causes:** Low $\tau$, high $f$, low $\psi$, and high $\nu$. |
Table 7. (Continued.)

| Exp. No. | Sample image | Comments |
|----------|--------------|----------|
| 4        | ![Sample Image](image1.jpg) | **Defects:** Degradation and small voids.  
**Causes:** High $\tau$, high $f$, high $\psi$ and high $\nu$. |

![Material Degradation](image2.jpg)  
![Agglomeration](image3.jpg)  
![Voids](image4.jpg)
| Exp. No. | Sample image | Comments |
|---------|--------------|----------|
| 23      | ![Image](https://via.placeholder.com/150) | **Comment:** The boundaries of different regions cannot be observed which suggest relatively better surface finish and mixing.  
**Causes:** Better mixing and low defects at medium $\nu$, $f$ and $\psi$ with high $\tau$. |
| Exp. No. | Sample image | Comments |
|---------|--------------|----------|
| 11      | ![Sample Image](image.png) | **Comment:** Relatively better mixing of nHA powder in matrix and small amount of agglomeration.  
**Causes:** Medium $\tau$, $f$ and $\psi$ with low $\nu$. |
| Exp. No. | Sample image | Comments |
|----------|--------------|----------|
| 1        | ![Sample Image](image) | **Comment:** Voids and agglomeration  
**Causes:** High $\nu$ and low $\psi$ |

Table 7. (Continued.)
selection of different levels of process parameters and their ranges and the use of materials of different mechanical properties. Hence, it can be inferred that in FSP/FSW of polymer and polymer composites, there exist the material-parameter interaction and more studies needed to explore this aspect.

3.7. Optimum condition
From the above results it is cleared that peeling, material degradation and voids occurred in following set of conditions.

\[ \psi = (600 \& \text{, } 1700) \text{rpm}, \ \nu = 15\%, \ \tau = (30 \&, \ 100) \degree \text{C}, \ f = (30 \& \text{, } 85) \text{mm/min} \]

Therefore, these conditions has to be avoided for fabrication of UHMW-PE/nHA composite. The optimum set of process parameters were suggested by design expert software as shown in table 9. These conditions resulted in achieving 78.3% UFS of the base material and 11.1% increases in the FYS. Moreover, material degradation was not observed on these conditions. So on these condition fabrications can be done.

3.8. Effects of increasing the number of passes on flexural properties of composite materials
It can be observed from table 4 that all the experiments showed less UFS as compared to the base material. This might be due to various defects on micro level such as micro voids, channels and agglomeration of reinforcing particles. Even in the optimum combination of parameters, micro voids and agglomeration has been observed (table 7, Experiment 11). Therefore, further investigations required to improve UFS of the composite material. Hence, the effects of increasing the number of passes has been performed. Increasing the number of passes means that the already processed area is processed again but keeping the processing parameters constant. The parameters suggested by the optimum condition section \((\psi = 1200 \text{ rpm}, \ \nu = 5\%, \ \tau = 70 \degree \text{C} \& \ f = 48 \text{ mm/min})\) were selected but the number of passes has been changed from 1 to 2 and 3. Table 10 shows that increasing the number of passes increases the flexural properties significantly. Due to increase in the number of passes, the reinforcing particles mixes with the matrix again and again. This might have resulted in
better mixing and distribution of reinforcing particles which resulted in better flexural properties. Therefore, increasing the number passes is recommended in the fabrication of polymer matrix composites through FSP technique.

4. Conclusions

In this investigation, processing of UHMW-PE/nHA polymer nanocomposite was successfully performed through friction stir processing (FSP). The effects of processing parameters (tool rotational speed ($\psi$), volume percentage ($\nu$) of nHA particles, tool feed rate ($f$), and tool shoulder temperature ($\tau$)) on ultimate flexural strength (UFS) and flexural yield strength (FYS) was investigated. It was observed that the experiments with combinations of low level of $\nu$ with medium levels of other parameters resulted in the highest flexural properties. Similarly, the experiments with combinations of higher levels of $\tau$ and $\psi$ resulted in material degradation. To further examine and verify the results scientifically, the microstructural and macro structural study was conducted which revealed that formation of various defects (agglomeration, cracks and voids) caused low flexural properties in experiments at low $\tau$, high $f$, low $\psi$, high $\nu$ or an improper combination of these conditions. Whereas, high $\psi$ along with low $f$ and high $\tau$ resulted in material burning and degradation which may affect the biocompatibility properties hence these conditions should also be avoided. Moreover, medium conditions of two or three parameters with low condition of the remaining parameters resulted in better surface finish, low defects and hence resulted in better flexural properties as compared to other combinations. ANOVA analysis confirmed that all the selected parameters were significant by 95% in case of UFS and FYS. Moreover, the optimum conditions for increased flexural properties were sorted out which resulted in less agglomeration and degradation free composite with enhanced flexural properties. In the end, the effect of increasing the number of passes has been investigated which suggests that the flexural properties has been significantly improved with it.

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Conflicts of interest

The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, or in the decision to publish the results.

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References

[1] Tanahashi M 2010 Development of fabrication methods of filler/polymer nanocomposites: with focus on simple melt-compounding-based approach without surface modification of nano fillers Materials 3 1593–619
[2] Paul D R and Robeson L M 2008 Polymer nanotechnology: nanocomposites Polymer 49 3187–204
[3] Kim H W, Kim H E and Salih V 2005 Stimulation of osteoblast responses to biomimetic nanocomposites of gelatin–hydroxyapatite for tissue engineering scaffolds Biomaterials 26 5221–30.
[4] Baibarac M and Gómez-Romero P 2006 Nanocomposites based on conducting polymers and carbon nanotubes: from fancy materials to functional applications J. Nanosci. Nanotechnol. 6 289–302
[5] Stein H L 1988 Ultra high molecular weight polyethylene (UHMWPE). Engineering Materials Handbook. 2 167–71
[6] Vallet-Regi M 2014 Bio-Ceramics with Clinical Applications (West Sussex, PO19 8SQ, United Kingdom : Wiley) pp 1–22
[7] Khan I, Hussain G, Al-Ghamdi K A and Umer R 2019 Investigation of impact strength and hardness of UHMW polyethylene composites reinforced with nano-hydroxyapatite particles fabricated by friction stir processing Polymers. 11 1041
[8] Gupta S and Riyad M F 2018 Synthesis and tribological behavior of novel UHMWPE-Ti3 SiC2 composites Polym. Compos. 39 254–62
[9] Kurtz S M 2004 The Uhmwpe Handbook Ultra high molecular weight polyethylene in total joint replacement (California, United States: Elsevier academic press) pp 1–305
10. Huang H Y, Liu Z H and Feng T 1997 O-6-85 - In vivo evaluation of porous hydroxyapatite ceramic as cervical vertebra substitute. *Clinical Neurology and Neurosurgery*. 99 S20–1

11. Andrews R, Jacques D, Minot M and Rantell T 2002 Fabrication of carbon multiwall nanotube/polymer composites by shear mixing. *Macromol. Mater. Eng.* 287 395–403

12. Jin L, Bower C and Zhou O 1998 Alignment of carbon nanotubes in a polymer matrix by mechanical stretching. *Appl. Phys. Lett.* 73 1197–9

13. Beyou E, Akbar S, Chaumont P and Cassagnau P 2013 Polymer nanocomposites containing functionalised multiwalled carbon nanotubes: a particular attention to polyolefin based materials. *Syntheses and Applications of Carbon Nanotubes and Their Composites* 1 77–115

14. Battisti M G and Friesenbichler W 2013 Injection-moulding compounding of PP polymer nanocomposites. *Strojniški Vestnik-Journal of Mechanical Engineering*. 59 662–8

15. Kim H, Miura Y and Macosko C W 2010 Graphene/polyurethane nanocomposites for improved gas barrier and electrical conductivity. *Chem. Mater.* 22 3441–50

16. Wei H, Tariq M, Hussain G, Khan I, Imran Khan M and Khan W A 2019 Butt joining of Bi-layered aluminum sheets through friction stir welding: tensile stresses, bending stresses, residual stresses, and fractography. *Metals*. 9 3441–50

17. Tariq M, Khan I, Hussain G and Farooq U 2019 Microstructure and micro-hardness analysis of friction stir welded bi-layered laminated aluminum sheets. *International Journal of Lightweight Materials and Manufacture*. 2 123–30

18. Khan I, Hussain G, Tariq M and Ilyas M 2018 Fabrication of UHMW polyethylene/nano-hydroxyapatite biocomposite via heat-assisted friction stir processing. *The Int. J. Adv. Manuf. Technol.* 96 3651–63

19. Hussain G and Khan I 2018 Characteristics of friction stir processed UHMW polyethylene based composite. *IOP Conference Series: Materials Science and Engineering*. 301 (Xiamen, China) (https://doi.org/10.1088/1757-899X/301/1/012109)

20. Bhardwaj A, Gupta A and Tse K M 2014 Mechanical response of femur bone to bending load using finite element method. *2014 Recent Advances in Engineering and Computational Sciences (RAECS) 2014 Mar 6 (Chandigarh, India)* pp 1–4

21. Strand S 2003 Joining plastics-can friction stir welding compete? *Proc.: Electrical Insulation Conf. and Electrical Manufacturing and Coil Winding Technology Conf.* (Cat. No. 03CH37480) 2003 Sep 25 (Indiana, USA) pp. 321–6

22. Azarsa E and Mostafapour A 2014 Experimental investigation on flexural behavior of friction stir welded high density polyethylene sheets. *Int. J. Manuf. Processes*. 16 149–55

23. Anderson M J and Whitcomb P J 2016 RSM simplified: optimizing processes using response surface methods for design of experiments 2 (Florida, United States: CRC Press Taylor & Francis Group) pp 1–297

24. Mostafapour A and Azarsa E 2012 A study on the role of processing parameters in joining polyethylene sheets via heat assisted friction stir welding: investigating microstructure, tensile and flexural properties. *International Journal of Physical Sciences*. 7 647–54