

# Investigation on Varying Weight Percentage of Nickel Nanoparticles on the Thermal Properties of Polymeric Nanocomposites

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## Abstract

Conducting composites are the most efficient and commonly used materials for electromagnetic interference (EMI) shielding. The coefficient of thermal expansion (CTE) of a nanocomposite plays a very important role in the use and manufacturing of conducting polymers. However, CTE significantly affects with type and weight percentage (wt.%) of fillers in composites. Nickel (Ni) having good conductivity and permeability is commonly preferred as reinforced material in conducting composite. This work investigates varying wt.% of Ni nanoparticles on the thermal properties of polymeric nanocomposites. The filler content was varied from 0 to 60 wt.%. Ni nanoparticles of 40 nm was used as filler material. This study found an increase in the char yield of the nanocomposites with an increase in Ni content in the poly(ether ketone) (PEK) matrix. A degradation temperature (i.e., 10 % weight loss, T10) was found to increase by 19 °C with increasing Ni content in the PEK/Ni nanocomposite. NC-60 (wt.% of Ni is 60%) has shown higher thermal stability. Above 170 °C there was an increase in the thermal strain of the nanocomposites significantly, but it is observed that it is lowered by the addition of Ni 50 - 60 wt.% as filler material.

## Keywords

Nanocomposite, Nanoparticles, Poly(ether ketone), Nickel

## Introduction

The CTE of a nanocomposite plays a very important role in use and manufacturing of conducting polymers. It has been found that CTE decreases with increasing filler content in composites [1]. It is reported that CTE is prominently influenced by the type of fillers in the neat epoxy. The pristine-single-walled nanotubes (SWNT)-epoxy samples' CTE values were discovered to be lower than those of the neat resin, however the difference was minor due to uneven dispersion and weak interfacial bonding between the pristine-SWNT and the epoxy. The CTEs of the functionalized SWNTs/epoxy nanocomposites, however, were significantly reduced, and the reduction was based on the specific functionalization techniques. The chopped-SWNT-epoxy and oxidized-SWNT-epoxy nanocomposites demonstrated a remarkable reduction in CTE. Additionally, a 14% CTE reduction was seen in the wrapped SWNT-epoxy compared to the tidy matrix [2].

In composites made of polyphenylene sulfide and copper, it was discovered that the CTE decreased as the matrix's copper concentration rose [3]. Similarly, the CTE of the PEEK/Al<sub>2</sub>O<sub>3</sub> composite prominently decreased compared to neat PEEK for the 43 volumes% micro-Al<sub>2</sub>O<sub>3</sub> composite and the 12 volumes% nano-Al<sub>2</sub>O<sub>3</sub> composites [4]. It was found that the linear CTE of the PTFE or

any other polymer is higher above the glass transition temperature than that of below glass transition temperature. It is because above glass transition temperature there is free rotation of each bond in the main chain in amorphous region while smaller scale motion in main chain in amorphous regions below the glass transition temperature [5]. The addition of hard fillers reduced the segmental movement of the polymer chains that lowered thermal expansion in ceramic filled polymer matrix composites [6]. Table 1 depicts the thermal, mechanical, and electrical properties of the reinforcements.

Conducting composites are the most efficient and commonly used materials for EMI shielding. It is well known that metals having good conductivity has low permeability, and materials with good permeability has low conductivity. For the material used as reinforced material in conducting composite, used in EMI shielding, should possess both properties of conductivity and permeability. Ni metal has both properties together, and that's why Ni is used as reinforcement into the PEK matrix.

The PEK has face centered cubic crystal structure, so it is very malleable and ductile in nature. A thermoplastic high-performance polymer is called PEK. The polymer is semicrystalline. Its melting point and glass transition are roughly 360 °C and 160 °C, respectively. Additionally, it has a very low tendency to creep and strong fatigue strength as well as good impact strength. It displays a Young's modulus of between 100 and 110 MPa. It is also chemically resistant [9-11]. It can be easily processed using conventional technique (i.e., injection or extrusion) in required shape easily, and the insulation can overcome by reinforcement of conductive particles or fibers. Despite good thermal, mechanical, wear, electrical and chemical properties, much attention was not paid on the PEK composites particularly filled with Ni particles.

Ni has good oxidation and corrosion resistance so can be used effectively in different applications. It has linear CTE of  $13.4 \times 10^{-6}/^{\circ}\text{C}$  and thermal conductivity of about 90.9 W/(m.K). All these properties of Ni will match with the requirement for reinforced materials used for EMI shielding materials. The Ni exhibits high absorption loss which gives good pro-

tection and can be used as reinforcement for the EMI shielded material.

Looking into the constraint of the materials, there is a necessity of developing a new electrically conducting high performance polymer matrix composite which have good flexibility, good electrical conductivity, high glass transition temperature and EMI-SE more than 20 dB.

A study on composites made of PEK and Ni, however, was not mentioned. This is true even though PEK has superior thermal, electrical, and mechanical capabilities when compared to common polymers. PEK is a high-performance low density semicrystalline thermoplastic polymer with outstanding chemical, thermal, mechanical, wear resistance properties. The thermal characteristics of polymeric nanocomposites with various Ni wt.% are included in the current study.

## Experimentation

### Methodologies and techniques

The fabrication of PEK/Ni nanocomposites with Ni powders (nano- and micro-sized particles) as fillers was used to fulfil the specified objectives. The filler content ranged from 0 to 60% of the total weight. Ni nanoparticles with an average size of 40 nm was used as a conductive reinforcement material without any surface treatment.

### Synthesis of nanocomposites

The nanoparticles of PEK and Ni were mixed, and then they were ground in a planetary ball mill. A planetary ball mill is shown in figure 1. Several compositions of PEK/Ni (micro and nano) with various Ni contents-varying from 0 to 60 wt.% were milled at ambient temperature.

Large surface-to-volume ratios of nanoparticles make them superior reinforcements to conventional fillers. The strong connections between nanoparticles make it challenging to disperse them into the polymer matrix, though. The interparticle interaction makes it extremely challenging to disperse nanoparticles uniformly, especially at higher particle loadings. In other words, using smaller particles at higher loadings caus-

**Table 1:** Properties of the reinforcements [3, 5, 7, 8].

Properties	Ag	Cu	Ni	CN	Al	Steel	SS	CF
<b>Thermal properties</b>								
Density (g/cc)	11	8.96	8.9	1.3 - 1.4	2.7	7.85	7.48	1.78
CTE ( $\times 10^{-6}/^{\circ}\text{C}$ )	18	17	13	-	24	13	10.1 - 17.3	2
Thermal conductivity (W/m.K)	429	401	91	205	205	43	16	21 - 180
<b>Electrical properties</b>								
Dielectric constant	-	6 - 6.2	7.3 - 11.8	-	1.6 - 1.8	4.5	-	-
Electrical conductivity ( $\times 10^5$ S/cm)	6.8	6.4	0.97	0.1 - 10	4	0.6	0.18	High
<b>Mechanical properties</b>								
BHN (MPa)	245	80 - 85	~ 195	-	100	120	200	120
Young's modulus (GPa)	83	110 - 128	200	270 - 950	70	200 - 215	189 - 210	200 - 240



Figure 1: Planetary ball milling.

es agglomeration, which affects the composites' thermal, mechanical, and electrical properties.

There are numerous techniques for incorporating reinforcements into polymer matrices, including mechanical alloying, melt mixing, *in-situ* polymerization, solution approach, and ball milling [7, 12, 13]. Better reinforcement dispersion is necessary since their agglomerates reduce the performance of the material by including voids that serve as favoured sites for crack initiation and failure. Due to the predominate intermolecular van der Waals interactions between them, nanoparticles (size 100 nm), as previously mentioned, tend to aggregate, or cluster [12]. Mechanical alloying, often known as the ball milling technique, has drawn a lot of interest in recent years as a potent tool for the creation of nanocomposites [12].

In ball milling, the degree of dispersion of nanoparticles in the matrix depends upon the milling time, balls to powder ratio, speed and mixing atmosphere. During milling process, fragments and cold re-welding of powder particles takes place due to continuously trapping between colliding balls and container surface what raises the level of microstructural strain. This leads to increase of hardness of polymer/reinforcements and a decrease of % crystallinity of polymer fraction [10]. According to Shakir et al. [14] ball mill method yields significant enhancement in EMI shielding due to proper dispersion of reinforcements in the matrix. Solution method can also be used for dispersing nanoparticles in the matrix but volatiles solvent in the environment is not good for health (Table 2) [15].

The addition of reinforcement into the matrix increased density of the composites, i.e., the density of the PEEK/ $\text{Al}_2\text{O}_3$  [16] and PVDF/Ni composites [16] increased due to higher density of  $\text{Al}_2\text{O}_3$  ( $4 \text{ g/cm}^3$ ) and Ni ( $8.9 \text{ g/cm}^3$ ) as compared

Table 2: Milling process parameters.

Ball diameter (mm)	5
Number of balls	34
Vial volume (ml)	150
Ball to powder weight ratio	10:01
Rotation speed of vial (rpm)	250
Ball and vial material	Zirconia
Atmosphere	Air
Temperature	Room temperature

to neat PEEK ( $1.3 \text{ g/cm}^3$ ) [15, 16]. Similarly, the density of the PEEK/ $\text{Si}_3\text{N}_4$  [8] and PPS/ $\text{AlN}$  [15] composites increased almost linearly with increasing reinforcements.

## Results and Discussion

Thermal properties involve material-dependent responses to heat, such as temperature increase, length change, or volume change. The CTE measures object size change with temperature, with lower coefficients indicating lower size change. A thermogravimetric analyzer (TGA) was used to investigate the composites' thermal stability. The samples were heated to  $900 \text{ }^\circ\text{C}$  from room temperature. In a nitrogen atmosphere, composite samples were heated at a rate of  $10 \text{ }^\circ\text{C/min}$ . The degradation temperature corresponded to a 10% loss in weight (T10). The maximum decomposition temperature determines the temperature corresponding to the peak obtained by the first-order derivative curve (DTG) ( $T_{\text{max}}$ ). The char yield is the residue that remains at  $850 \text{ }^\circ\text{C}$  in a nitrogen atmosphere.

A dilatometer was used to measure the linear CTE or dimensional stability of the composites as they were heated from  $30$  to  $250 \text{ }^\circ\text{C}$  (Figure 2).  $5 \text{ }^\circ\text{C/min}$  of heat was applied to the samples. Over specific temperature ranges of  $50 - 100 \text{ }^\circ\text{C}$  (below the glass transition temperature,  $T_g$ ) and  $180 - 230 \text{ }^\circ\text{C}$ , the average linear CTE was calculated (i.e., above  $T_g$ ).

### Thermal stability

The thermal stability and CTE of a micro/nanocomposite are important considerations in the use and production of conducting polymers. Figure 3 shows the thermal stability of the nanocomposites as a function of temperature. It was discovered that pure PEK degraded at  $567 \text{ }^\circ\text{C}$ , or 10% weight loss (T10). As T10 increased by  $19 \text{ }^\circ\text{C}$  with increasing Ni content, the thermal stability of the nanocomposites increased up to NC-60. This suggests that the addition of Ni to the nanocomposites enhances their ability to withstand higher temperatures without significant degradation. However, further increases in Ni content beyond NC-60 may not result in a significant improvement in thermal stability, indicating a potential saturation point for the effect of Ni on the nanocomposites' thermal stability.

The nanocomposites' DTG traces are displayed in figure 4. The derivative increases significantly when significant weight loss starts, and it continues to rise until the rate of weight loss



Figure 2: Dilatometer for measuring linear CTE.

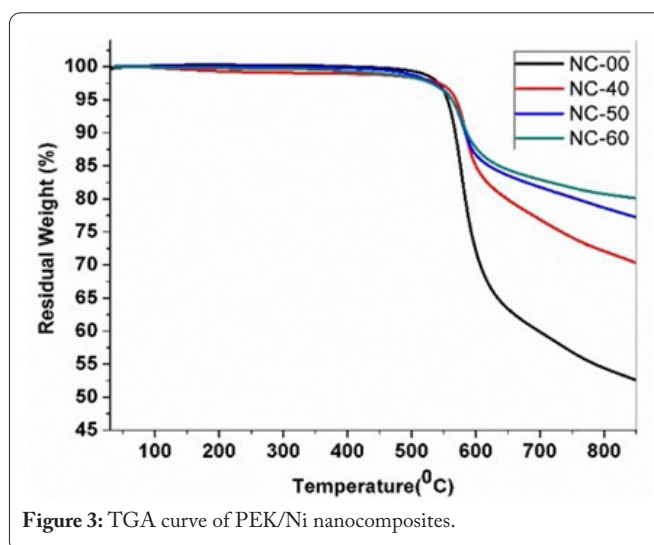


Figure 3: TGA curve of PEK/Ni nanocomposites.

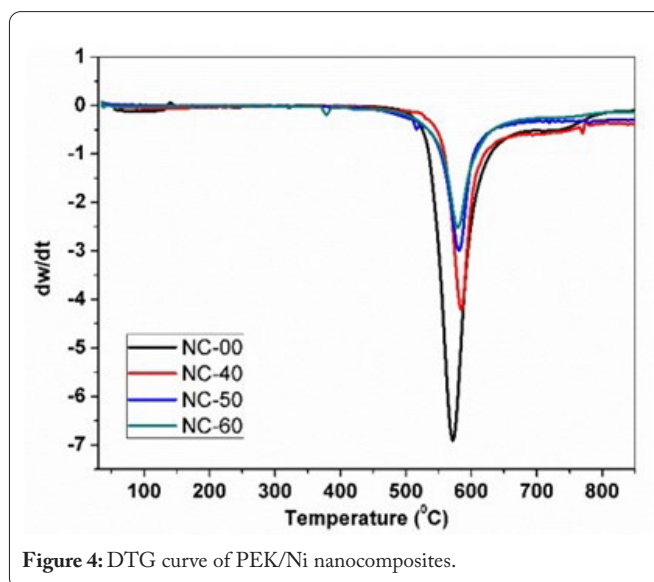


Figure 4: DTG curve of PEK/Ni nanocomposites.

reaches its maximum. It demonstrates that for NC-40 nanocomposites, the maximum decomposition temperature rises to 13 °C and then falls to 4 °C for NC-50 nanocomposites with additional Ni addition.

This trend suggests that the addition of Ni up to a certain concentration enhances the thermal stability of the nanocomposites, but beyond that point, it starts to have a detrimental

effect. The observed decrease in maximum decomposition temperature for NC-50 could be attributed to an excessive amount of Ni, which may lead to the formation of less stable chemical bonds within the nanocomposite structure.

According to table 3, the nanocomposites' char yield slightly increases as the Ni content of the PEK matrix rises. The main molecular chains may break down, causing polymer thermal decomposition, which could explain the weight loss. On the one hand, the connected network makes moving molecular chains and volumetric products more challenging. Because the surface energy of the Ni particles is higher at higher Ni concentrations, aggregates develop. As a result, Ni and the matrix's effective interfacial area are smaller.

This reduction in interfacial area hinders the transfer of stress between the Ni particles and the matrix, leading to a decrease in mechanical properties. Additionally, the formation of aggregates can also result in the formation of voids and defects within the composite material, further contributing to its weight loss.

### CTE

For the estimation of dimensional stability, the change in length (parallel to pressing direction or out-of-plane direction) with increasing temperature from 50 °C to 250 °C has been measured during a second heating cycle and is shown in figure 5. The out-of-plane linear CTE was estimated over the linear curves, or between 50 °C and 100 °C (below T<sub>g</sub>) and 180 °C and 230 °C (above T<sub>g</sub>). Figure 5 demonstrates that the thermal strain or change in length relative to the original length of the nanocomposites, dramatically increased above 170 °C (also known as T<sub>g</sub>). This is due to the availability of sufficient thermal energy for molecular movements, and as a result, free volume rises significantly with temperature, causing more thermal strain than at lower temperatures.

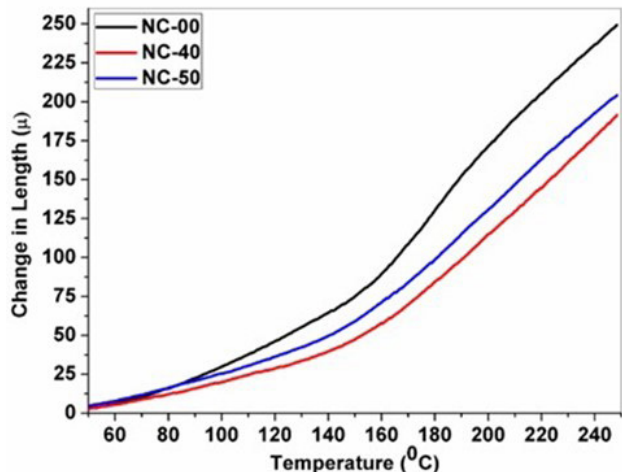
This increase in thermal strain can be attributed to the increased mobility of polymer chains and nanoparticles at higher temperatures. Additionally, the expansion of the nanocomposites is also influenced by the expansion coefficient of the materials used in their composition.

In other words, due to relaxation effects during measurement, the as-molded sample increases nonlinearly between 120 °C and 180 °C (about T<sub>g</sub>) and linearly between 50 °C and 100 °C (below T<sub>g</sub>) and 180 °C and 230 °C (above T<sub>g</sub>) [8]. The addition of 50 - 60 wt.% of Ni nanoparticles to the PEK matrix lowers the thermal strain of the nanocomposites because Ni nanoparticles inhibit the mobility of polymer chains with temperature. The overall expansion for pure PEK from 50 to 250 °C is decreased for NC-60 nanocomposites from 1.3% to around 1.13%, demonstrating a considerable increase in dimensional stability.

This increase in dimensional stability is attributed to the presence of Ni nanoparticles, which restrict the movement of polymer chains at elevated temperatures. This enhanced thermal strain resistance makes NC-60 nanocomposites a promising material for applications requiring dimensional stability over a wide temperature range (Table 4).

**Table 3:** Thermal properties of PEK/Ni nanocomposites from TGA.

Sample code	T <sub>max</sub>	T <sub>10</sub>	Char yield at 850 °C
NC-00	571	567	52.5
NC-40	584	584	70.3
NC-50	516	584	77.2
NC-60	579	586	80

**Figure 5:** Change in length with respect to temperature of the PEK/Ni nanocomposites.**Table 4:** The CTE of the PEK/Ni nanocomposites.

Sample code	CTE below T <sub>g</sub> (x 10 <sup>-6</sup> )	CTE above T <sub>g</sub> (x 10 <sup>-6</sup> )
NC-00	58	133.8
NC-50	48	124.5
NC-60	45	123

In contrast to the NC-60 nanocomposite, the linear CTE of pure PEK is only about 45 x 10<sup>-6</sup>/°C below T<sub>g</sub> (50 - 100 °C), as shown in table 4. Additionally, it was discovered that the CTEs of the nanocomposites evaluated above T<sub>g</sub> (180 - 230 °C) were significantly lower than those of pure matrix. Due to the lower intrinsic linear CTE of the Ni compared to pristine PEK and the lower volume fraction of PEK in the composites, the CTEs of the nanocomposites were reduced. This indicates that the addition of NC-60 nanocomposite not only reduces the linear CTE of pure PEK below T<sub>g</sub>, but also improves its thermal stability above T<sub>g</sub>. The lower intrinsic linear CTE of the Ni and the lower volume fraction of PEK in the nanocomposites contribute to this decrease in CTE.

## Conclusions

The thermal properties, such as the thermal stability and the CTE of a nanocomposite play a very important role in the use and manufacturing of conducting polymers. However, they are prominently influenced by the type and wt.% of fillers in composites. Ni having good conductivity and permeability is commonly preferred as reinforced material in

conducting composite. This work investigated varying wt.% of Ni nanoparticles on the thermal properties of polymeric nanocomposites. The filler content was varied from 0 to 60 wt.%. Micrometre-sized PEK (1100PF grade) powder and Ni nanopowder with an average size of 40 nm was used. The following conclusions could be drawn from the present study.

- The PEK matrix and Ni nanoparticles found strong interfacial adhesion. As the Ni content of the PEK matrix grew, the char yield of the nanocomposites was increased.
- The degradation temperature of pure PEK was determined to be 567 °C (i.e., 10% weight loss, T<sub>10</sub>), and it increased by 19 °C with increasing Ni content in the PEK/Ni nanocomposite. More thermal stability was found for NC-60 (60 percent Ni by weight).
- The addition of 50 - 60 wt.% of Ni nanoparticles to the PEK matrix reduced the thermal strain and the total expansion showing a notable improvement in dimensional stability of the nanocomposites. This enhanced thermal strain resistance makes NC-60 nanocomposites a promising material for applications requiring dimensional stability over a wide temperature range.
- The addition of Ni to the nanocomposites enhances their ability to withstand higher temperatures without significant degradation. However, further increases in Ni content beyond NC-60 may not result in a significant improvement in thermal stability, indicating a potential saturation point for the effect of Ni on the nanocomposites' thermal stability.

## Acknowledgments

None.

## Conflict of Interest

None.

## References

1. Goyal RK, Jadhav P, Tiwari AN. 2011. Preparation and properties of new polyphenylene sulfide/AlN composites for electronic packaging. *J Electron Mater* 40: 1377-1383. <https://doi.org/10.1007/s11664-011-1520-5>
2. Wang S, Liang Z, Gonnet P, Liao YH, Wang B, et al. 2007. Effect of nanotube functionalization on the coefficient of thermal expansion of nanocomposites. *Adv Funct Mater* 17(1): 87-92. <https://doi.org/10.1002/adfm.200600760>
3. Goyal RK, Kambale KR, Nene SS, Selukar BS, Arbu S, et al. 2011. Fabrication, thermal and electrical properties of polyphenylene sulfide/copper composites. *Mater Chem Phys* 128(1-2): 114-120. <https://doi.org/10.1016/j.matchemphys.2011.02.065>
4. Goyal RK, Tiwari AN, Mulik UP, Negi YS. 2007. Novel high performance Al<sub>2</sub>O<sub>3</sub>/poly(ether ether ketone) nanocomposites for electronics applications. *Compos Sci Technol* 67(9): 1802-1812. <https://doi.org/10.1016/j.compscitech.2006.10.020>
5. Araki Y. 1965. Thermal expansion coefficient of polytetrafluoroethylene in the vicinity of its glass transition at about 400 °K. *J Appl Polym Sci* 9(2): 421-427. <https://doi.org/10.1002/app.1965.070090203>
6. Mahesh KV, Balanand S, Raimond R, Mohamed AP, Ananthakumar S. 2014. Polyaryletherketone polymer nanocomposite engineered with

- nanolaminated  $Ti_3SiC_2$  ceramic fillers. *Mater Des* 63: 360-367. <https://doi.org/10.1016/j.matdes.2014.06.034>
7. Choudhary V, Dhawan S, Saini P. 2012. Polymer Based Nanocomposites for Electromagnetic Interference (EMI) Shielding. In Jaroszewski M, Ziaja J (eds) EMI Shielding - Theory and Development of New Materials. Research Signpost, India.
  8. Suryanarayana C. 2001. Mechanical alloying and milling. *Prog Mater Sci* 46(1-2): 1-84. [https://doi.org/10.1016/S0079-6425\(99\)00010-9](https://doi.org/10.1016/S0079-6425(99)00010-9)
  9. Al-Saleh MH, Saadeh WH, Sundararaj U. 2013. EMI shielding effectiveness of carbon based nanostructured polymeric materials: a comparative study. *Carbon* 60: 146-156. <https://doi.org/10.1016/j.carbon.2013.04.008>
  10. Kim BJ, Bae KM, Lee YS, An KH, Park SJ. 2014. EMI shielding behaviors of Ni-coated MWCNTs-filled epoxy matrix nanocomposites. *Surf Coat Technol* 242: 125-131. <https://doi.org/10.1016/j.surfcoat.2014.01.030>
  11. Chou KS, Huang KC, Shih ZH. 2005. Effect of mixing process on electromagnetic interference shielding effectiveness of nickel/acrylonitrile-butadiene-styrene composites. *J Appl Polym Sci* 97(1): 128-135. <https://doi.org/10.1002/app.21740>
  12. Al-Saleh MH. 2015. Influence of conductive network structure on the EMI shielding and electrical percolation of carbon nanotube/polymer nanocomposites. *Synth Met* 205: 78-84. <https://doi.org/10.1016/j.synthmet.2015.03.032>
  13. Goyal R, Sulakhe R. 2015. Study on poly(vinylidene fluoride)/nickel composites with low percolation. *Adv Mater Lett* 6(4): 309-317. <https://doi.org/10.5185/amlett.2015.5627>
  14. Shakir MF, Khan AN, Khan R, Javed S, Tariq A, et al. 2019. EMI shielding properties of polymer blends with inclusion of graphene nano platelets. *Results Phys* 14: 102365. <https://doi.org/10.1016/j.rinp.2019.102365>
  15. Singh B, Mohanty A. 2017. Review paper on the effect of dispersion of particles and its effect on the properties of composite materials. *Int J Mech Eng Technol* 8(9): 140-149.
  16. Wei D, Dave R, Pfeffer R. 2002. Mixing and characterization of nano-sized powders: an assessment of different techniques. *J Nanopart Res* 4: 21-41. <https://doi.org/10.1023/A:1020184524538>