Study on Two Body Abrasive Wear behaviour of Carboxyl-Graphene Reinforced Epoxy Nano-composites

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Abstract. The focus of this study was to investigate the effect of carboxylic acid (COOH) functionalized graphene (CGr) content on abrasive wear behavior of epoxy nanocomposites. CGr-reinforced epoxy (CGr//Ep) nanocomposites were fabricated using probe sonicator for dispersion and vacuum oven for curing. The percentage of CGr in the developed composites was varied from 0.2 to 1 wt. % with an increament of 0.2 wt. %. The abrasive wear tests were conducted on the developed CGr/Ep composites on SiC abrasive paper with two grit sizes at constant velocity and constant load for varying abrading distance. The worn surfaces were analyzed using Scanning Electron Microscope and the images reveals that the developes nanocomposites exhibits good tribolofical performance at low filler loading (≤0.6 wt. %). Neat epoxy showed the highest specific wear rate as well as high wear volume. On the other hand, epoxy with 0.6 wt. % of CGr exhibited the least friction coefficient and superior wear resistance for 320 grit SiC abrasive paper. It is predicted that the good interfacial adhesion between CGr and epoxy matrix and also tribo-chemical reactions between CGr layer and epoxy matrix for reducing wear rate of the composite materials.

1. Introduction

The understanding of wear process in case of polymers and its composites are difficult. Because of interacting of materials in the crack formation prompting the creation of wear particles [1]. A few scientists have given major importance in the study of wear performance of composite materials because of their good tribo-mechanical properties [2]. Nevertheless, there are some tribological limitations in polymer composite due to its low thermal and hardness property which reduces the wear behavior of composites at high loads and high temperatures [3]. In order to overcome from this limitations, polymers should be reinforced with fibers/fillers especially carbon based materials which improves the wear properties of the material.

The nano-filler reinforced polymer composites play an important role in order to improve the wear behavior of the material because of large surface area which leads to high strength to weight ratio of the material [4]. Generally the optimum filler content ranges between 1 – 4 vol. %, if the filler content increases, there might be a chances of improper dispersion causes agglomeration, ultimately which leads to reduce the properties of the material [5]. Hence the proper dispersion of nanoparticles in the

matrix and good interfacial adhesion between filler and matrix improves wear the performance of the nanocomposites [6].

Graphene is a single layer of carbon atoms arranged in a hexagonal honeycomb lattice is an effective nano particle compared to carbon nanotubes and fullerenes which improves the tribo-mechanical properties of the material [7-9]. Suresha et al [10] studied the two body abrasive wear behavour of graphite reinforced carbon-epoxy composites. They showed that wear rate reduced drastically with the presence of graphite filler in the composites; and also wear volume reduced considerably with increase in the content of graphite. Also showed about the interaction between fillers and distribution of the filler in matrix enhances the wear resistance of the C-E composite. Friedrich et al. [11] investigated that nanofiller loading play a vital part on the tribological performance in nanocomposites. They showed that larger addition of fillers agglomeration takes place, which decrease uniformal distribution of particles and wear property. Afroza et al. [12] studied the wear behavior of CNF/epoxy nanocomposites. They showed that CNF content in the composite significantly affected specific wear rate but not frictional coefficient. Prithu et al. [13] focused on the recent development in tribo-mechanical behavior of self-lubricating metallic nanocomposites reinforced by carbonaceous nano materials namely, graphene and CNT. They showed that adding graphene and CNT to metals reduces wear rate and COF as well as increases the tensile strength. Nay et al. [14] researched the impacts of graphene filler on tribological and mechanical properties of epoxy composites. They reported that hardness, stiffness, and tensile strength of the composites increases with increasing graphene content due to the superior elastic modulus and hardness of graphene filler than those of neat epoxy matrix.

The two dimensional nano material graphene exhibits better wear resistance in tribological applications [15]. Because of good self lubricant property of graphene, which slides over a surface can be sheared of easily due to morphology of graphene and it provides good COF for longer period due to the formation of transfer film [16]. Lee et al. [17] reported that graphene used in tribological application is limited and showed that graphene is promising reinforcing material in polymer composite due to its good adhesion and good compatibility with the matrix. Shen et al. [18] compared the graphene with other nano fillers which is reinforced to polymeric resin. They showed that wear resistance increases and specific wear rate reduces with increasing graphene content and also they compared the graphene with other nano fillers namely CNT, TiO_2, Al_2O3, SiO_2, the role of graphene is better in enhancing material properties. In another investigation by Steurer et al. [19] presence of functionalized carboxyl graphene improves the dispersion quality however the existence of OH group provides the better mechanical adhesion with the epoxy matrix.

From the literature it is observed that tribo-mechanical behaviour of epoxy reinforced by carbon based materials namely, graphene, CNT, fullerene and CNF are rare or scanty. Considering that the improvement of tribo-mechanical of the polymers as a result of the inclusion of graphene nanoparticles is not remarkable enough, the current work is bringing the effect of nano-graphene particles into play and having a deeper understanding of the mechanisms involved as well. Thus, in the present work, mechanical and wear properties of surface modified graphene-reinforced epoxy composites aim was studied. The concentration of graphene was varied from 0.2 to 1 wt. % in steps of 0.2 wt. %. Our hypothesis, based on prior literature is that CGr will react with the epoxy resin and form a new CGr-Ep nanocomposite with improved cohesion strength and this will improve the mechanical properties as well as wear resistance.

2. Materials and Methods

2.1. Materials

Araldite LY 1564 SP is an epoxy resin and curing agent cycloaliphatic polyamine hardener (Aradur 3486) were by M/S. Seema Fine Chem Industry LPP, Maharashtra. The epoxy resin is having a viscosity of 1200-1400 cps, density of 1.15 g/cm^3 and having flash point of 185 ◦C. The functionalized (COOH) graphene (CGr) is supplied by United Nanotech Bangalore, India. The nano-sized CGr sheet

has an average length of 1-10 microns, thickness of 0.8-1.6 nm, average number of layers is 3-6, and bulk density is around 0.24 g/cm^3. The Graphene was functionalized with 1.23 wt. % of carboxylic groups (COOH).

2.2. Fabrication of composites

The carboxyl functionalized graphene powder was first dispersed in ethanol by mechanical shearing followed by ultra sonication at 60℃ for 2 hrs. The so-obtained functionalized graphene– ethanol solutions with varying weight fractions of CGr were then dispersed in epoxy (epoxy LY1564) matrix by bath sonication for 30 min. The viscous liquids were further processed by probe sonication for 60℃ for 1 hr; the mixture was then degassed at 60℃ for 1 day to effect ethanol removal, followed by addition of hardener (hardener XB3486) in weight ratio of 100:34. The resultant solution immediately poured into a mould cavity for 30 hrs in order to cure the specimen. The composite so obtained is further processed by a post curing in a vacuum oven at 80℃ for 2 hrs. Figure 1 shows the fabrication process of CGr/Ep composite.

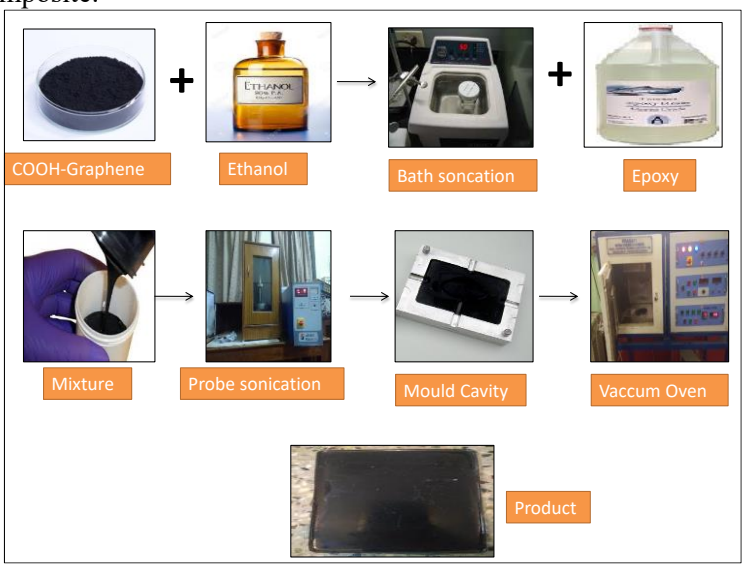

Figure1. Fabrication process of CGr/Ep nanocomposites.

3. Wear test

The abrasive wear test was conducted using pin-on-disc apparatus. A pin-on-disc setup (Make: Magnum Engineers) is as shown in Figure. 2a is used for abrasive wear experiments. The sample (6 mm × 6 mm × 2.5 mm) was subjected to abrasive wear test with a load of 5 N; sliding velocity of 0.5 m/s; and sliding distance of 2 m, 4 m. 6 m, and 8 m with a track diameter of 80 mm. The samples were set parallel to the abrasive surface and abrasive wear was measured by calculating the difference in initial weight and final weight.

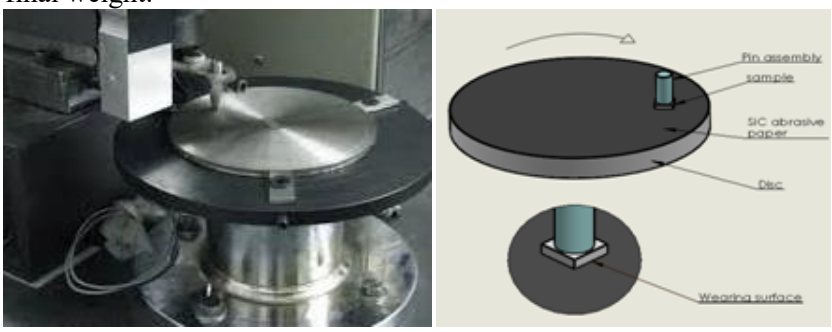

Figure 2. a) Pin-on-disc wear tester b) Abrasive wear mechanism

Table 1. Wear test conditions.

Composites	AppliedLoad [N]	Sliding velocity [m/s]	Sliding distance [m]
Neat epoxy (Ep)	5	0.5	2 m 4 m 6 m 8 m
0.2wt. % graphene (0.2CGr-Ep)			
0.4wt. % graphene (0.4CGr-Ep)			
0.6wt. % graphene (0.6CGr-Ep)			
0.8wt. % graphene (0.8CGr-Ep)			
1.0wt. % graphene (1.0CGr-Ep)			

The wear behaviour of CGr/Ep nanocomposite samples was assessed using pin-on-disk wear tribo-tester under dry conditions at room temperature under the test conditions listed in Table 1. The wear was measured by the loss in weight, which was then converted into wear volume using the measured density data. Then from the test the specific wear is calculated using the relation:

$$Ks = \frac{\Delta V}{F_N \times L} \quad [\frac{mm^3}{Nm}] \tag{1}$$

Where, ΔV = Volume of wear loss ; F_N = Normal load and L = Sliding distance

4. Results and Discussion

4.1. Wear Volume and specific wear rate in CGr/Ep nanocomposites

Abrasive wear volume of CGr-Ep nanocomposites worn on 150 grit and 320 grit size of SiC abrasive paper with a distance of 2 m, 4 m, 6 m, and 8 m and constant velocity of 120 rpm under a applied normal load of 5 N is shown in Figure 3a and b. The Figure 3.a shows that, increase the abrading distances, the wear volume of the composite samples increases and at 0.6 wt. % of CGr content in epoxy composites show less abrasive wear volume compare to other nanocomposites.

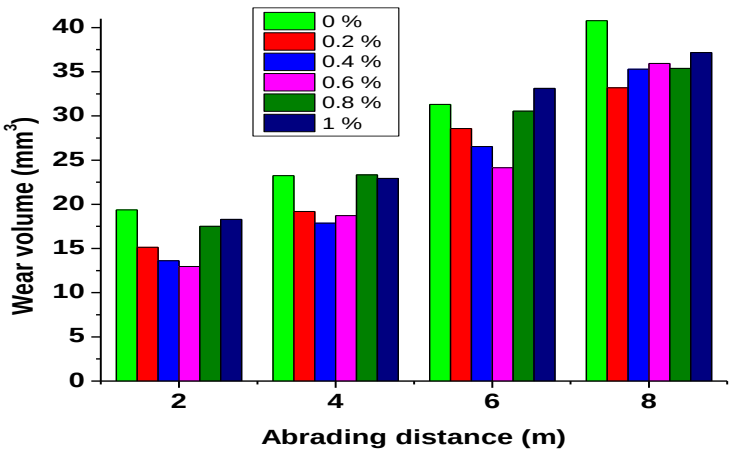

Figure 3 a. Wear volume vs. abrading distance of samples at 5 N, 150 grit

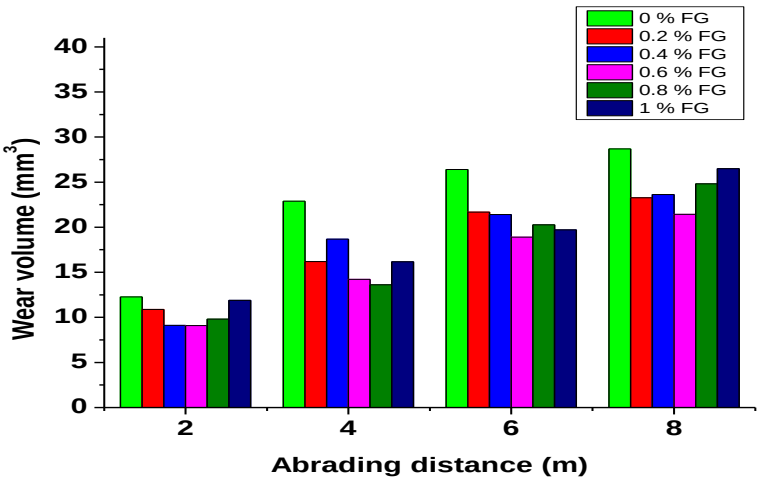

Figure 3 b. Wear volume vs. abrading distance of samples at 5 N, 320 grit

Similarly Figure 3.b shows that, uneven abrasive wear volume of composite samples, almost at 0.6 wt. % of CGr-Ep sample show less abrasive wear volume and also it shows that the abrasive wear volume mainly depends upon the grit size of the abrasive paper, applied normal load, sliding velocity and abrading distances.

Specific wear rate of CGr-Ep nanocomposites worn on 150 grit and 320 grit size of SiC abrasive paper with a distance of 2 m, 4 m, 6 m, and 8 m and constant sliding velocity of 120 rpm under a applied normal load of 5 N is shown in Figure 4a and b. The results show that, the specific wear rate of all the samples decreased with increasing sliding distance and grit size of the SiC abrasive paper. Specific wear rate of unfilled and CGr filled epoxy nanocomposites at 5 N and 150 grit sizes is depicted in Figure 4 a.

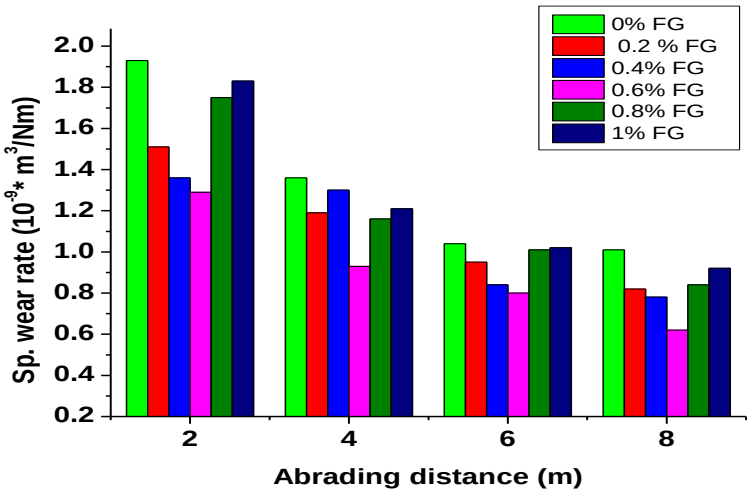

Figure 4 a. Specific wear rate vs. abrading distance of samples at 5N, 150 grit

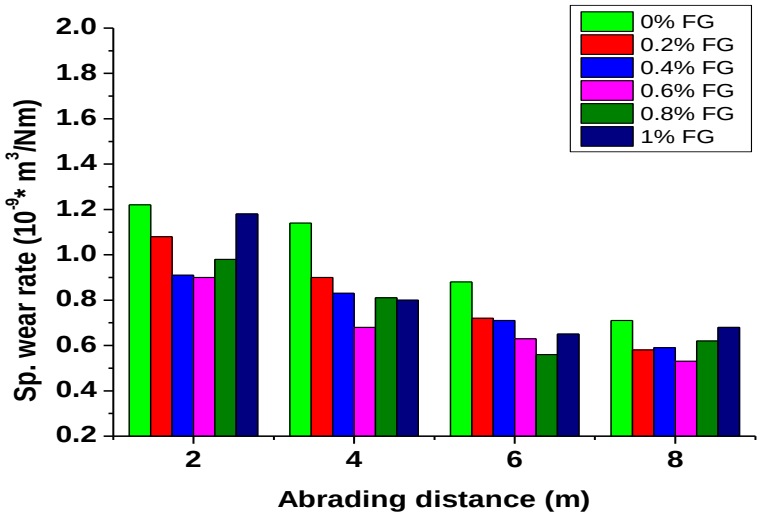

Figure 4 b. Specific wear rate vs. abrading distance of samples at 5 N, 320 grit

The rate of wear decreases significantly from 1.9× 10^{-9} m^3/Nm for neat epoxy sample to less than 0.65× 10^{-9} m^3/Nm for 0.6 wt. % CGr into epoxy matrix under 8 m distance and 5 N applied load. During the first interval, CGr-Ep samples exhibited a higher wear rate after that gradually reduced in repeated intervals of time. It can be seen that as the filler content increases the specific wear rate decreases. This may be due to the increased filler loading, there will be less exposure of epoxy material to the contacting surface, and hence less wear was found in CGr-Ep nanocomposites. In Figure 4b, the grit size of the abrasive paper increase from 150 to 320, that is coarse size to fine size. Herein results reveal that increase in grit size of the abrasive paper there was decrease in specific wear rate, this because of large particles of abrasive leads to damage the surfaces compare to fine particles of abrasive and also it seems that from figure that the wear resistance of CGr-Ep is high when compare with it for neat epoxy matrix. This may due to the uniform distribution as well better filler/matrix interfacial adhesion in CGr-Ep nanocomposites. From Figure 4a and b, it can be seen that as the filler content increases the specific wear rate decreases. This may be due to the increased filler loading, there will be less exposure of epoxy material to the contacting surface, and hence less wear was found in CGr-Ep nanocomposites. Further, during abrasive wear process, the debris formed, which are fine particles of epoxy matrix as well as graphene, act as rollers and hence protects the material from wearing when rubbed against the counterface.

4.2. Worn surface morphology

The morphologies of worn surfaces of the neat epoxy and its composites with varying wt. % of CGr were presented in Figure 5and Figure 6.

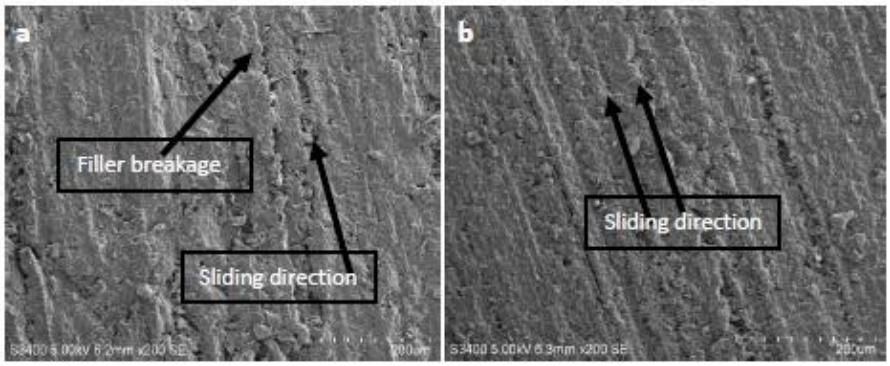

Figure 5. SEM pictures of 0.2 % CGr-Ep abraded against (a) 150 grit and (b) 320 grit for 4m.

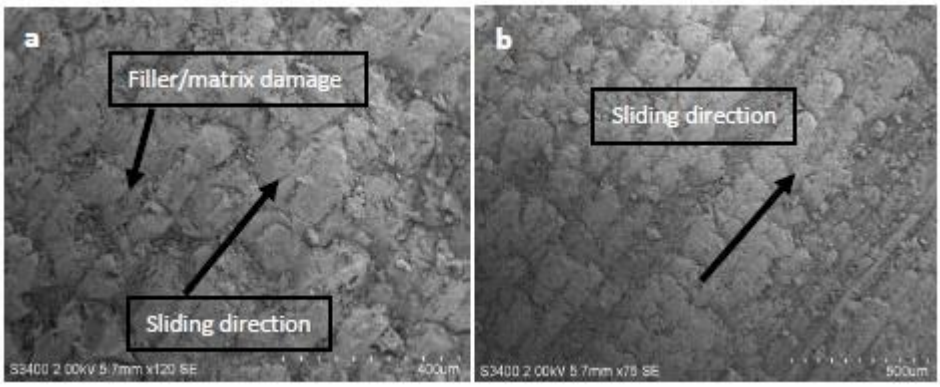

Figure 6. SEM pictures of 0.6 % CGr-Ep abraded against 150 grit SiC paper (a) 4 m and (b) 8 m.

During abrasion process there are several mechanism seen to play a key role which results removal of material from the surfaces. Normally two body abrasive wear process involves four different mechanisms: Micro ploughing, mico cutting, mcro fatigue and micro cracking.

Figure 5a and b shows the worn surfaces of 0.2 wt. % of CGr-Ep samples abraded against 150 grit SiC and 320 grit SiC abrasive paper at an abrading distance of 4 m under a load of 5 N respectively. Figure 5a shows composite which reveals that matrix damage could be occur due to the coarse size of the abrasive particle which is visible from the micrograph. Figure 5b shows the smooth surface of the specimen when it is abraded at 320 grit SiC abrasive paper has less matrix damage and also observed at some regions voids and cracks from the micrograph.

Figure 6a and b shows the worn surfaces of 0.6 wt. % of CGr-Ep samples abraded against 150 grit SiC abrasive paper under a load of 5 N at an abrading distance of 4 m and 8 m respectively. Figure 6a shows composite which reveals that matrix damage could be occur due to the ploughing and cutting action by large size of the abrasive particle which is in the form of large matrix debris visible from the micrograph. Figure 6b shows composite which reveal that matrix damage may be due to the ploughing and cutting action by larger size of the abrasive particle which is in the form of comparatively less matrix debris visible from the micrograph. It is clear that larger size of the SiC abrasive particles becomes ineffective when these particles get crushed by increasing the abrading distances.

5. Conclusions

The abrasive wear characteristics of neat epoxy and CGr reinforced epoxy nanocomposites abrading against SiC abrasive paper have been investigated. The influence of reinforcement with 0.2 to 1.0 wt. % loading in the epoxy nanocomposite have been tested under different abrading distance with constant load and abrading velocity.

- In abrasive wear test, the composite samples mainly depend on the grit size of the abrasive paper, abrading distance, load and speed.
- The grit size of the abrasive paper increases, the wear volume of the CGr-Ep composite decreases.
- With increase in the distance, the specific wear rate decreases for all the composite samples. Moreover, 0.6 wt. % CGr-Ep nanocomposite showed optimum wear rate and relatively low wear volume

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References

[1] M.Z. Rong, M.Q. Zhang, H. Liu, H.M. Zeng, B. Wetzel, K. Friedrich, Microstructure and tribological behavior of polymeric nanocomposites, Ind Lubr Tribol 53 (2001) 72–77.
[2] M.Q. Zhang, M.Z. Rong, S.L. Yu, B. Wetzel, K. Friedrich, Improvement of tribological performance of epoxy by the addition of irradiation grafted nano-inorganic particles, Macromol Mater eng 287 (2002) 111–115.
[3] M.R. Scanlon, R.C. Cammrata, Mechanical properties of nano composite granular metal thin films, J Appl Phys 76 (1994) 3387–3393.
[4] S. K. Rhee, M.G. Jacko, P.H.S. Tsang, Role of friction film in friction, wear and noise of automotive brakes, Wear 146 (1991) 89-97.
[5] K Friedrich, Z. Zhang, A.K. Schlarb, Effects of various fillers on the sliding wear of polymer composites, Compos Sci Technol 65 (2005) 2329-2343.
[6] Prithu, P. Mukhopadhyay, R.K. Gupta, Graphite, Graphene, and their polymer nanocomposites, CRC Press 2012.
[7] Long-Cheng Tang, Yan-Jun Wan, Dong Yan, Yong-Bing Pei, Li Zhao, Yi-Bao Li, Lian-Bin Wu, Jian-Xiong Jiang, Guo-Qiao Lai, The effect of graphene dispersion on the mechanical properties of graphene/epoxy composites, Carbon 60 (2013) 16 –27.
[8] Kuilla, Tapas, Sambhu Bhadra, Dahu Yao, Nam Hoon Kim, Saswata Bose, Joong Hee Lee, Recent advances in graphene based polymer composites, Progress in polym sci 35 (2010) 1350-1375.
[9] B. Suresha, B. N. Rmesh, K. M. Subbayya, B. N. Ravikumar, G. Chandramohan, Influence of graphite filler on two-body abrasive behavior of carbon fabric reinforced epoxy composites, Materials and Design 31 (2010) 1833-1841.
[10] M.Z. Rong, M.Q. Zhang, H. Liu, H.M. Zeng, B. Wetzel, K. Friedrich, Microstructure and tribological behavior of polymeric nanocomposites, Ind Lubr Tribol 53 (2001) 72–77.
[11] K. Friedrich, A.K. Schlarb, Tribology of polymeric nanocomposites: Friction and wear of bulk materials and coatings, 2nd ed., Oxford Butterworth-Heinemann 2013.
[12] Afroza Khanam, Bablu Mordina, R. K. Tiwari, Statistical evaluation of the effect of carbon nanofibre content on tribological properties of epoxy nanocomposites, Journal of composite materials 49(2015) 2497-2507.
[13] Prithu, P. Mukhopadhyay, , R.K. Gupta, Graphite, Graphene, and their polymer nanocomposites, CRC Press 2012.
[14] Nay Win Khun, He Zhang, Mechanical and tribological properties of grapheme modifies epoxy composites, Kmutnb Int J Apply Sci Technol, 8(2015) 101-109.
[15] D.G. Teer, New solid lubricant coatings, Wear 251 (2001) 1068-1074.
[16] K. Friedrich, A.K. Schlarb, Tribology of polymeric nanocomposites: Friction and wear of bulk materials and coatings, Elsevier 2011.
[17] H. Lee, N. Lee, Y. Seo, J. Eom, Comparison of frictional forces on graphene and graphite, Nanotechnology 20 (2009) 325701-6.
[18] X.J. Shen, X.Q. Pei, Y. Liu, S.Y. Fu, Tribological performance of carbon nanotube-graphene oxide hybrid/epoxy composites, Compos Part B Eng 57 (2014)120-125.
[19] P. Steurer, R.Wissert, R. Thomann, R. Muelhaupt, Functionalized graphene and thermoplastic nanocomposites based upon expanded graphite oxide, Macromol Rapid Commun 30 (2009) 316-327.