CHAPTER (3)

RESULTS & DISCUSSION

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3.1 INTRODUCTION

The present chapter introduces results obtained from studying the physical, mechanical, and tribological properties of epoxy-based composite materials under consideration. The results were discussed and compared to other researchers investigations whenever possible.

3.2 MICROSTRUCTURAL EXAMINATIONS

Microstructure of epoxy/Al_2O_3, epoxy/SiC and epoxy/graphite particulate reinforced polymeric matrix composites (PMCs) under study are shown in Figures 3-1 to 3-3 respectively.

Microstructural examinations revealed that the distribution of the ceramic particulates inside the epoxy matrix was fairly homogenous, particularly, at low volume fraction (i.e. 10%). Few agglomeration sites were observed at PMCs containing 10% volume fraction of the particulates. However, increasing the volume fraction of the particulates increased the agglomeration% of the particulates inside the matrix. For instance, increasing the SiC particulates volume fraction from 10% to 30% increases dramatically the agglomeration % (compare Figures 3-2a and 3-2c).

It is important to mention that, in the present investigation, the agglomeration % of the particulates was evaluated only using optical microscope. The sites contain more than three particles in contact to each other were considered as an agglomeration site. In fact, at low volume fraction (up to 10%) there was a low tendency of the particles to agglomerate when compared with high volume fractions (up to 30%). Accordingly, good distribution of the particles inside the matrix can be obtained easily in such composites.
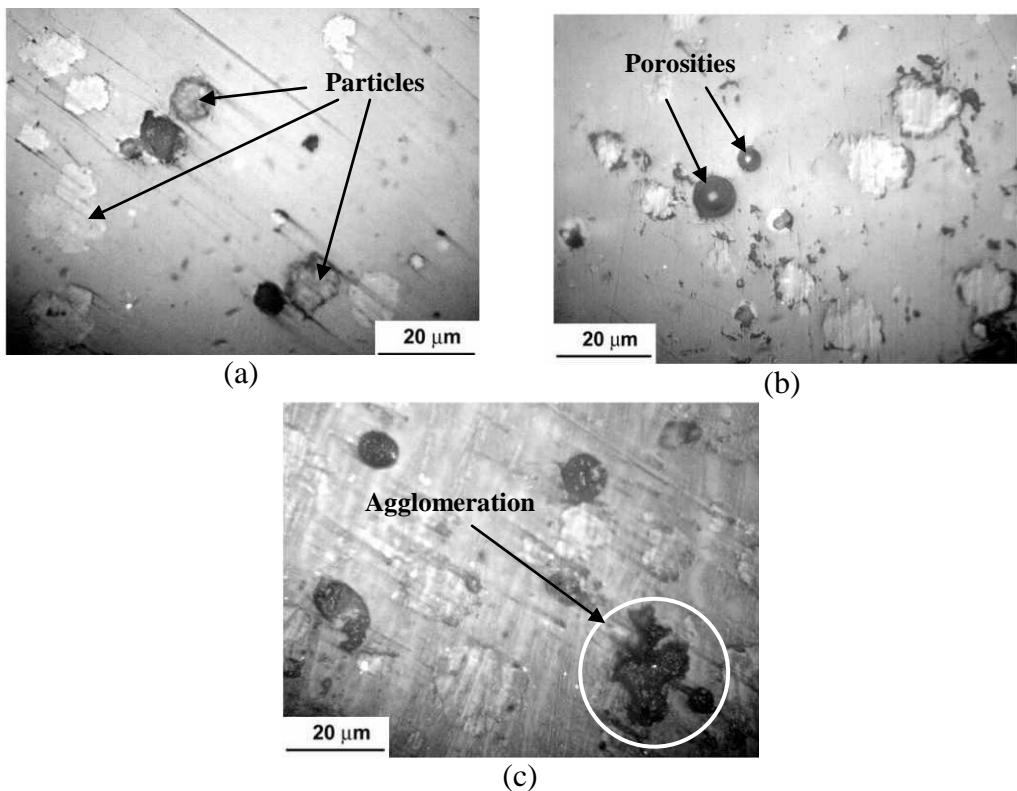
Figure 3-1: Microstructure of epoxy/Al$_2$O$_3$ PMCs (a) epoxy/10% vol.-% Al$_2$O$_3$, (b) epoxy/20% vol.-% Al$_2$O$_3$ and (c) epoxy/30% vol.-% Al$_2$O$_3$,

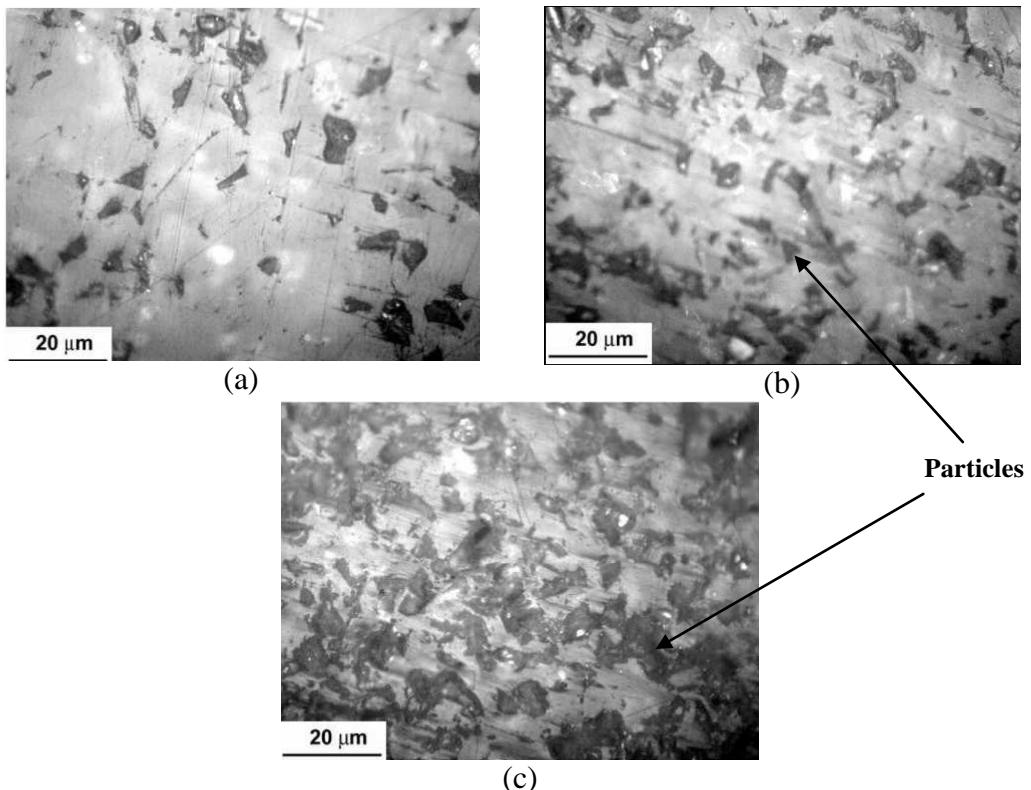Figure 3-2: Microstructure of epoxy/SiC PMCs (a) epoxy/10% vol.-% SiC, (b) epoxy/20% vol.-% SiC and (c) epoxy/30% vol.-% SiC,

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<div align="center">(a)</div>

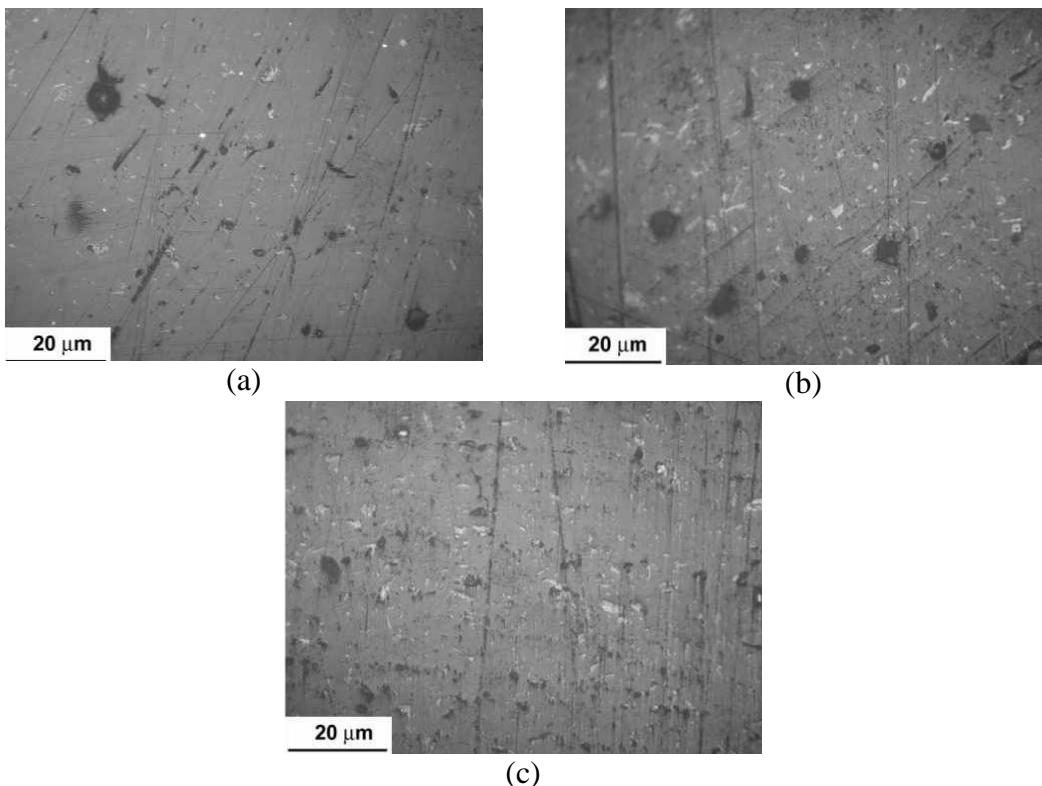<div align="center">(b)</div>

<div align="center">(c)</div>

Figure 3-3: Microstructure of epoxy/graphite PMCs (a) epoxy/10% vol.-% gr.,
(b) epoxy/20% vol.-% gr. and (c) epoxy/30% vol.-% gr.,

3.3   DENSITY MEASUREMENTS

Density of the pure epoxy matrix was measured and found to be 1.14 gm/cm^3. Table 3-1 gives the numerical values of the measured densities of PMCs under study. Figure 3-4 shows the variation of the measured density of materials under study with volume fraction of particulates for different composites under investigation.

Table 3-1: Measured densities of the PMCs under study.

| Material	Measured Density (5 samples) (gm/cm^3)					Average Measured density (gm/cm^3)
Epoxy matrix (Ep)	1.13	1.15	1.14	1.16	1.14	1.14
Ep+10% Al$_2$O$_3$	1.17	1.14	1.16	1.15	1.19	1.16
Ep+20% Al$_2$O$_3$	1.29	1.25	1.20	1.23	1.23	1.24
Ep+30% Al$_2$O$_3$	1.32	1.28	1.30	1.23	1.32	1.29
Ep+10% SiC	1.32	1.28	1.33	1.26	1.25	1.29
Ep+20% SiC	1.33	1.32	1.36	1.35	1.28	1.33
Ep+30% SiC	1.44	1.45	1.46	1.49	1.41	1.45
Ep+10% graphite	1.20	1.15	1.12	1.15	1.17	1.16
Ep+20% graphite	1.12	1.15	1.14	1.12	1.15	1.14
Ep+30% graphite	1.16	1.22	1.24	1.20	1.22	1.21
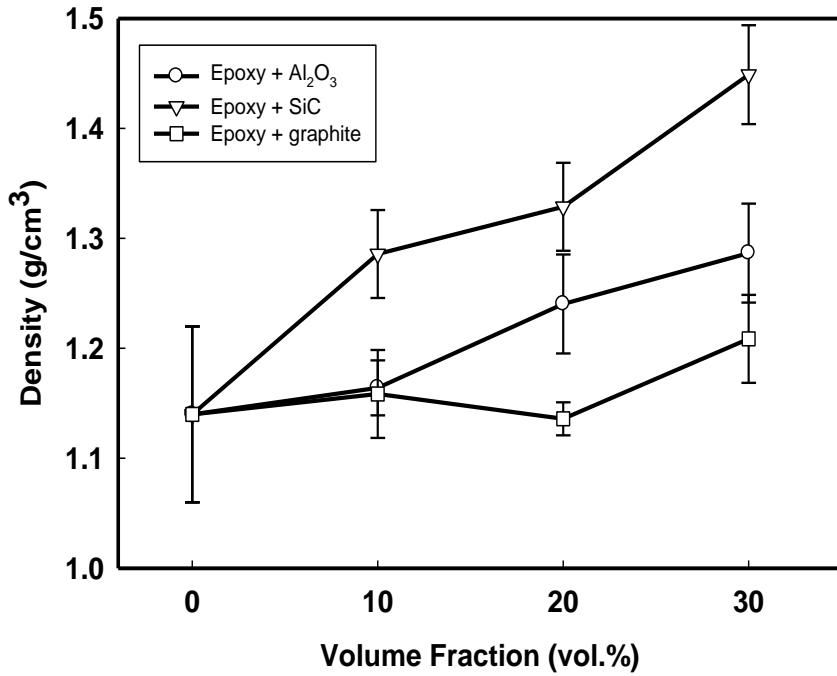
Figure 3-4: Variation of the measured density of materials under study
with volume fraction of particulates for different composites.

Generally, composite materials exhibited higher densities than the pure epoxy. Moreover, it has been noticed that epoxy/SiC composites exhibited higher densities when compared with the other epoxy/Al_2O_3 and epoxy/graphite composites. The epoxy/graphite composites exhibited the lowest densities among the investigated composites. This trend can be explained by the difference in bulk densities of particulates. SiC, Al_2O_3, and graphite have bulk densities of 3.99, 3.21 and 2.27 g/cm^3, respectively [61].

It has been noticed that density of the composite reinforced with 10 vol. % Al_2O_3 particulates is approximately equal to the density of base matrix (1.16 gm/cm^3), and this phenomena can be emphasized when knowing that the effect of micro air bubbles entrapped into the mixture on decreasing density is much greater than the effect of amount of Al_2O_3 particles on increasing density. When increasing the amount of Al_2O_3 particles in the composite material its density increased alleviating the effect of micro bubbles of air. As increasing the amount of SiC particulates the density of the composite material increased due to the high density of SiC particles with relative to the base matrix. Increasing the amount of graphite particulates till to

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20 vol. % did not show any noticeable change in the density of the composites because of increasing the effect of porosity introduced due to the entrapped air bubbles on the surface of graphite particulates.

According to the results shown in Figure 3-4, it can be concluded that the addition of Al_2O_3 and SiC particulates to the epoxy matrix increases the density of the composites. Such increase was found to be proportional to the volume fraction of the ceramic particulates added to the matrix. The aforementioned results obtained in the current work had been reported by many workers [62, 63]. For example, *Agarwal and Broutman* [63] stated that, the density of reinforced matrix is usually higher than that of the pure resin.

3.4   MECHANICAL CHARACTERISTICS OF COMPOSITES

3.4.1   Compressive Strength

The pure epoxy matrix exhibited a compressive strength of about 86 MPa and a modulus of elasticity of about 1 GPa. The compressive strengths as well as the modulus of elasticity of the different composites are given in Table 3-2 and Table 3-3 respectively. Figures 3-5 and 3-6 graph the variation of both compressive strength and modulus of elasticity of composites respectively with the volume fraction of particulates.

Table 3-2: Measured compressive strengths (MPa) of the PMCs under study

Material	Strength measurements (MPa) (3 samples)			Average strength (approx.)
Epoxy matrix	86.39	85.50	85.41	86±0.5
Ep+10% Al_2O_3	95.07	96.20	99.99	97±2.5
Ep+20% Al_2O_3	84.88	108.48	100.39	98±11.8
Ep+30% Al_2O_3	103.10	99.03	99.43	101±2.0
Ep+10% SiC	85.34	97.78	90.09	91±6.2
Ep+20% SiC	101.29	103.78	105.59	104±2.2
Ep+30% SiC	98.35	101.07	96.88	99±2.0
Ep+10% graphite	91.45	89.98	91.45	91±0.7
Ep+20% graphite	90.20	86.47	90.43	89±2.0
Ep+30% graphite	58.74	78.32	60.44	66±10.0

Table 3-3: Modulus of elasticity (GPa) of the PMCs under study

Material	Modulus of elasticity (GPa) (3 samples)			Average Modulus (approx.)
Epoxy matrix	1.4	0.9	0.8	1.0±0.3
Ep+10% Al_2O_3	0.9	1.7	2.4	1.7±0.8
Ep+20% Al_2O_3	3.4	2.8	1.6	2.6±0.9
Ep+30% Al_2O_3	2.3	2.3	1.8	2.1±0.3
Ep+10% SiC	2.5	2.7	2.6	2.6±0.1
Ep+20% SiC	1.7	1.8	1.8	1.8±0.04
Ep+30% SiC	3.2	2.3	3.0	2.8±0.5
Ep+10% graphite	0.9	0.5	1.5	1.0±0.5
Ep+20% graphite	1.8	1.1	1.1	1.3±0.4
Ep+30% graphite	1.9	2.2	1.9	2.0±0.15

The results revealed that both epoxy/Al_2O_3 and epoxy/SiC composites have higher compressive strength and modulus of elasticity when compared with the pure epoxy matrix. For epoxy/Al_2O_3 composites, it has been found that the compressive strength is proportional to the volume fraction of Al_2O_3 particulates, while its modulus of elasticity decreases at 30 vol. % of particulates. For epoxy/SiC composites, the maximum compressive strength was exhibited by composites containing 20 vol. % of particulates. These composites exhibited a modulus of elasticity lower than that of other epoxy/SiC composites. This can be referred to the occurrence of brittle fracture. Furthermore, for the epoxy/graphite composites, the maximum compressive strength was exhibited by composites containing 10 vol. % of particulates. Modulus of elasticity of epoxy/graphite composites was found to be proportional to volume fraction of particulates.
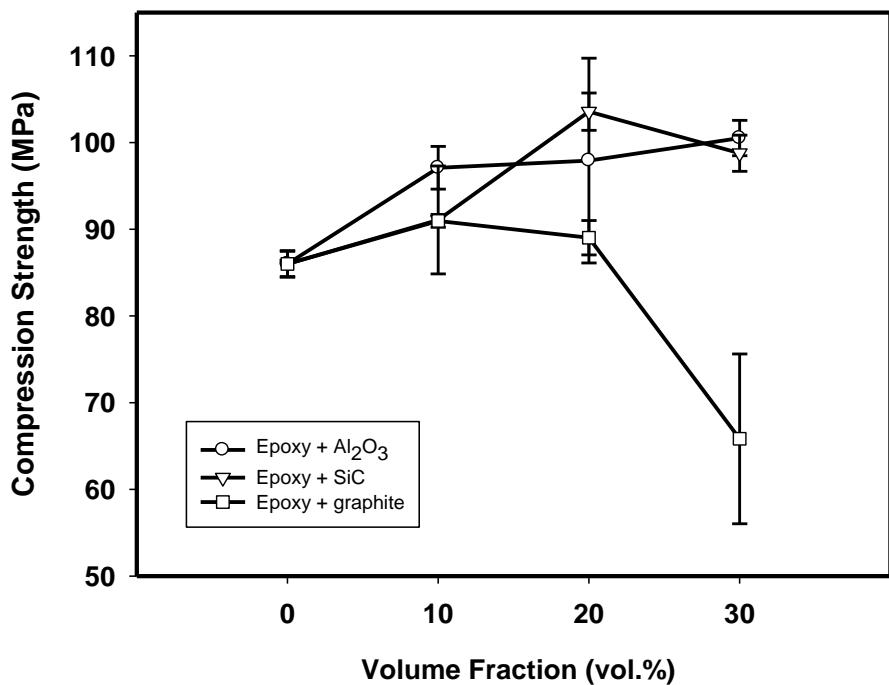
Figure 3-5: Variation of compressive strength of materials under study
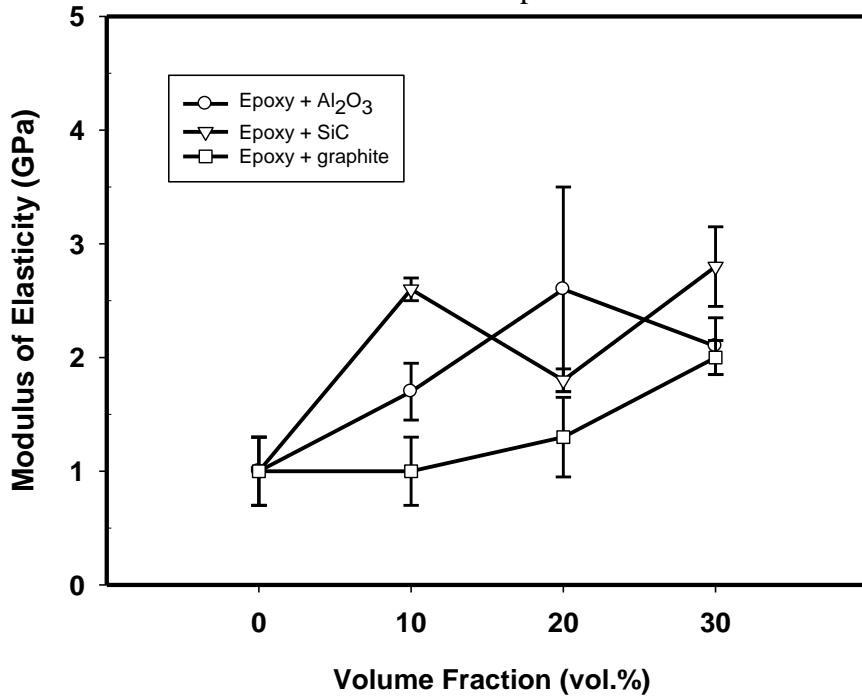with volume fraction of particulates

Figure 3-6: Variation of modulus of elasticity of materials under study
with volume fraction of particulates

Epoxy based composites with 20 vol. % SiC particulates showed the highest compressive strength (104 MPa) and those reinforced with 30 vol. % SiC showed the highest modulus of elasticity (2.8 GPa) ; while composites containing 30 vol. % graphite particulates showed the lowest compressive strength (66 MPa) which is lower than that of the pure epoxy (86 MPa).

The aforementioned results, suggest that there is a critical volume fraction of the ceramic particulates. Increasing the volume fraction above this critical volume fraction tends to reduce the compressive strength of the composites. This critical volume fraction was found to be 10 vol. % and 20 vol. % for epoxy/graphite and epoxy/SiC composites respectively. For epoxy/Al_2O_3 composites, the critical volume fraction was not determined up to 30 vol. %.

The results suggest that there is a lower degree of particle–polymer interaction occurred at higher particulates contents (30 vol. %) for epoxy/SiC and epoxy/graphite composites. This caused interfacial de-bonding or sliding during compression test and thus reduced the compressions strength. Moreover, the tendency of forming particles agglomeration may result in lower compression strength [33,34].

The increase of the compression strength due to the addition of particulate reinforcements was reported by many workers [64-66]. For example, *Antonoio Piratelli* [65] recorded that the maximum compression strength was observed for the granite-epoxy sample than that of the pure resin.

3.4.2   Hardness Measurements

Hardness of the base matrix was found to be about 19 VHN. The hardness of composites, under investigation, is listed in Table 3-4. Figure 3-7 shows the variation of hardness of composites with volume fraction of particulates.

Generally, it has been found that the addition of SiC and Al_2O_3 particulates to the epoxy matrix increased the hardness of composites. Such increase, however, was found to be little proportional to the volume fraction of SiC and Al_2O_3 particulates. For example, the epoxy/Al_2O_3 composites containing 10, 20 and 30 vol. % of Al_2O_3 particulates exhibited hardness of 39, 41 and 42 VHN, respectively.

It was noticed that the epoxy based composites reinforced with Al_2O_3 particulates have hardness more than twice that of the base matrix. Composites reinforced with 30 vol. % Al_2O_3 particles were the hardest among materials under study. It was noticed also that the effect of SiC on hardness of the matrix has the same trend as that of Al_2O_3 particles but with lower values (35 VHN), and this can be

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recognized as a consequence of the functionalized site of Al_2O_3 particulates which are able to form cross linkage with the base epoxy matrix and therefore enhance the hardness of the composite material [19].

The epoxy/graphite composites showed nearly the same hardness of the pure unreinforced epoxy. It has been found that increasing the volume fraction of graphite particulates has no effect on the hardness of epoxy/graphite composites. The epoxy/graphite composites exhibited the lowest hardness when compared with epoxy/Al_2O_3 and epoxy/SiC composites. This can be recognized as a consequence of the low capability of graphite particulates to form cross linkage with the base epoxy matrix when compared with that of both SiC and Al_2O_3 particulates [19].

The increase in hardness of epoxy-based PMCs with the addition of micro-particles was reported by many workers. For example, Chun-Ki Lama *et al*. showed that the hardness increases with increasing nanoclay content in epoxy composites [30].

Table 3-4: Measured hardness (VHN) of the PMCs under study.

| Material	Hardness (5 Samples) (VHN)					Average hardness (VHN). (approx.).
	1	2	3	4	5	
Epoxy matrix	19.75	22.69	18.71	18.32	19.33	19
Ep+10% Al_2O_3	29.27	37.79	45.91	36.83	42.76	39
Ep+20% Al_2O_3	39.81	32.49	43.62	45.77	45.49	41
Ep+30% Al_2O_3	38.61	37.73	39.58	44.85	42.57	42
Ep+10% SiC	33.01	34.05	28.90	30.72	31.24	32
Ep+20% SiC	29.56	31.56	36.27	32.66	32.66	32
Ep+30% SiC	29.34	37.62	33.32	32.32	36.63	35
Ep+10% graphite	25.28	22.10	20.55	21.51	20.99	21
Ep+20% graphite	17.63	19.57	17.18	17.28	16.06	18
Ep+30% graphite	19.98	19.96	18.09	19.45	20.92	20
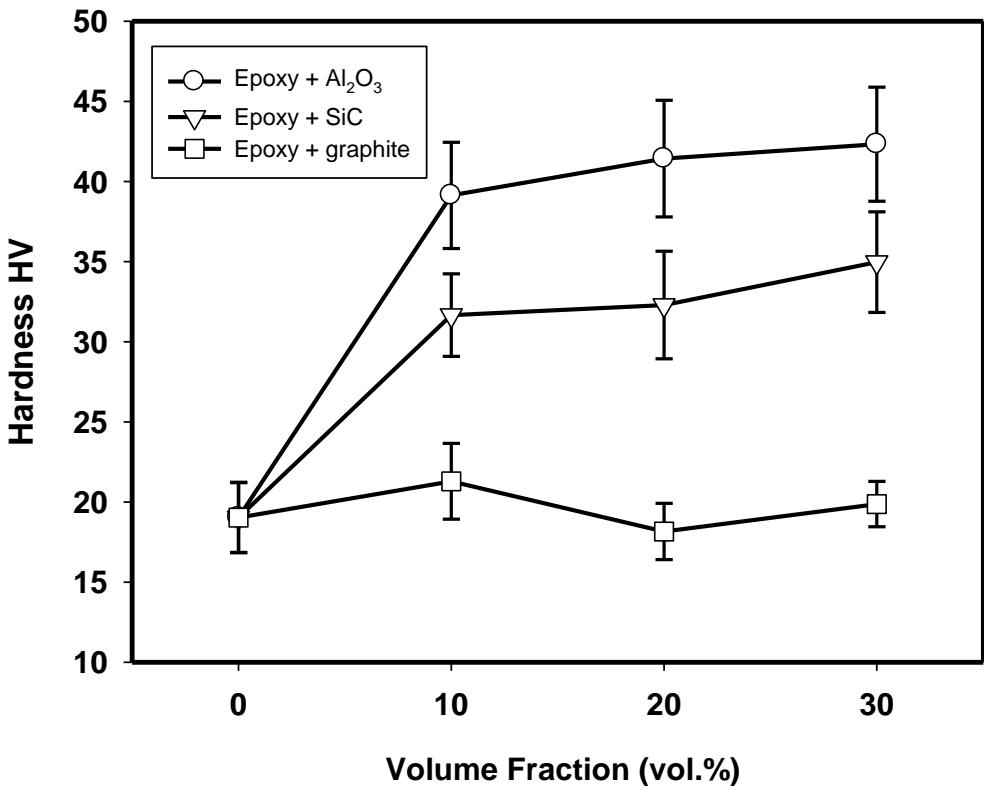
Figure 3-7: Variation of hardness of materials under study
with volume fraction of particulates

3.5 TRIBOLOGICAL CHARACTERISTICS OF COMPOSITES

As mentioned before, tribological behavior of materials under study is evaluated by studying their tribological characteristics. Wear rate (q), wear coefficient (K), wear resistance (R), and coefficient of friction (μ) were evaluated under both dry and water lubricated sliding conditions. Variation of wear characteristics were plotted separately against particle volume fraction, normal load, and particle type. Details of data obtained under both dry and water lubricated sliding conditions are listed in appendices 1 and 2 respectively. These data were used by *Minitab software* to study its variance using ANOVA, and to get regression equations of wear results as functions of wear parameters.

3.5.1 Tribological Characteristics Under Dry Sliding Conditions

Tables 3-5 to 3-8 list the average values of q, K, R, and μ obtained under dry sliding conditions.

Table 3-5: wear results of Epoxy matrix obtained under dry sliding conditions.

Experiment No.	Variable parameters		q (mm^3/m) $\times 10^{-3}$	K $\times 10^{-3}$	R	μ
	f (vol.%)	N (kg)				
1	0	2.4	3.289	0.078	12780	0.7
2	0	4.4	87.719	1.138	879	0.6
3	0	6	450.780	4.289	233	0.6

Table 3-6: wear results of Ep/Al$_2$O$_3$ composites obtained under dry sliding conditions.

Experiment No.	Variable parameters		q (mm^3/m) $\times 10^{-3}$	K $\times 10^{-3}$	R	μ
	f (vol.%)	N (kg)				
1	10	2.4	7.543	0.369	2710	0.4
2	10	4.4	294.918	7.868	127	0.6
3	10	6	1340.996	26.237	38	0.6
4	20	2.4	8.065	0.418	2394	0.5
5	20	4.4	327.621	9.255	108	0.6
6	20	6	1202.603	24.912	40	0.6
7	30	2.4	3.876	0.206	4864	0.4
8	30	4.4	58.140	1.682	595	0.5
9	30	6	486.396	10.319	97	0.6

Table 3-7: wear results of Ep/SiC composites obtained under dry sliding conditions.

Experiment No.	Variable parameters		q (mm^3/m) $\times 10^{-3}$	K $\times 10^{-3}$	R	μ
	f (vol.%)	N (kg)				
1	10	2.4	5.814	0.230	4346	0.6
2	10	4.4	442.968	9.562	105	0.7
3	10	6	1139.991	18.046	55	0.6
4	20	2.4	2.820	0.114	8787	0.6
5	20	4.4	210.526	4.635	216	0.6
6	20	6	842.105	13.596	74	0.5
7	30	2.4	42.241	1.846	542	0.6
8	30	4.4	546.419	13.028	77	0.6
9	30	6	1642.036	28.711	35	0.6

Table 3-8: wear results of Ep/ graphite composites obtained under dry sliding conditions.

Experiment No.	Variable parameters		$q\ (mm^3/m)$ $\times 10^{-3}$	K $\times 10^{-3}$	R	μ
	f (vol.%)	N (kg)				
1	10	2.4	10.776	0.287	3487	0.5
2	10	4.4	788.177	11.441	87	0.7
3	10	6	2241.379	23.859	42	0.7
4	20	2.4	10.965	0.249	4018	0.5
5	20	4.4	555.556	6.879	145	0.6
6	20	6	1827.485	16.594	60	0.6
7	30	2.4	107.438	2.668	375	0.5
8	30	4.4	550.964	7.464	134	0.7
9	30	6	1700.572	16.895	59	0.6

3.5.1.1 *Effect of Particle Volume Fraction*

Figures 3-8 to 3-10 show the variation of wear characteristics with particle volume fraction under dry sliding conditions at loads of 2.4, 4.4, and 6 kg.

With changing particle content, epoxy/Al_2O_3 and epoxy/SiC composites exhibited a unique behavior under different loads. They showed a minimum wear rate at compositions of 30 vol. % and 20 vol. % respectively. Epoxy/graphite composites exhibited different behaviors at different loads. At load 2.4 kg, they showed a minimum wear rate at 10 vol. % of graphite. At higher loads, the minimum wear rate was exhibited at 30 vol. % of graphite. This may be referred to the change in wear mechanism from adhesive to abrasive.
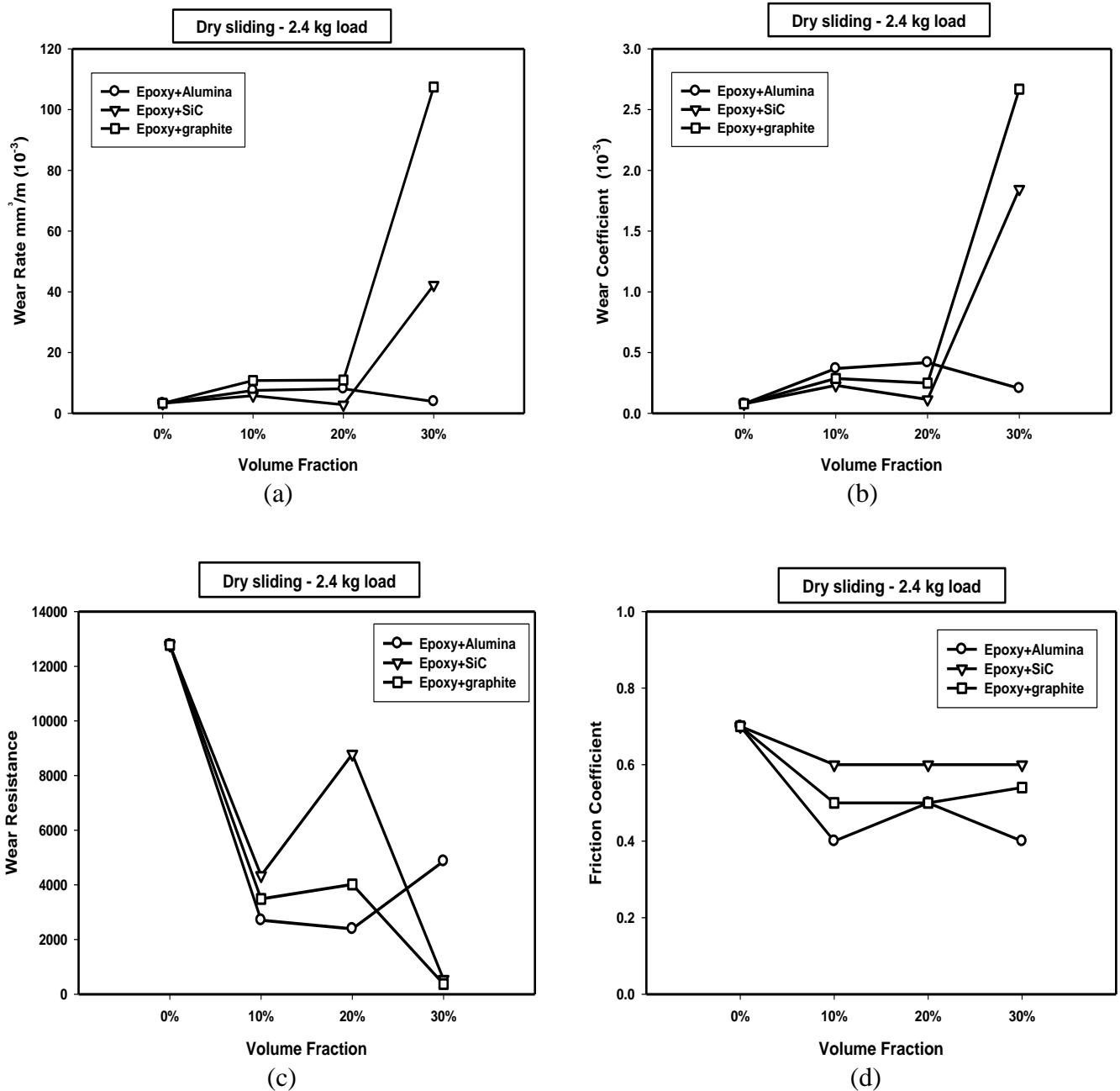
Figure 3-8: wear characteristics of composites under dry sliding conditions and 2.4 kg load.

As mentioned before, wear coefficient is directly proportional to wear rate and hardness of the material. Thus, behavior of wear coefficient is the same as that of wear rate but with different scale due to the difference in hardness. For example, epoxy/Al_2O_3 composites with particle content up to 20 vol.% exhibited a wear rate lower than that of epoxy/graphite composites. On the other hand, epoxy/Al_2O_3 composites having hardness higher than that of epoxy/graphite composites, exhibited

a higher wear coefficient. Generally, epoxy/Al$_2$O$_3$ composites containing 30 vol.% of particulates exhibited the lowest wear rate and wear coefficient.
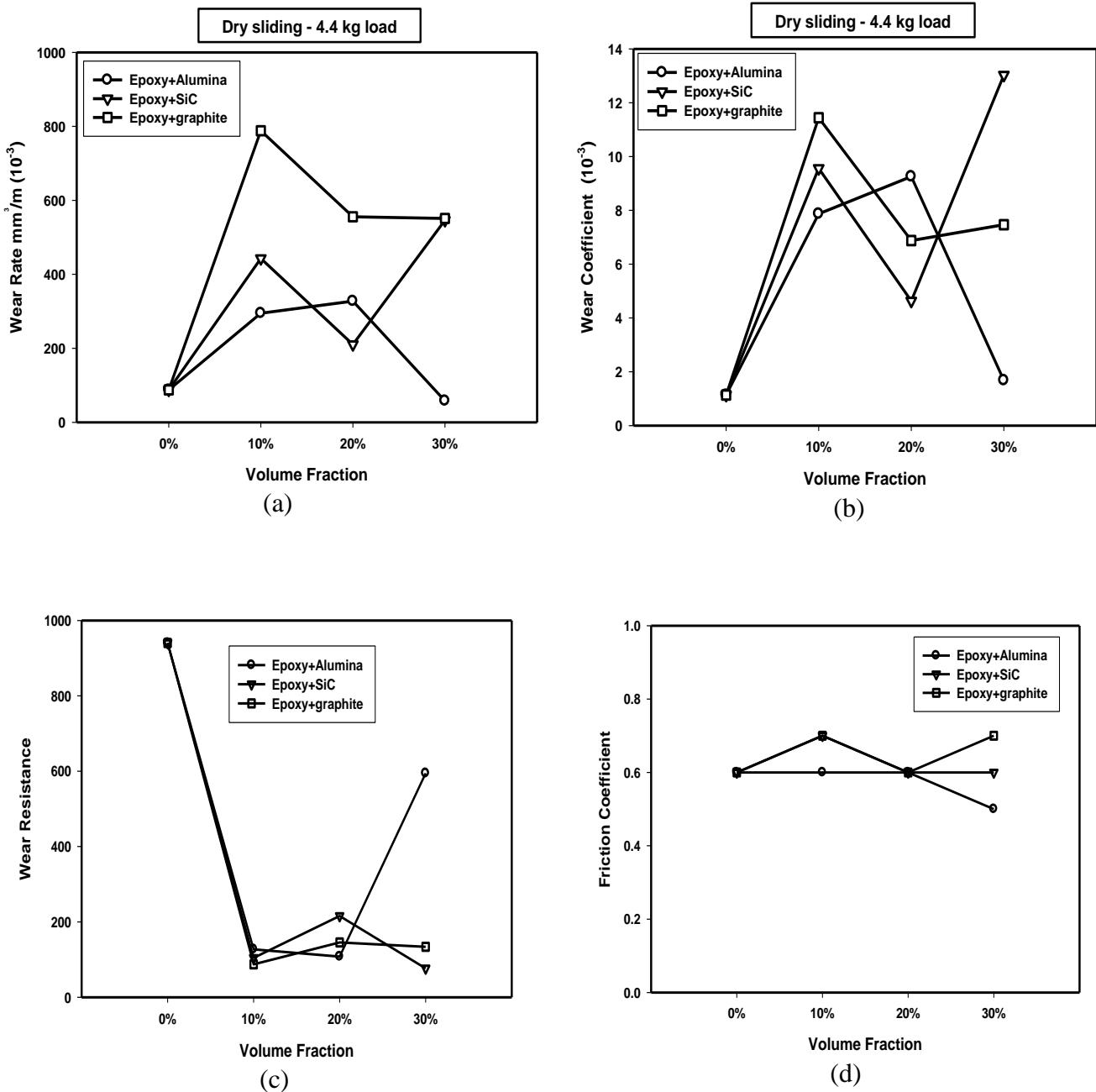
Figure 3-9: wear characteristics of composites under dry sliding conditions and 4.4 kg load.

Wear resistance is the reciprocal of wear coefficient. Generally, wear resistance of epoxy/particulates composites under dry sliding was lower than that of the base matrix. At load 2.4 kg, epoxy/SiC composites containing 20 vol.% SiC exhibited the highest wear resistance. At higher loads, epoxy/ Al$_2$O$_3$ composites containing 30 vol.% Al$_2$O$_3$ exhibited the highest wear resistance.

Values of friction coefficient were ranged from 0.4 to 0.7. Epoxy/Al_2O_3 composites showed the lowest coefficient of friction
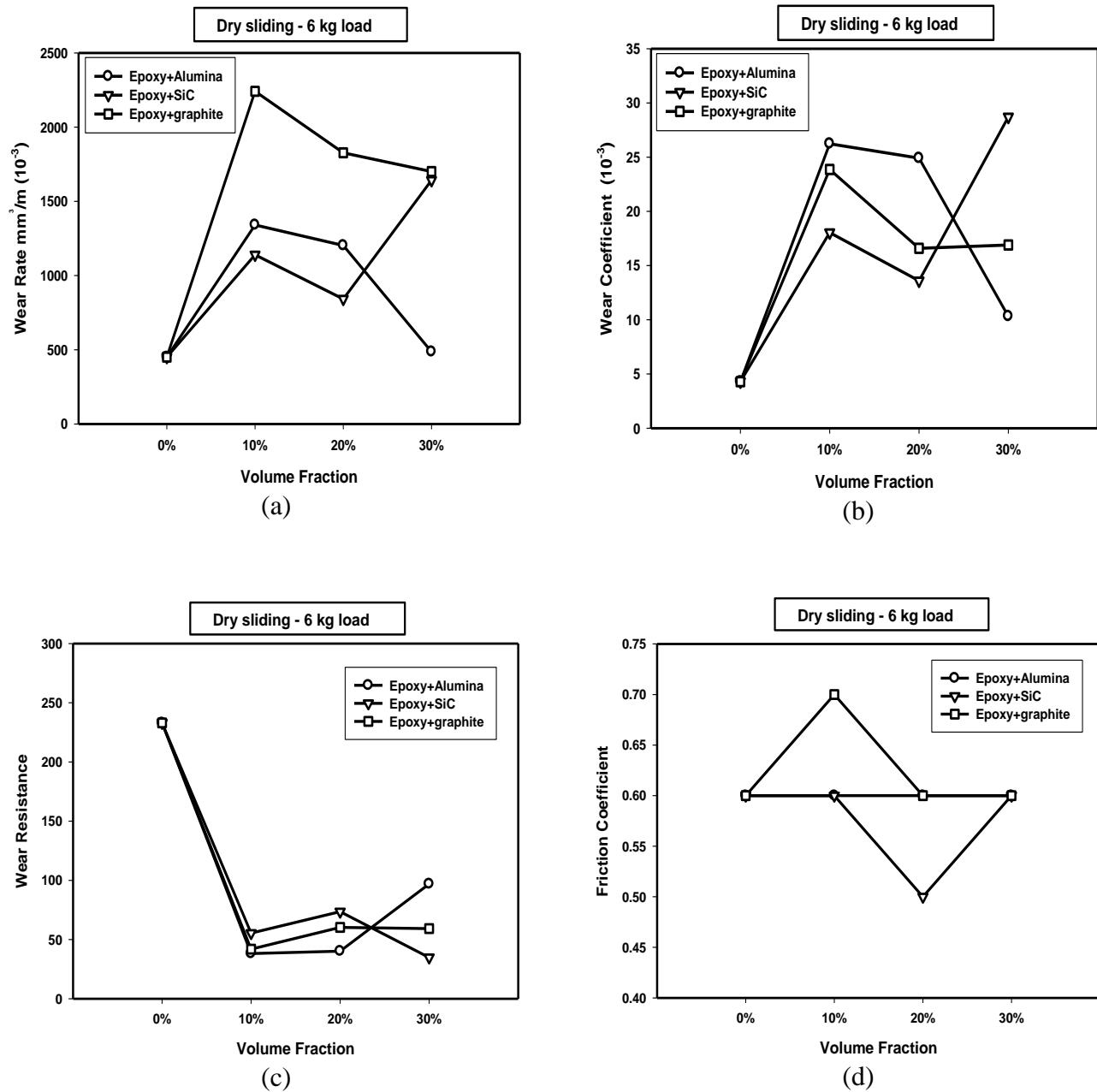
Figure 3-10: wear characteristics of composites under dry sliding conditions and 6 kg load.

The increase in wear rate of composites with particle content was reported by Bernd Wetzel *et al.* [45]. This phenomenon can be emphasized as the occurrence of change in wear mechanism due to the large amount of hard particles causing a higher abrasive wear now.

3.5.1.2 *Effect of Normal Load*

Generally as the load increases, wear rate and wear coefficient of composites at any composition increases. This is due to the effect of load on breaking cohesiveness between particulates and the base matrix. Figures 3-11 to 3-13 show the effect of load on wear characteristics of composites under dry sliding conditions.
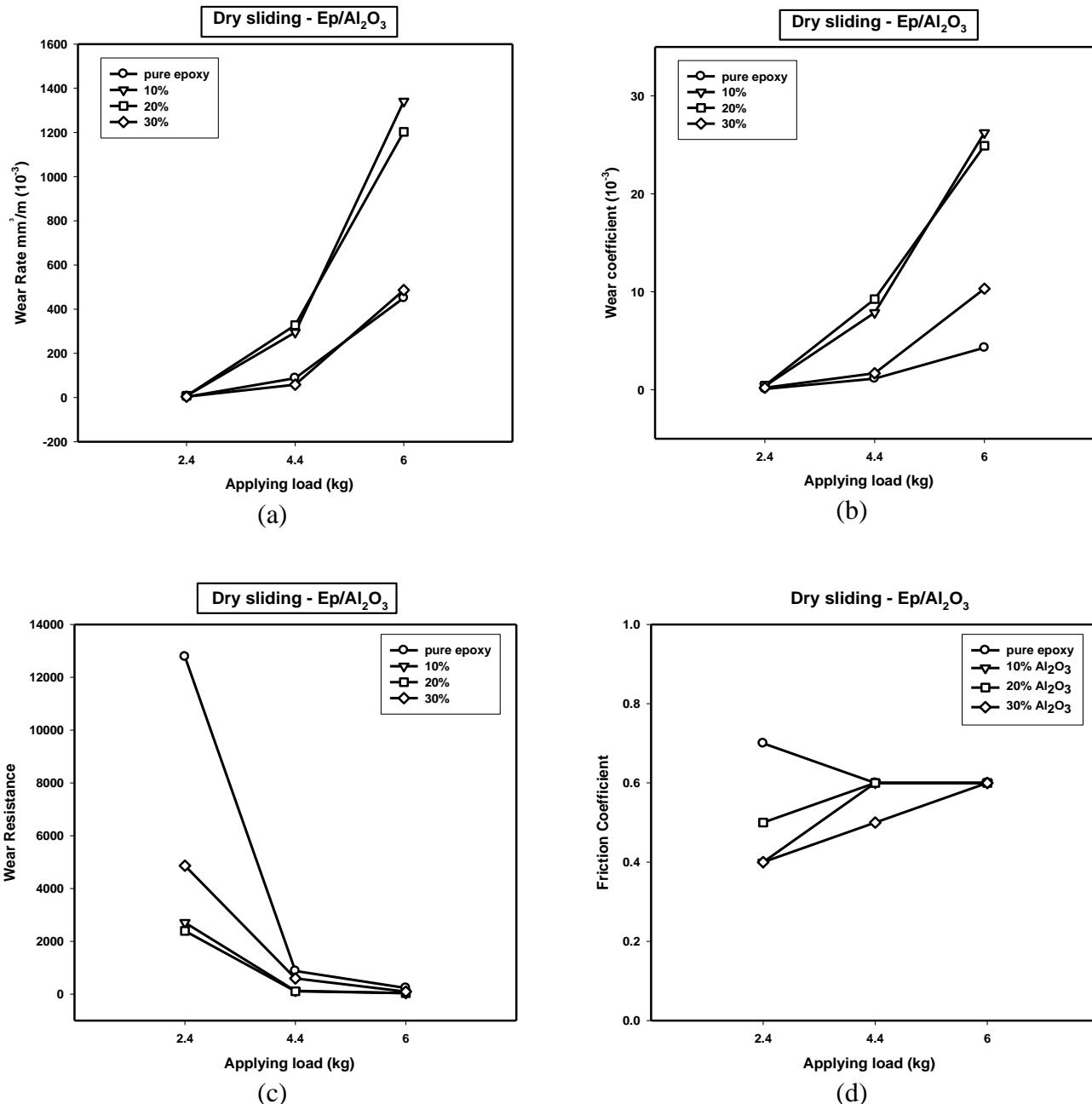
Figure 3-11: wear characteristics of epoxy- Al_2O_3 composites under dry sliding conditions.
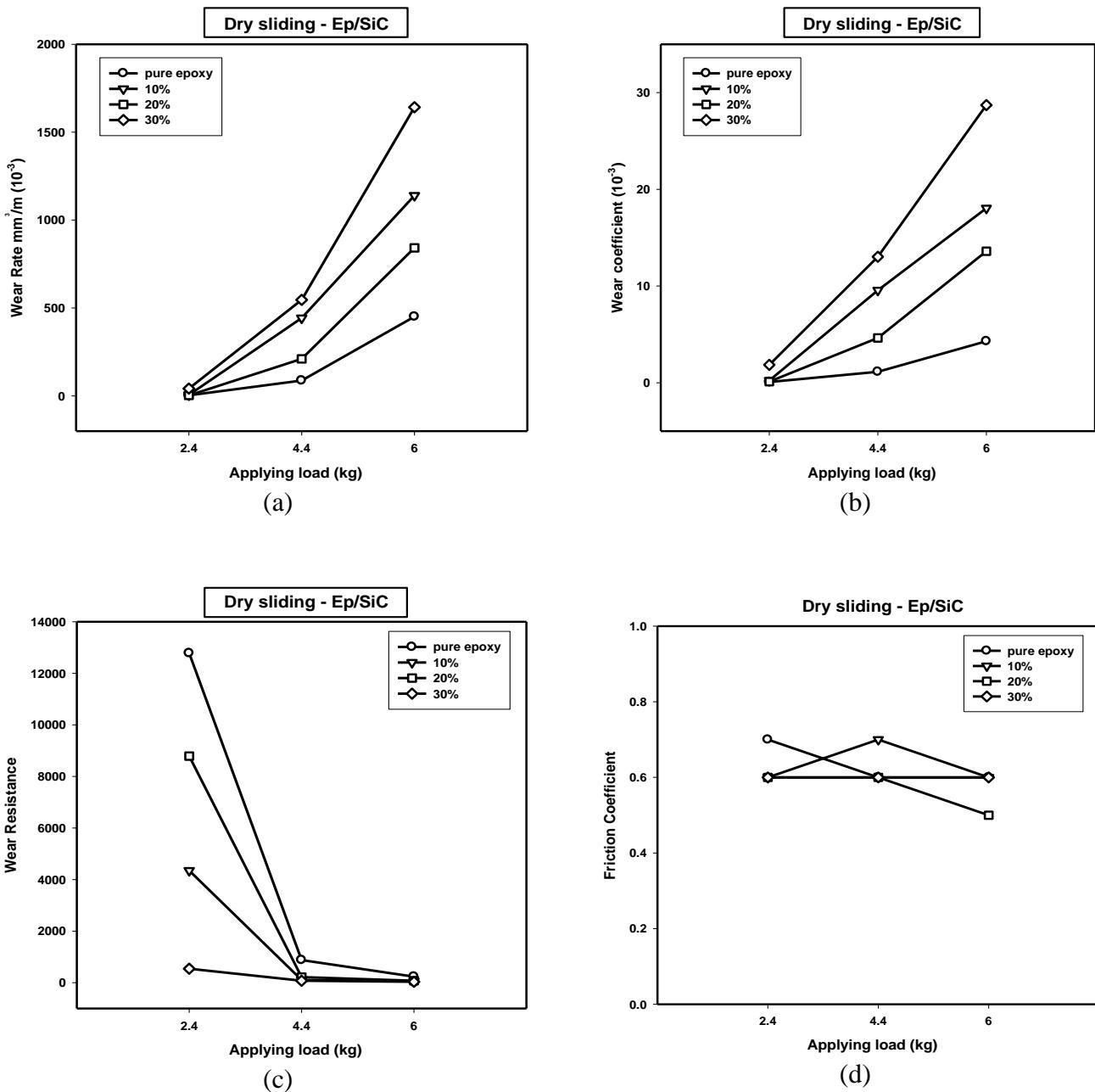
Figure 3-12: wear characteristics of epoxy-SiC composites under dry sliding conditions.
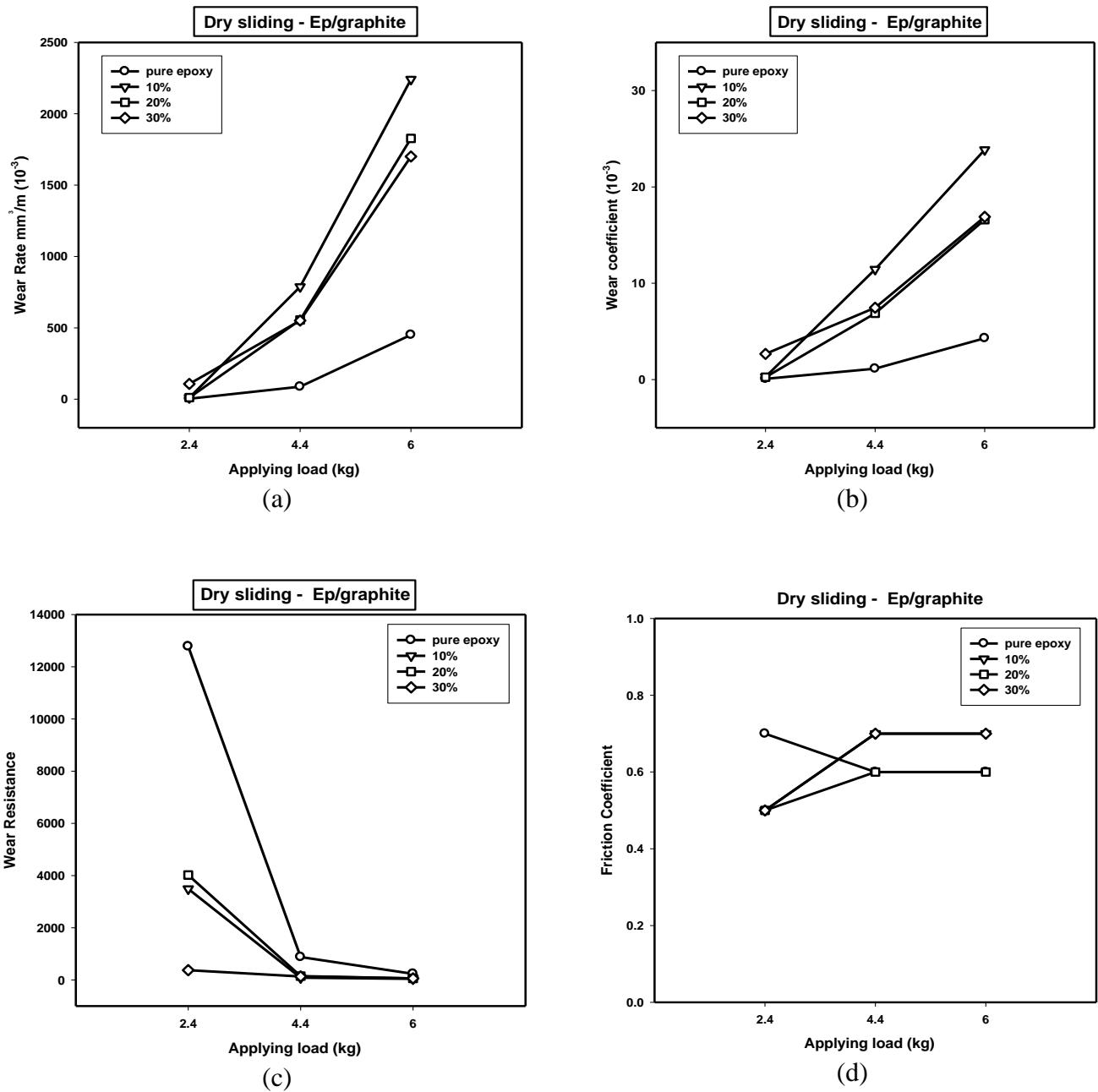
Figure 3-13: wear characteristics of epoxy-graphite composites under dry sliding conditions.

Bassani *et al.* [47] had shown that wear rate of epoxy composites increases with the applied pressure. Under dry sliding conditions, as load increases, the wear mechanism was noticed to change from adhesive to abrasive due to the presence of particles and fragmented debris between the mating surfaces, and thus, wear rate noticeably increases.

3.5.1.3   Effect of Particle Type

Under dry sliding conditions and 2.4 kg normal load, for compositions up to 20 vol. % of particulates, epoxy/SiC composites showed the lowest wear rate and wear coefficient followed by epoxy/ Al_2O_3 composites (Figure 3-14). This phenomenon can be emphasized by recognition of cohesiveness between particles and polymeric chains of the base matrix. Also the increased hardness of these composites has a great effect on decreasing wear rate.

(a)   (b)

(c)   (d)
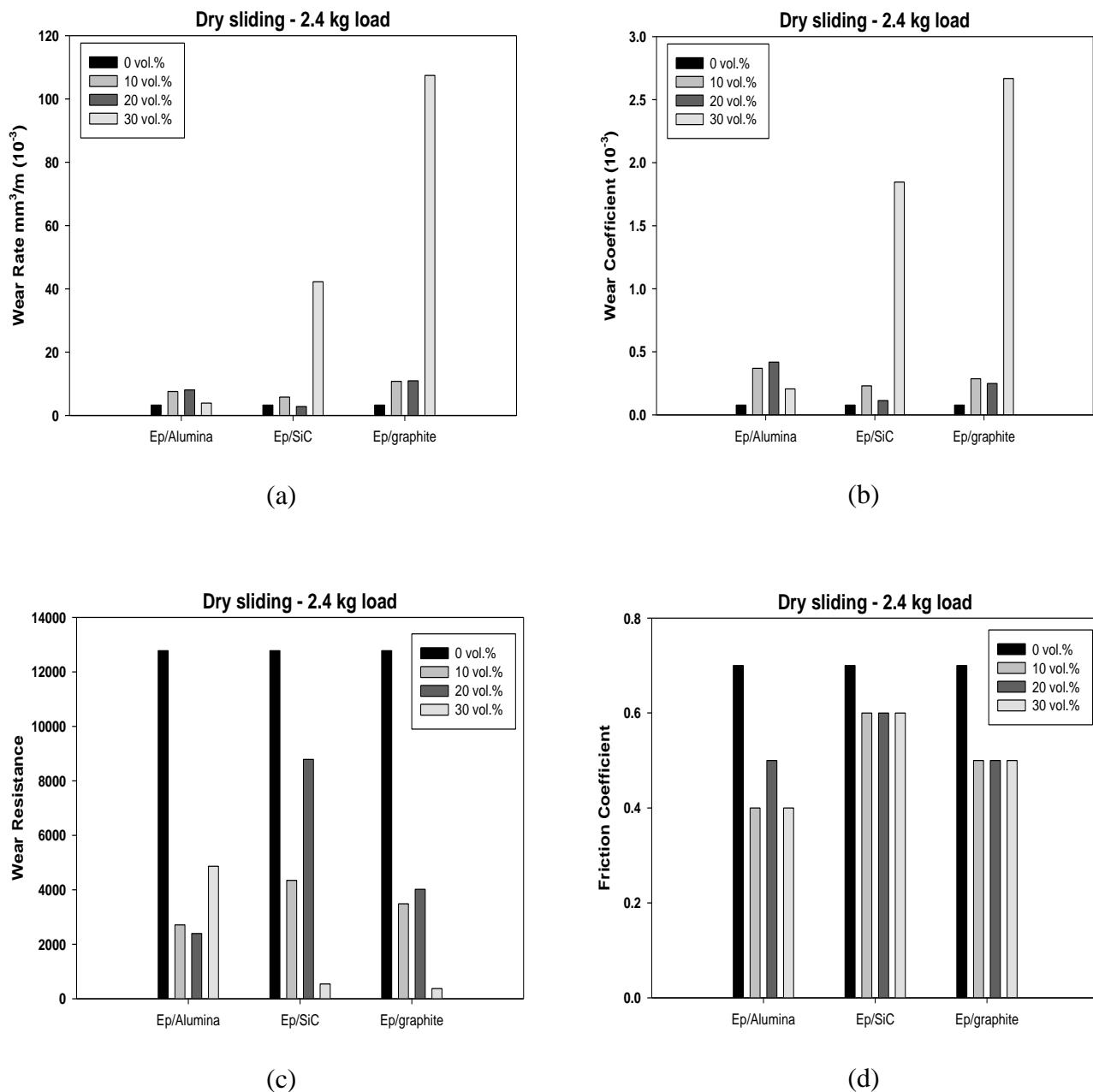
Figure 3-14: wear characteristics versus particle type under dry sliding conditions and 2.4 kg load

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For epoxy/30 vol. % of graphite particulates, under load of 2.4 kg, wear rate increased about 20 times higher than that of the base matrix due to the decrease in its hardness, and for 30 vol. % of SiC particulates, wear rate increased about 8 times higher than that of the base matrix due to the increase in the abrasive effect of particles.
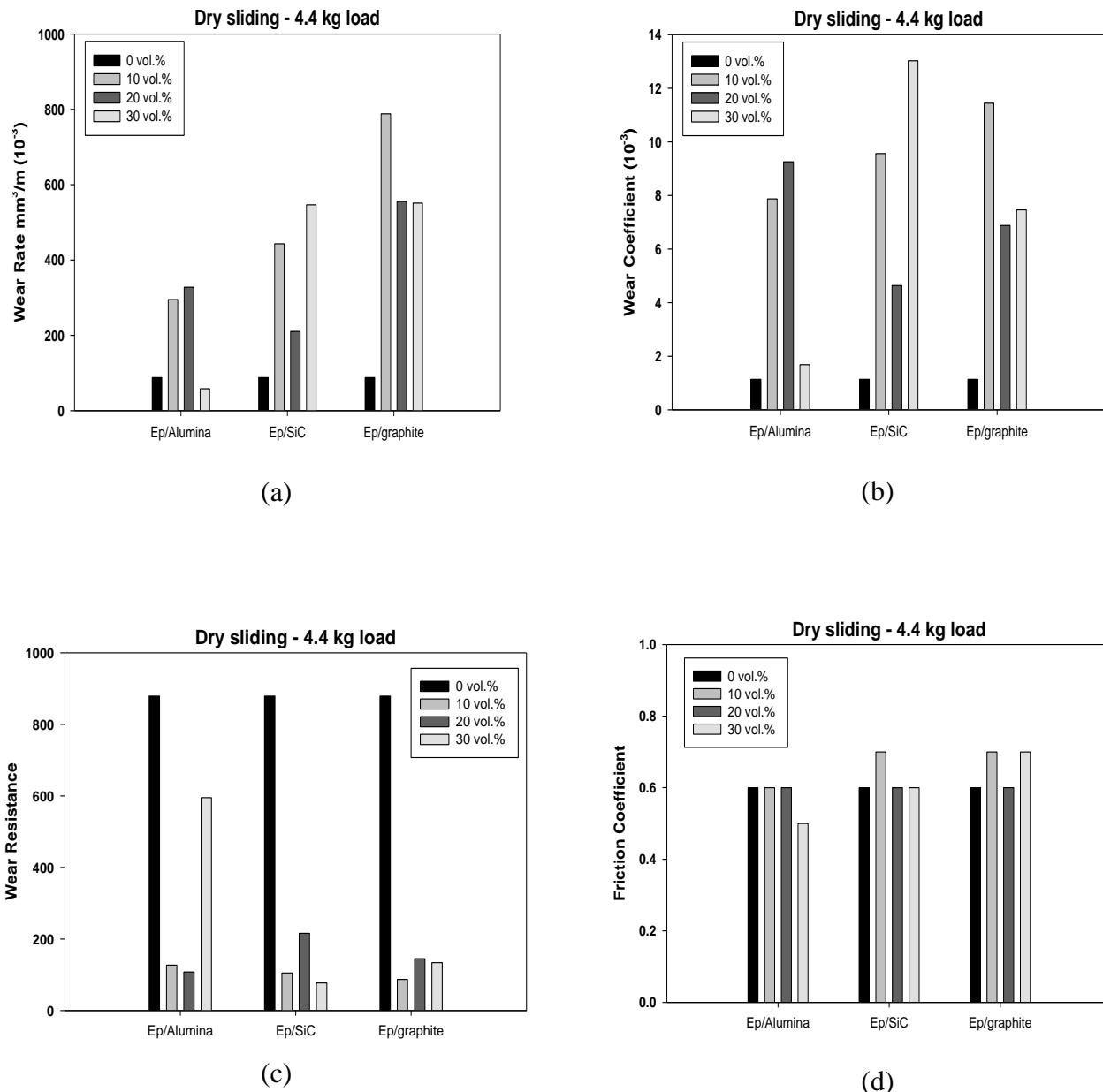
Figure 3-15: wear characteristics versus particle type under dry sliding conditions and 4.4 kg load

At higher loads, epoxy/Al$_2$O$_3$ composites generally showed a lower wear rate (Figures 3-15 and 16). The improvement of wear rate of epoxy/Al$_2$O$_3$ composites

may be attributed to the increase in hardness of these composites. As the hardness of epoxy/graphite composites is the lowest, their wear rate is the highest.

(a)

(b)

(c)

(d)
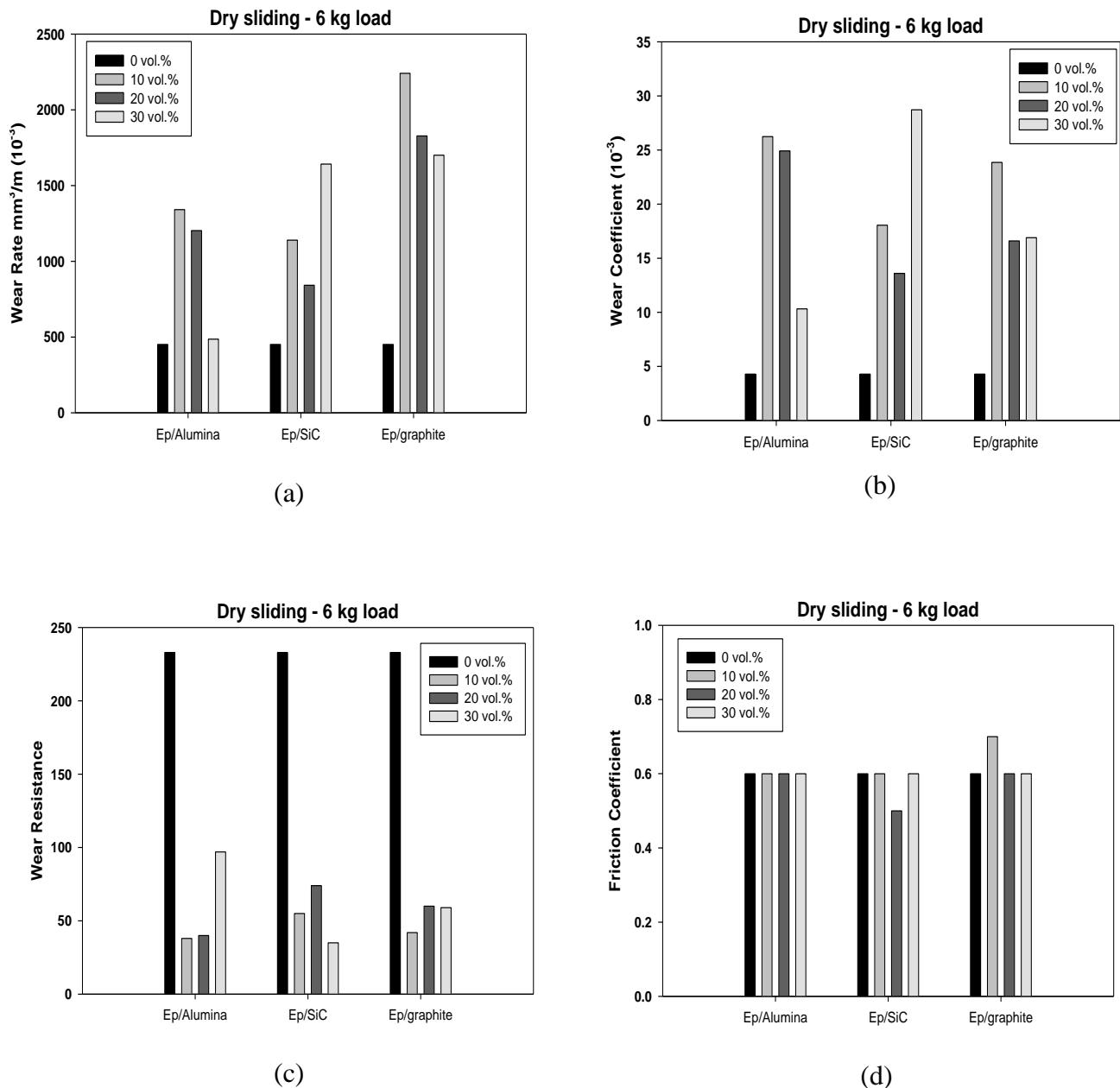
Figure 3-16: wear characteristics versus particle type under dry sliding conditions and 6 kg load

Zhang *et al.* [1] compare the independent effect of many types of filler on the wear resistance of epoxy matrix composites. Their results showed that both graphite and PTFE reinforced epoxy can be considered as 'soft' phases dispersed into a 'hard' phase generating a thin film to reduce the friction between the composite and the counterpart

3.5.2 Tribological Characteristics Under Lubricated Sliding Conditions

Tables 3-9 to 3-12 list the average values of q, K, R, and μ obtained under water lubricated sliding conditions.

Table 3-9: wear results of Epoxy matrix obtained under water lubricated sliding conditions.

Experiment No.	Variable parameters		q (mm^3/m) ×10^{-6}	K ×10^{-6}	R ×10^6	μ
	f (vol.%)	N (kg)				
1	0	20	61	0.174	5.751	0.08
2	0	22	146	0.379	2.636	0.10
3	0	24	292	0.696	1.438	0.07

Table 3-10: wear results of Ep/Al$_2$O$_3$ composites obtained under water lubricated sliding conditions.

Experiment No.	Variable parameters		q (mm^3/m) ×10^{-6}	K ×10^{-6}	R ×10^6	μ
	f (vol.%)	N (kg)				
1	10	20	30	0.176	5.692	0.06
2	10	22	66	0.354	2.826	0.07
3	10	24	129	0.632	1.581	0.08
4	20	20	56	0.348	2.873	0.05
5	20	22	161	0.911	1.097	0.06
6	20	24	302	1.566	0.639	0.06
7	30	20	54	0.343	2.919	0.06
8	30	22	155	0.897	1.115	0.07
9	30	24	291	1.542	0.649	0.08

Table 3-11: wear results of Ep/SiC composites obtained under water lubricated sliding conditions.

Experiment No.	Variable parameters		q (mm^3/m) ×10^{-6}	K ×10^{-6}	R ×10^6	μ
	f (vol.%)	N (kg)				
1	10	20	135	0.639	1.565	0.05
2	10	22	352	1.521	0.657	0.06
3	10	24	646	2.557	0.391	0.07
4	20	20	157	0.759	1.318	0.05
5	20	22	451	1.986	0.503	0.06
6	20	24	846	3.414	0.293	0.06
7	30	20	718	3.768	0.265	0.09
8	30	22	1724	8.222	0.122	0.11
9	30	24	3448	15.073	0.066	0.10

Table 3-12: wear results of Ep/ graphite composites obtained under lubricated sliding conditions.

Experiment No.	Variable parameters		q (mm^3/m) $\times 10^{-6}$	K $\times 10^{-6}$	R $\times 10^6$	μ
	f (vol.%)	N (kg)				
1	10	20	60	0.191	5.231	0.05
2	10	22	144	0.417	2.397	0.07
3	10	24	287	0.765	1.308	0.07
4	20	20	122	0.332	3.013	0.08
5	20	22	281	0.695	1.439	0.09
6	20	24	554	1.258	0.795	0.09
7	30	20	172	0.513	1.949	0.06
8	30	22	381	1.034	0.968	0.07
9	30	24	744	1.847	0.541	0.08

3.5.2.1 *Effect of Particle Volume Fraction*

It was noticed, under water lubricated sliding conditions, epoxy/particulates composites exhibited a unique behaviour under different loads (Figures 3-17 to 3-19). For epoxy/Al$_2$O$_3$ composites, wear rate and wear coefficient were not significantly affected by Al$_2$O$_3$ content in the material. For epoxy/graphite composites, wear rate was low proportional to particle content. For epoxy/SiC composites, wear rate and wear coefficient showed a significant increase at 30 vol. % of particulates.

Wear coefficient of epoxy/Al$_2$O$_3$ and epoxy/graphite composites were equal and lower than that of epoxy/SiC composites. Epoxy/Al$_2$O$_3$ composites containing 10 vol.% of particulates exhibited the highest wear resistance which is higher than that of the base matrix. Values of friction coefficient were ranged from 0.05 to 0.11.
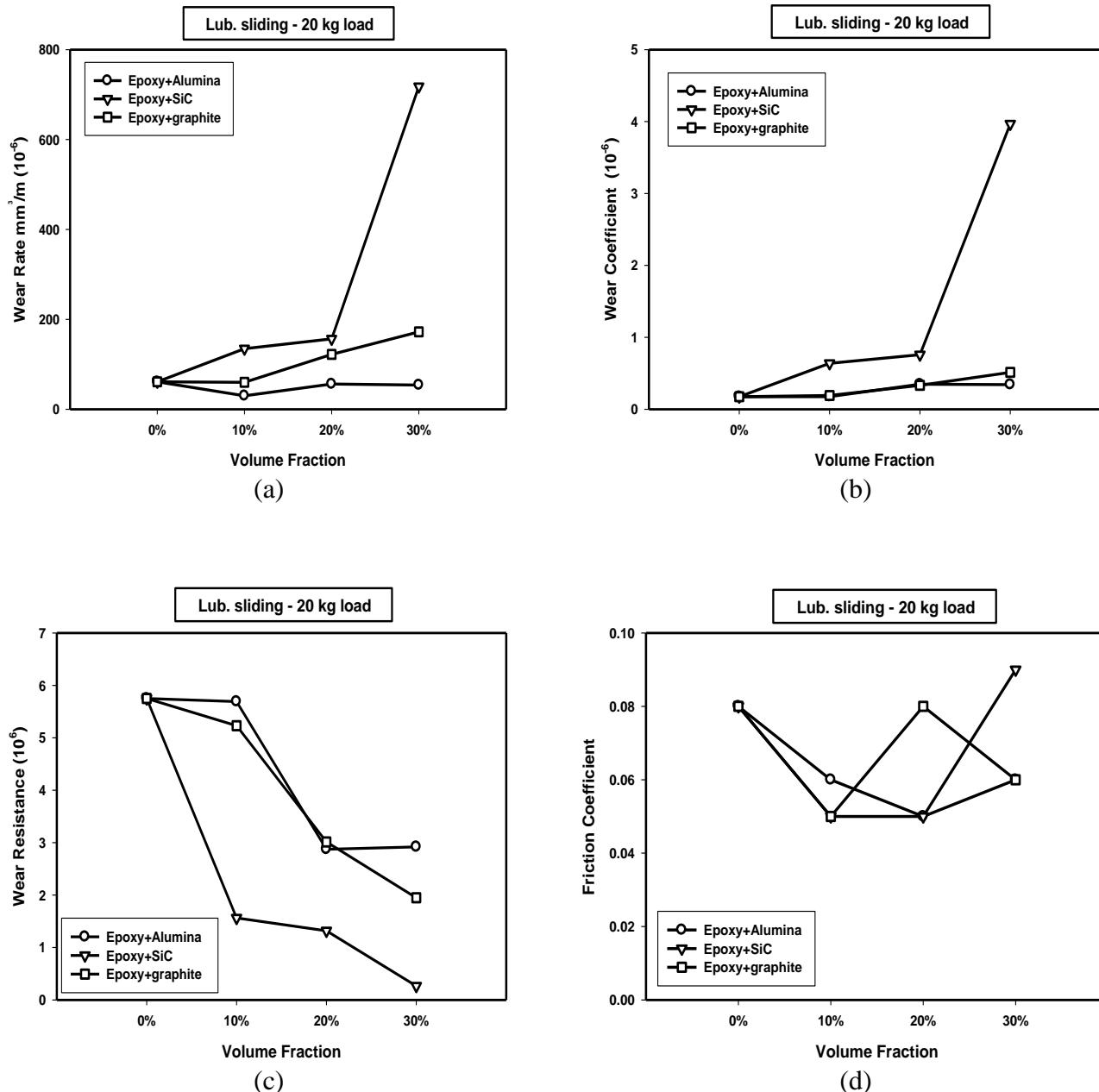
Figure 3-17: wear characteristics of composites under water lubricated sliding conditions and 20 kg load.
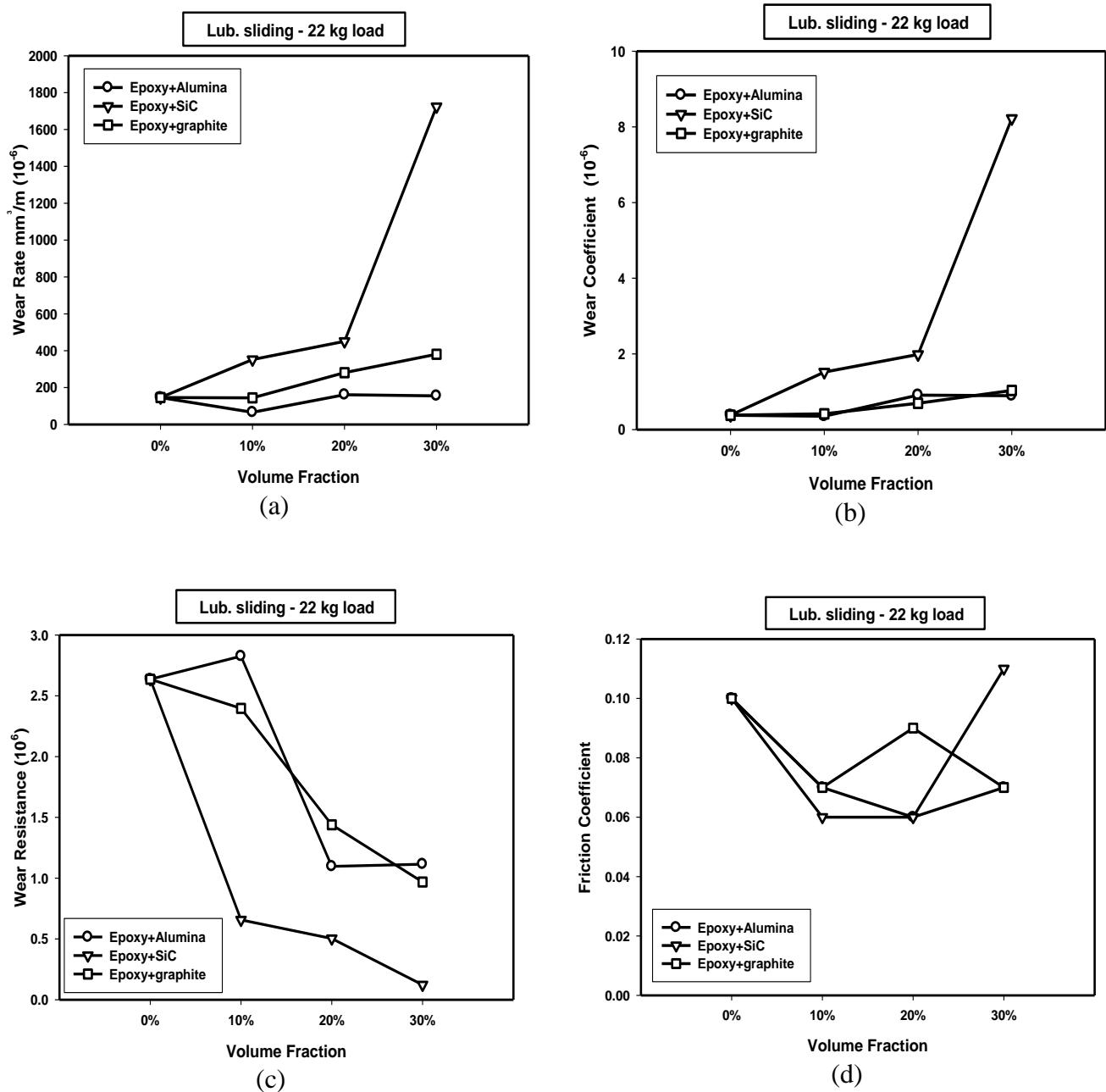
Figure 3-18: wear characteristics of composites under water lubricated sliding conditions and 22 kg load.
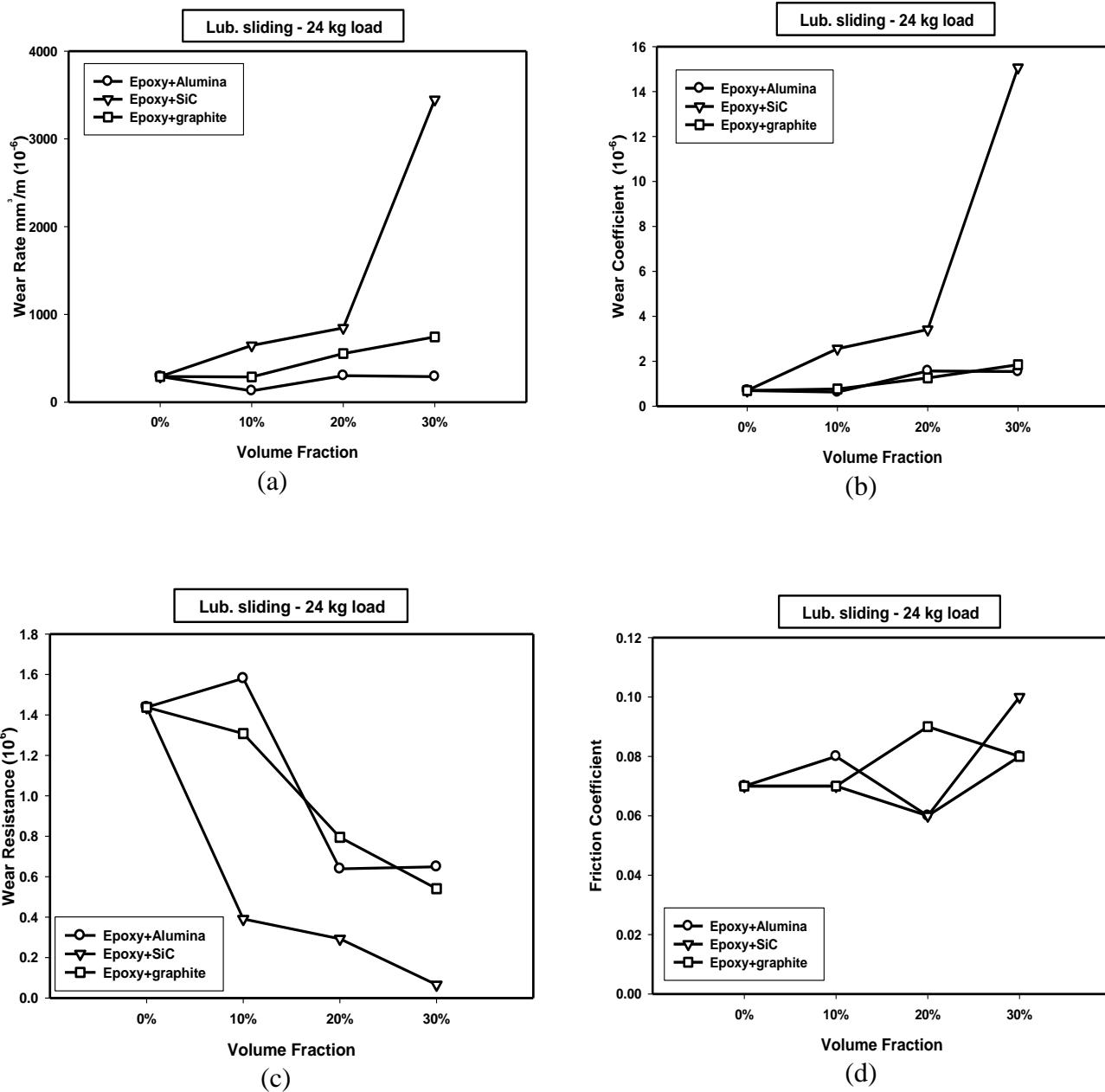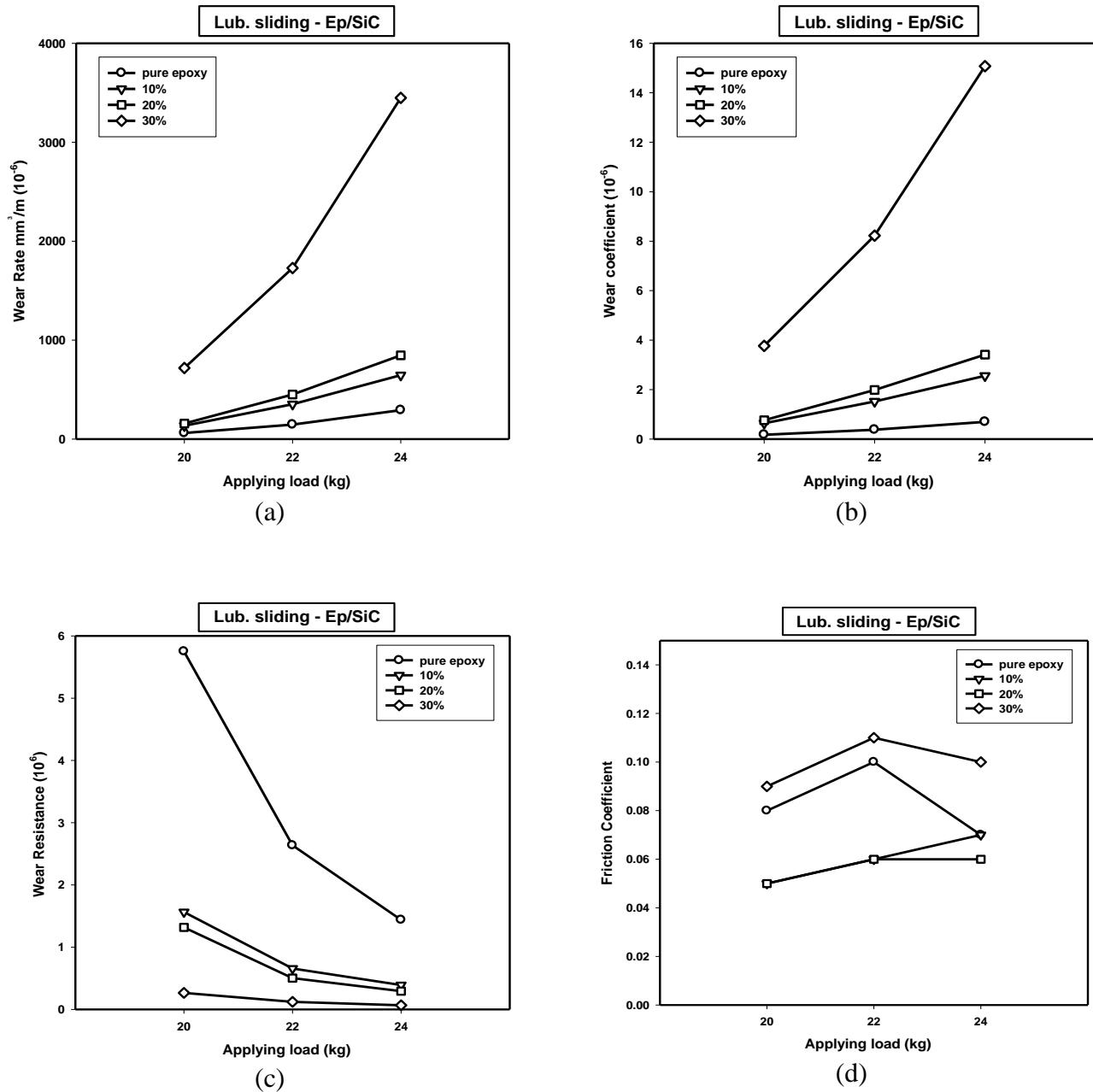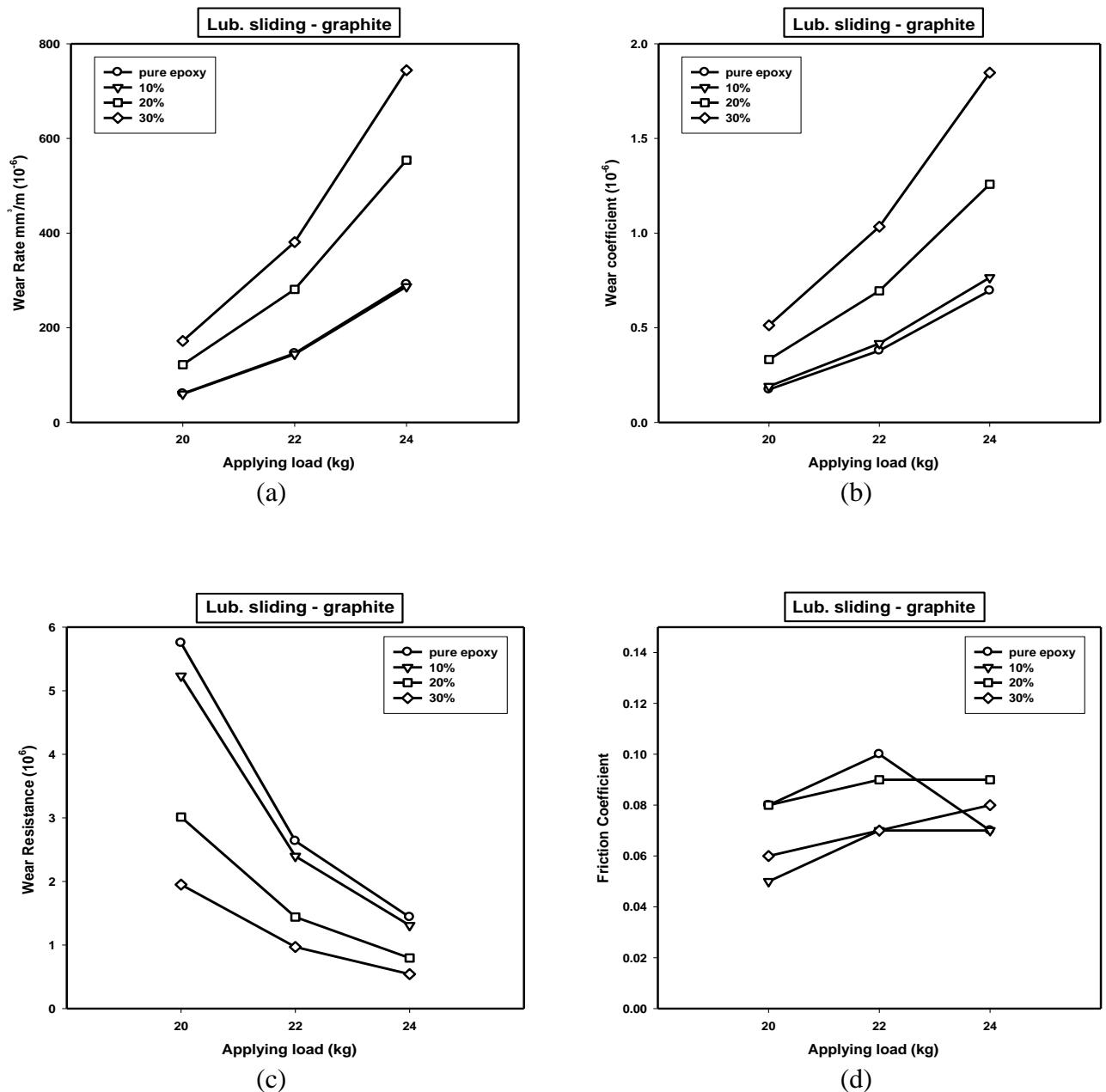
Figure 3-19: wear characteristics of composites under water lubricated sliding conditions and 24 kg load.

The increase in wear rate of epoxy/SiC composites with increasing SiC content was reported by Prehn *et al.* [50]. This behaviour can be referred to the abrasive effect of SiC particles on the steel counterpart. As the counterpart surface is abraded then it is cutting into the composite material and increasing wear rate.

3.5.2.2 *Effect of Normal Load*

Figures 3-20 to 3-22 show the effect of load on wear characteristics of composites under water lubricated sliding conditions. It was noticed that, as the load increased, the pressure on the specimen surface increased, and consequently, wear rate increased. Epoxy/10 vol. % Al_2O_3 composites showed a wear rate lower than that of the base matrix.
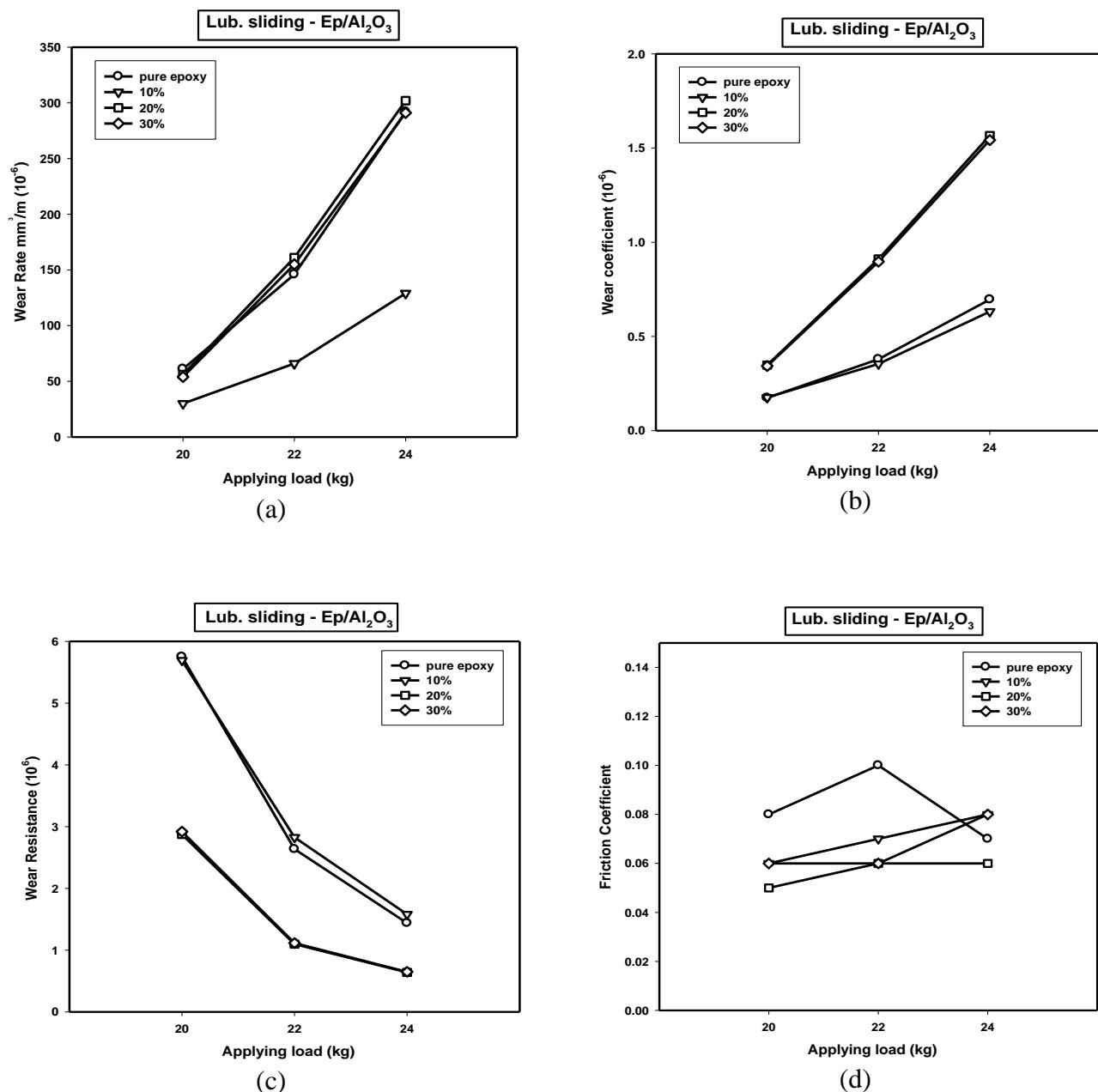
Figure 3-20: wear characteristics of epoxy- Al_2O_3 composites under water lubricated sliding conditions.

Figure 3-21: wear characteristics of epoxy-SiC composites under water lubricated sliding conditions.

Figure 3-22: wear characteristics of epoxy-graphite composites under water lubricated sliding conditions.

3.5.2.3 Effect of Particle Type

Under water lubricated sliding conditions, epoxy/Al_2O_3 composites showed the lowest wear rate among materials under consideration. While composites of epoxy/30 vol. % SiC showed the highest wear rate.
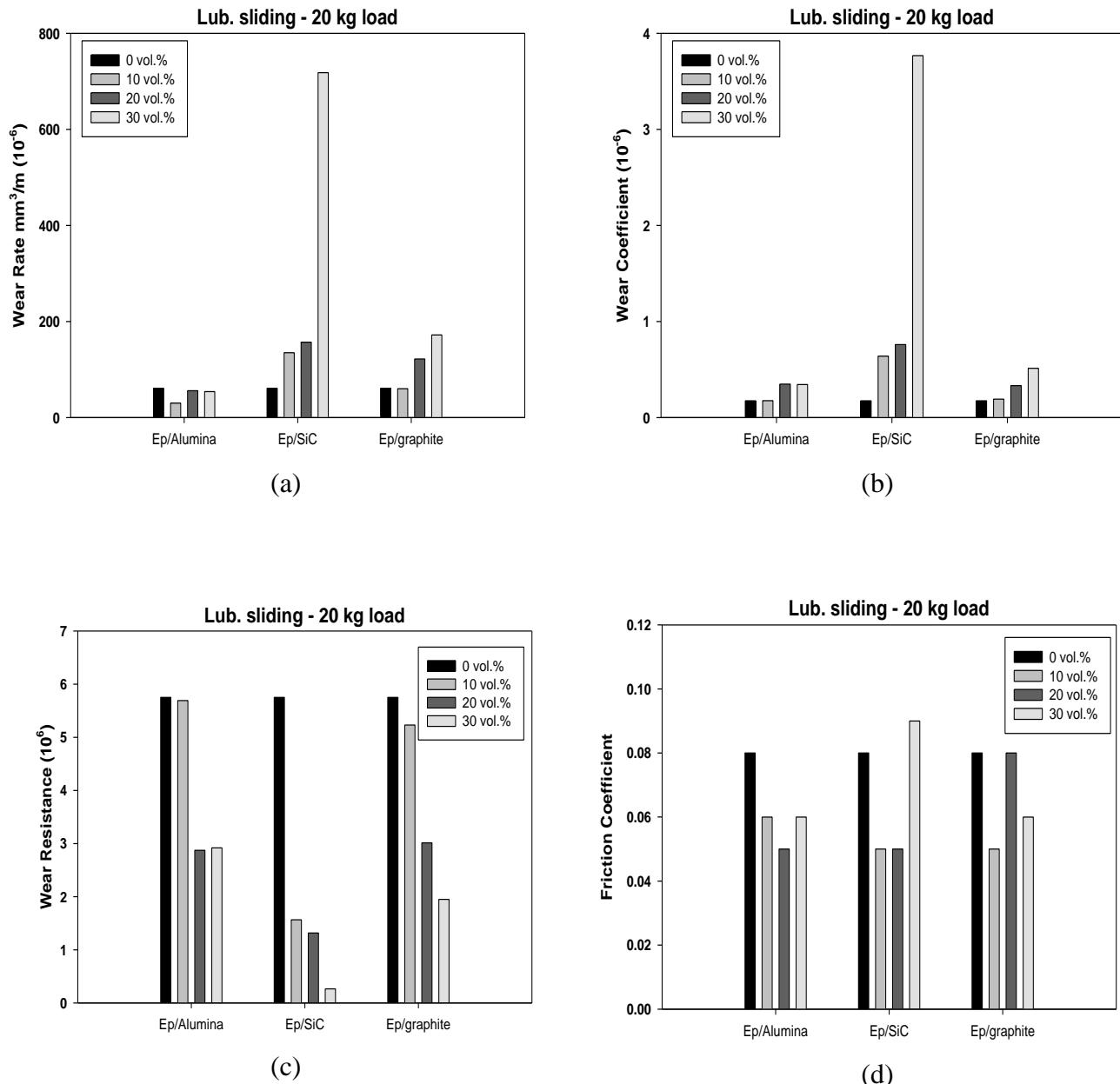
Figure 3-23: wear characteristics versus particle type under wet sliding conditions and 20 kg load

(a)

(b)

(c)

(d)
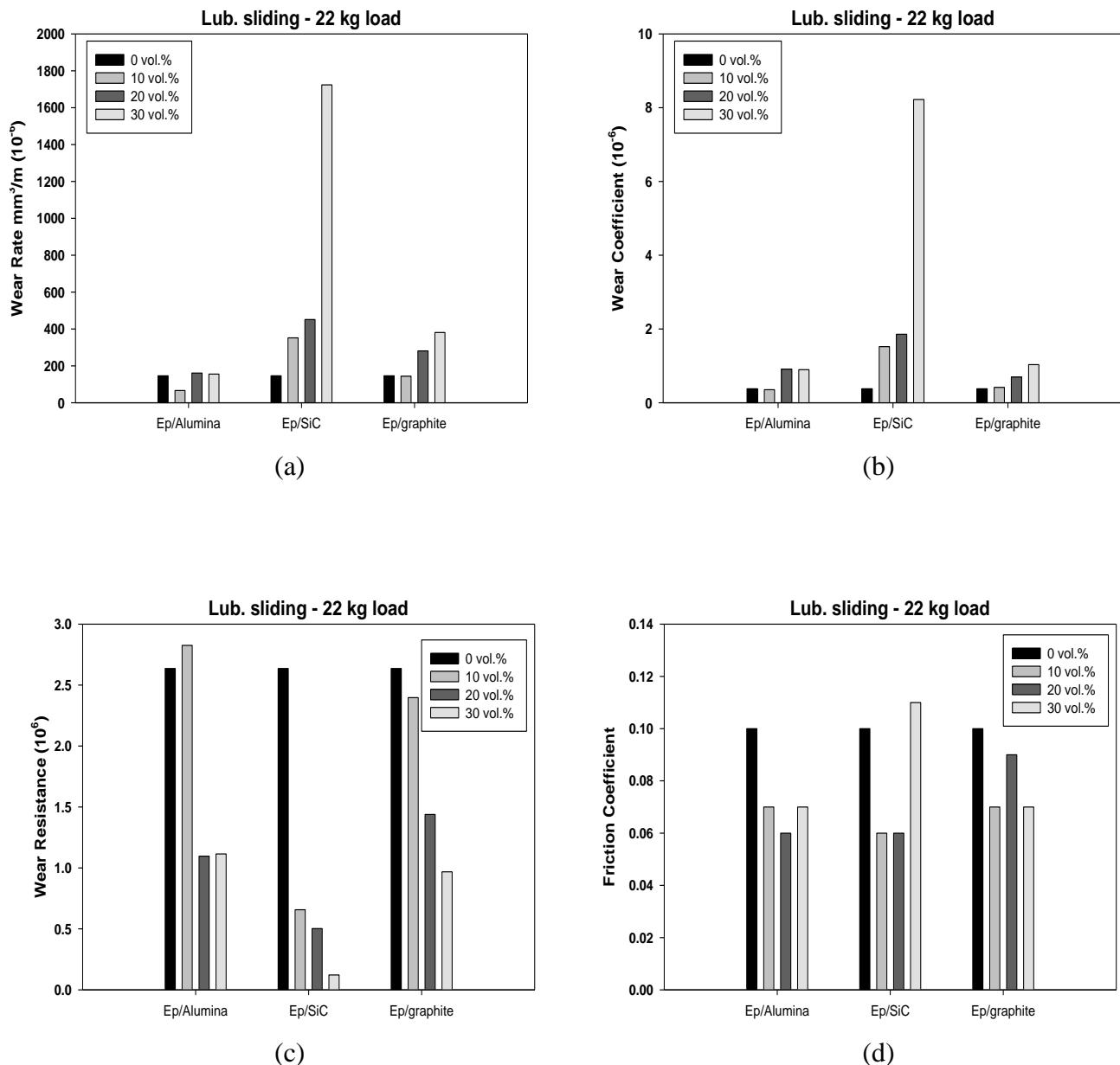
Figure 3-24: wear characteristics versus particle type under wet sliding conditions and 22 kg load

76
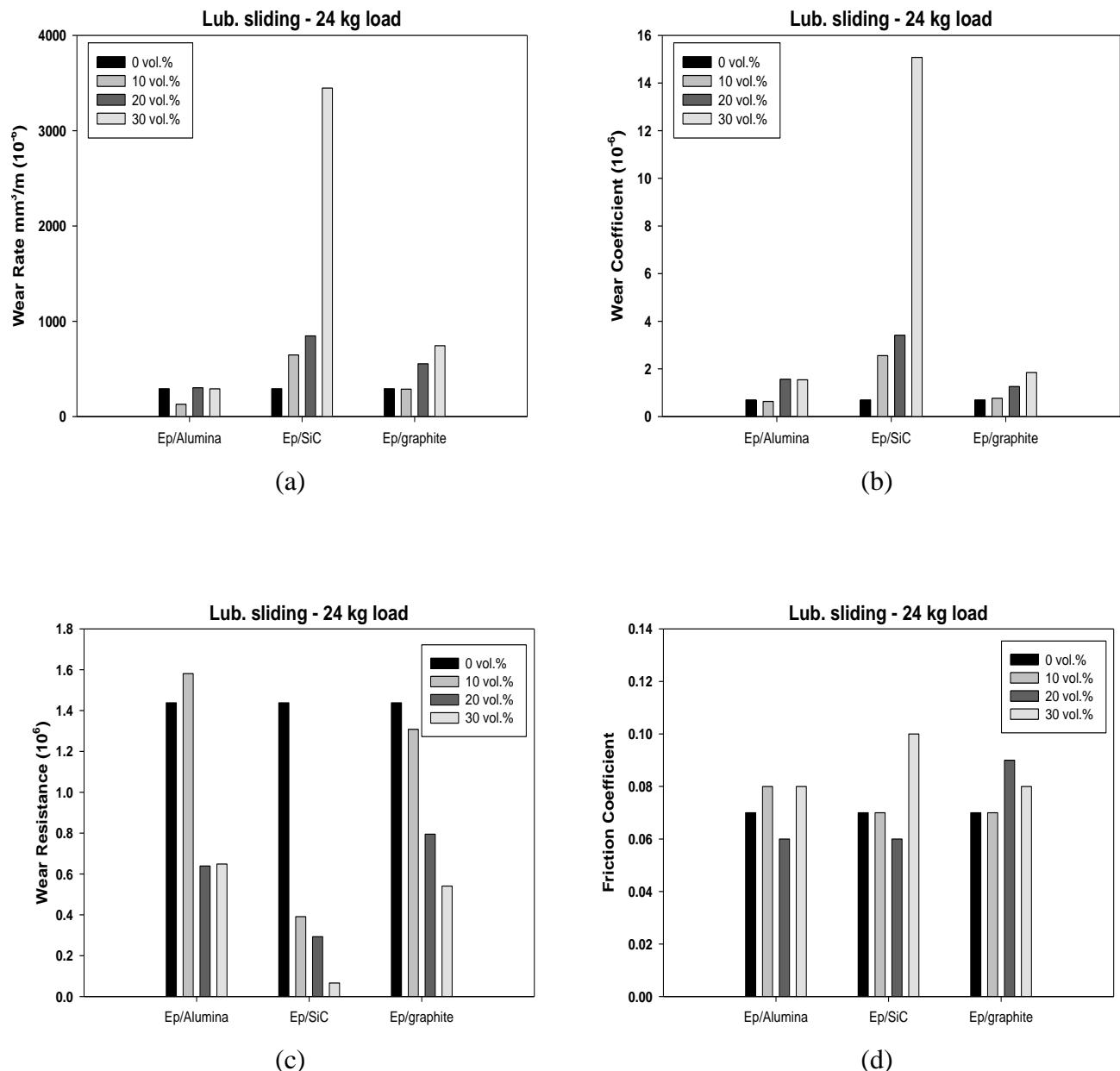
Figure 3-25: wear characteristics versus particle type under wet sliding conditions and 24 kg load

Comparing results of wear rates of composites under water lubricated sliding conditions with those under dry sliding conditions, it can be concluded that water lubrication dramatically reduces wear rate. As a result of heat generated in case of dry sliding conditions, surface asperities of epoxy based materials were plastically deformed. And thus, frictional force and wear rate increased. However, under water lubricated sliding conditions, the formation of a water film between mating surfaces can reduce the direct contact area and thus reducing frictional force. Also most of heat generated during rubbing is dissipated by water and thus there is no considerable plastic deformation and wear rate is decreased.

Prehn *et al.* [50] studied the sliding wear performance of polymeric composites under abrasive and water lubricated sliding conditions. Their results indicated that increasing particle size and content has a negative influence on wear under dry sliding conditions but a positive effect under lubricated sliding conditions. Wu and Cheng have studied the tribological properties of Kevlar pulp reinforced epoxy composites under dry and water lubricated sliding conditions [51]. They showed that wear rate of composites under water lubricated sliding conditions is five order of magnitude lower than that under dry sliding conditions.

The variability of the obtained results under dry and lubricated sliding conditions can be referred to the divergence in wear mechanism. Under dry sliding conditions, the main wear mechanism is adhesive/abrasive depending on load and particle content. But under water lubricated conditions, the main wear mechanism is erosive.

3.6 ANALYSIS OF VARIANCE (ANOVA) AND REGRESSION EQUATIONS OF WEAR RATE RESULTS:

Figure 3-26 shows the ANOVA results of wear rate of epoxy/Al_2O_3 composites under dry sliding conditions.

```
General Linear Model: Ep/Al2O3 wear rate versus particle volume fraction and load
under dry sliding conditions

Factor              Type    Levels  Values
volume fraction     fixed        3  10, 20, 30
load                fixed        3  2.4, 4.4, 6.0

Analysis of Variance for Ep/Al2O3 wear rate, using Adjusted SS for Tests

Source                 DF    Seq SS    Adj SS    Adj MS       F      P
volume fraction         2    739697    739697    369849   34.06  0.000
load                    2   4993162   4993162   2496581  229.89  0.000
volume fraction*load    4    657713    657713    164428   15.14  0.000
Error                  18    195476    195476     10860
Total                  26   6586048

S = 104.210   R-Sq = 97.03%   R-Sq(adj) = 95.71%

Unusual Observations for Alumina wear rate

       Ep/Al2O3
Obs   wear rate      Fit  SE Fit  Residual  St Resid
 15      919.64  1202.60   60.17   -282.97     -3.33 R
 24     1414.83  1202.60   60.17    212.22      2.49 R

R denotes an observation with a large standardized residual.
```

Figure 3-26: ANOVA results of epoxy/Al$_2$O$_3$ wear rate under dry sliding conditions

Where;

DF: degree of freedom (number of levels of a factor -1),

Seq. SS: sequential sum of squares depends on the order of factors,

Adj. SS: adjusted sum of squares dose not depend on the order of factors,

Adj. MS: adjusted mean of squares (Adj. SS / DF),

F: F-test determines the significance of a factor (Adj MS of a factor /Adj MS of error). The higher the F value, the higher the significance of the factor,

P: probability of insignificance,

S: standard deviation (S^2 = Adj. MS of error),

R-Sq: R^2 is a coefficient indicates how much variation in the response is explained by the model. R^2=1-(SS of error/ total SS). The higher the R^2, the better the model fits data,

R-Sq(adj.): adjusted R^2 accounts for the number of factors in the model. R^2 adj. =1-(MS of error / (total SS * total DF)),

Fit: predicted value from the model,

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SE Fit: standard error of the fitted value,

Residual: the difference between the observed value and the predicted or fitted value, and

St Resid.: the standardized residual which is the residual scaled by its standard deviation.

It was noticed that all factors have a strong effect on wear rate (p value is lower than 0.05). F-test indicated that the effect of load is more significant than that of particle volume fraction and the combined effect of both factors showed the lowest significance.

The regression equation of wear rate of epoxy/Al_2O_3 composites under dry sliding conditions is:

$$q = 1679 - 69f - 1078N + 1.15f^2 + 158N^2 + 32.6fN - 1.5fN^2 - 0.36Nf^2 - 0.051f^2N^2$$

(3.1)

Where;

q : wear rate (10^{-3} mm^3/m),

f : volume fraction (vol. %), and

N : normal load (kg)

This equation is fitted to data with adjusted R^2 value of 95.71% and standard deviation of 104×10^{-3} mm^3/m. Observations no 15 and 24 showed the maximum residual. Figure 3-27 shows percent, frequency of residuals, and residual plots versus fitted values and run order. From normal probability plot, it was noticed that the percent of zero-value residual is about 40 − 60 %, and its occurrence was noticed from the histogram to be 15 times.

ANOVA was performed by Russell *et al.* [60] to determine only the principal effects of different variables such as cure temperature, initiator concentration, and rubber concentration upon the phase distribution in rubber-modified epoxy resin. Variables that exhibited a very strong effect (probability < 0.01), strong effect (probability between 0.01 and 0.05) or moderate effect (probability between 0.05 and 0.10) were reanalyzed using a means statement.
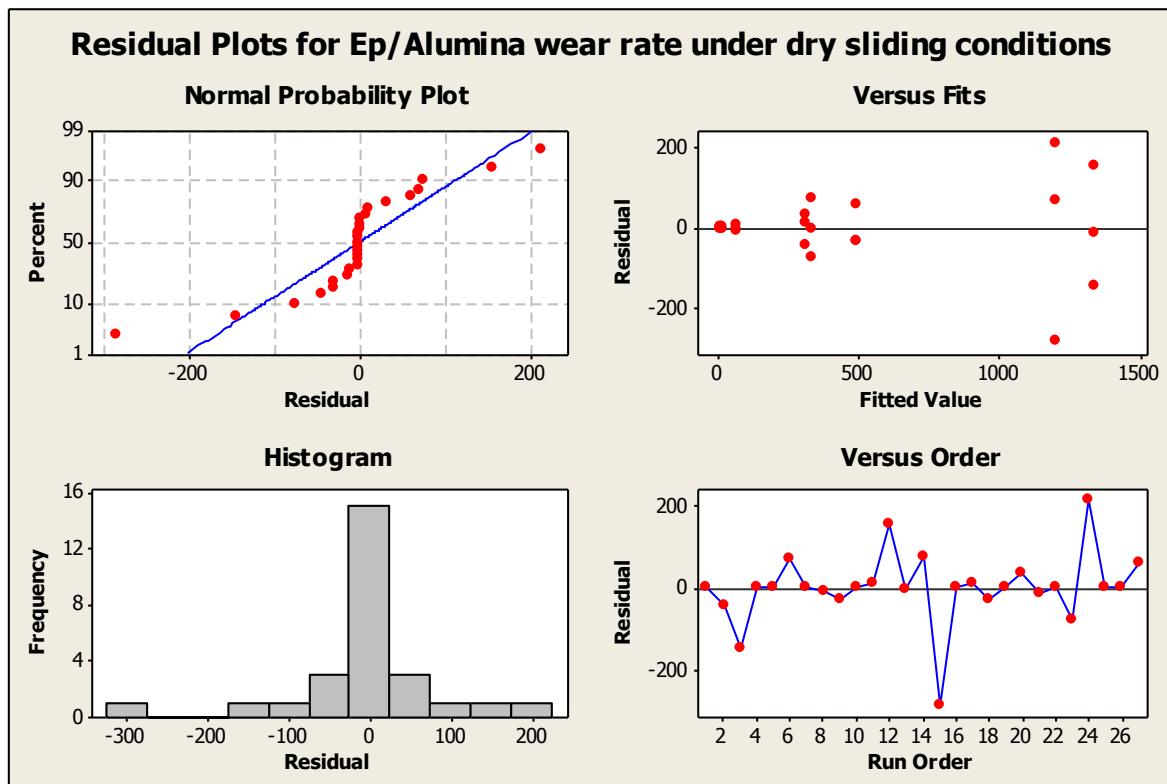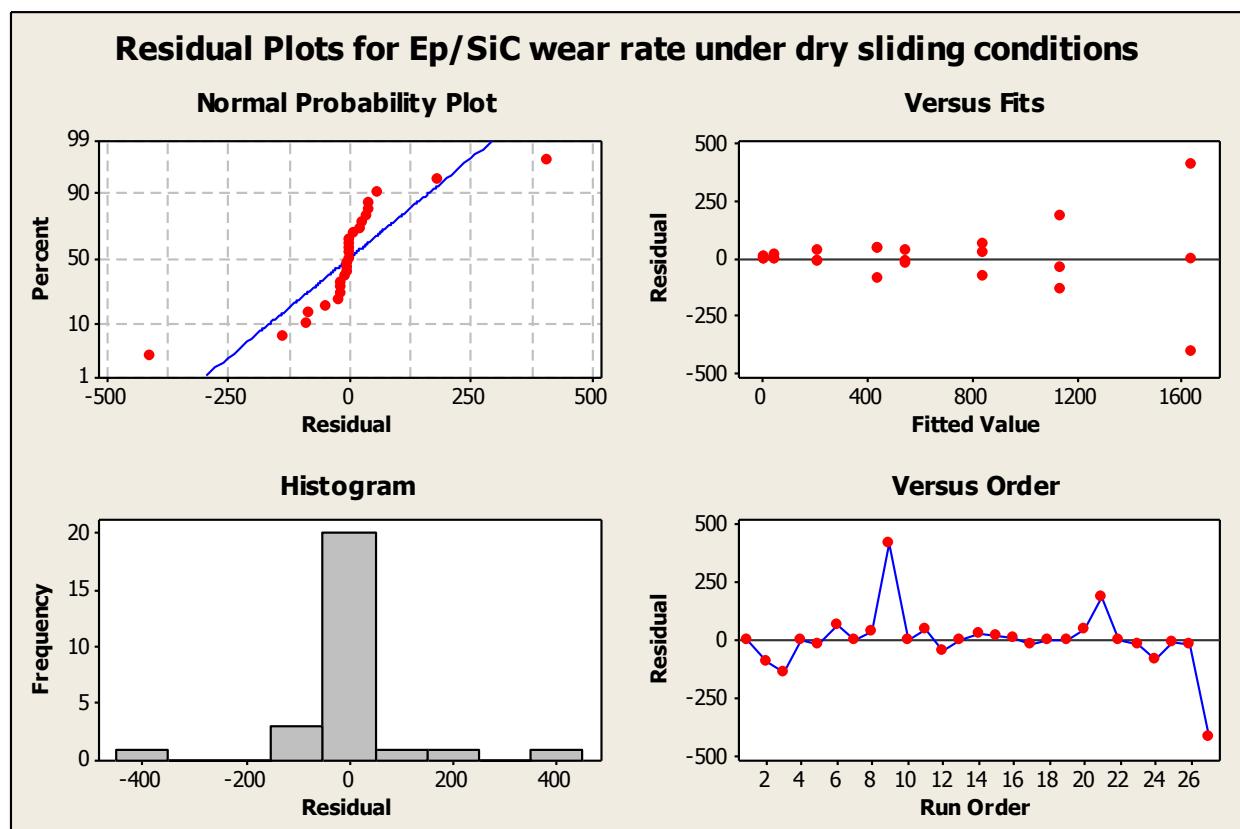
Figure 3-27: residual plots for epoxy/Al₂O₃ wear rate under dry sliding conditions

Figure 3-28 shows the ANOVA results of wear rate of epoxy/SiC composites under dry sliding conditions. It is also noticed that all factors have a strong effect on wear rate (p value is lower than 0.05). F-test indicated that the effect of load is more significant than that of particle volume fraction and the combined effect of both factors showed the lowest significance.

The regression equation of wear rate of epoxy/SiC composites under dry sliding conditions is:

$$q = -760 + 107\,f + 197\,N - 1.95\,f^2 + 59\,N^2 - 46\,f\,N - 0.8\,f\,N^2 + 0.67\,N\,f^2 + 0.094\,f^2\,N^2$$
$$(3.2)$$

This equation is fitted to data with adjusted R^2 value of 92.68% and standard deviation of 152×10^{-3} mm³/m. Observations no 9 and 27 showed the maximum residual. Figure 3-29 shows percent and frequency of residuals and residual plots versus fitted values and run order. From normal probability plot, it was noticed that the percent of zero-value residual is about 20 – 90 %, and its occurrence was noticed from the histogram to be 20 times.

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```
General Linear Model: Ep/SiC wear rate versus particle volume fraction and load
under dry sliding conditions

Factor                Type    Levels  Values
volume fraction  fixed        3  10, 20, 30
load                  fixed        3  2.4, 4.4, 6.0

Analysis of Variance for SiC wear rate, using Adjusted SS for Tests

Source                    DF    Seq SS   Adj SS   Adj MS       F      P
volume fraction            2    692566   692566   346283   14.93  0.000
load                       2   6655097  6655097  3327548  143.49  0.000
volume fraction*load       4    468553   468553   117138    5.05  0.007
Error                     18    417420   417420    23190
Total                     26   8233635

S = 152.283   R-Sq = 94.93%   R-Sq(adj) = 92.68%

Unusual Observations for SiC wear rate

       Ep/SiC
Obs   wear rate       Fit   SE Fit   Residual   St Resid
  9    2052.55   1642.04    87.92     410.51       3.30 R
 27    1231.53   1642.04    87.92    -410.51      -3.30 R

R denotes an observation with a large standardized residual.
```

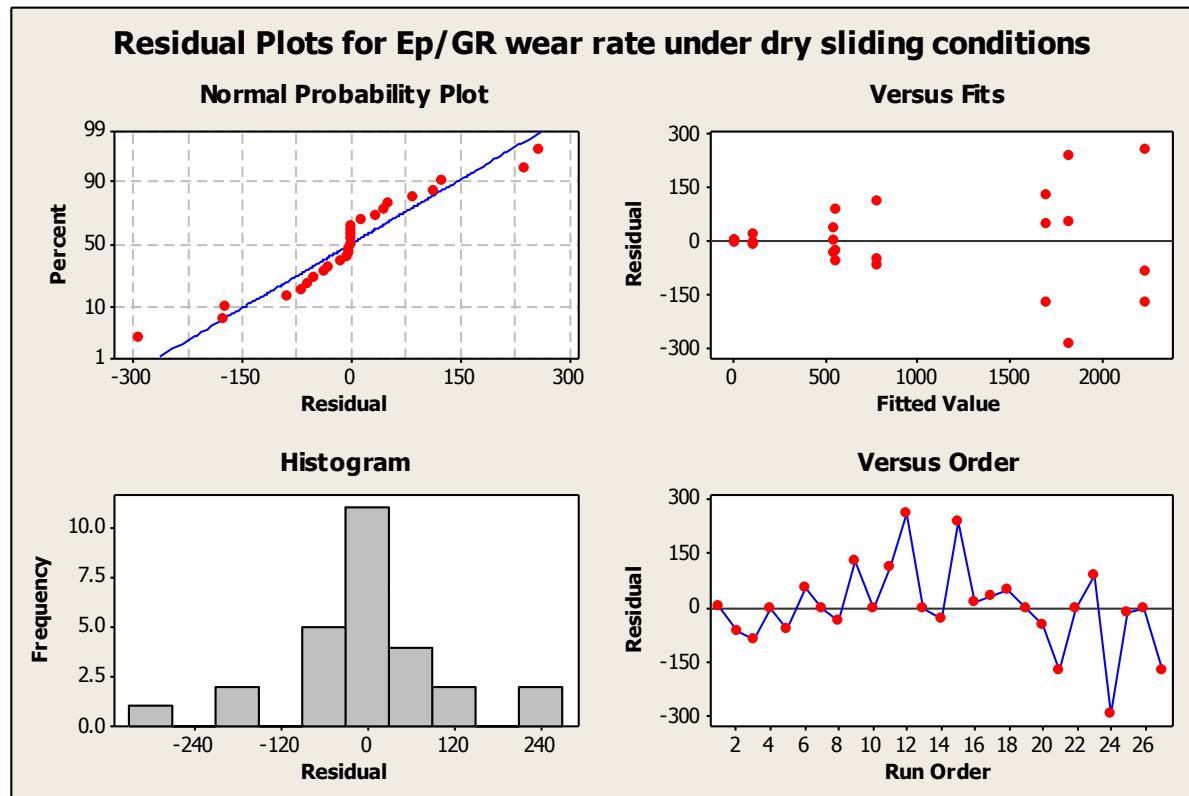Figure 3-28: ANOVA results of epoxy/SiC wear rate under dry sliding conditions

Figure 3-29: residual plots for epoxy/SiC wear rate under dry sliding conditions

Figure 3-30 shows the ANOVA results of wear rate of epoxy/graphite composites under dry sliding conditions. The results indicated that all factors have a strong effect on wear rate (p value is lower than 0.05). P-value and F-test indicated that the effect of load is more significant than that of particle volume fraction and the combined effect of both factors showed the lowest significance.

The regression equation of wear rate of epoxy/graphite composites under dry sliding conditions is:

$$q = 167 + 51\,f - 350\,N - 0.73\,f^2 + 135\,N^2 - 30\,f\,N + 1.3\,f\,N^2 + 0.60\,N\,f^2 - 0.040\,f^2\,N^2$$

$$(3.3)$$

This equation is fitted to data with adjusted R^2 value of 97.26% and standard deviation of $136 \times 10^{-3}\,\mathrm{mm^3/m}$. Observations no 12, 15, and 24 showed the maximum residual. Figure 3-31 shows percent and frequency of residuals and residual plots versus fitted values and run order. From normal probability plot, it was noticed that the percent of zero-value residual is about 30 – 70 %, and its occurrence was noticed from the histogram to be 11 times.

```
General Linear Model: Ep/graphite wear rate versus particle volume fraction and
load under dry sliding conditions

Factor              Type    Levels  Values
volume fraction     fixed        3  10, 20, 30
load                fixed        3  2.4, 4.4, 6.0

Analysis of Variance for Ep/graphite wear rate, using Adjusted SS for Tests

Source                  DF     Seq SS      Adj SS     Adj MS       F       P
volume fraction          2     294493      294493     147247     7.95  0.003
load                     2   16648363    16648363    8324181   449.27  0.000
volume fraction*load     4     314640      314640      78660     4.25  0.014
Error                   18     333506      333506      18528
Total                   26   17591002

S = 136.118   R-Sq = 98.10%   R-Sq(adj) = 97.26%

Unusual Observations for Ep/graphite wear rate

     Ep/graphite
Obs wear rate      Fit  SE Fit  Residual  St Resid
 12   2500.30  2241.48   78.59    258.82      2.33 R
 15   2066.72  1827.49   78.59    239.23      2.15 R
 24   1535.09  1827.49   78.59   -292.40     -2.63 R

R denotes an observation with a large standardized residual.
```

Figure 3-30: ANOVA results of epoxy/graphite wear rate under dry sliding conditions

Figure 3-31: residual plots for epoxy/graphite wear rate under dry sliding conditions

Figure 3-32 shows the ANOVA results of wear rate of epoxy/Al$_2$O$_3$ composites under water lubricated sliding conditions. The results indicated that the combined effect of particle volume fraction and load factors has a weak effect on wear rate (p value is higher than 0.05). F-test indicated that the effect of load is more significant than that of particle volume fraction.

The regression equation of wear rate of epoxy/Al$_2$O$_3$ composites under water lubricated sliding conditions is:

$$q = 1541 - 48\,f - 101\,N - 3\,f\,N + 1.0\,f^2 + 1.1\,N^2 + 0.06\,f^2\,N + 0.28\,f\,N^2 - 0.006\,f^2\,N^2 \tag{3.4}$$

Where; q is the wear rate (10^{-6} mm^3/m)

This equation is fitted to data with adjusted R^2 value of 73.89% and standard deviation of 55×10^{-6} mm^3/m. Observations no 15, 18, 24, and 27 showed the maximum residual. Figure 3-33 shows percent and frequency of residuals and residual plots versus fitted values and run order. From normal probability plot, it was noticed that the percent of zero-value residual is about $30 - 70$ %, and its occurrence was noticed from the histogram to be 11 times.

```
General Linear Model: Ep/Al₂O₃ wear rate versus particle volume fraction and load
under water lubricated sliding conditions


Factor              Type    Levels  Values
volume fraction     fixed        3  10, 20, 30
load                fixed        3  20, 22, 24

Analysis of Variance for Ep/Al. wear rate, using Adjusted SS for Tests

Source                DF   Seq SS   Adj SS   Adj MS      F      P
volume fraction        2    54280    54280    27140   9.01  0.002
load                   2   171146   171146    85573  28.40  0.000
volume fraction*load   4    20442    20442     5111   1.70  0.195
Error                 18    54243    54243     3014
Total                 26   300112

S = 54.8955   R-Sq = 81.93%   R-Sq(adj) = 73.89%

Unusual Observations for Ep/Al. wear rate

         Ep/Al.
Obs   wear rate      Fit  SE Fit  Residual  St Resid
 15     202.000  302.333  31.694  -100.333     -2.24 R
 18     194.000  291.000  31.694   -97.000     -2.16 R
 24     403.000  302.333  31.694   100.667      2.25 R
 27     388.000  291.000  31.694    97.000      2.16 R

R denotes an observation with a large standardized residual.
```

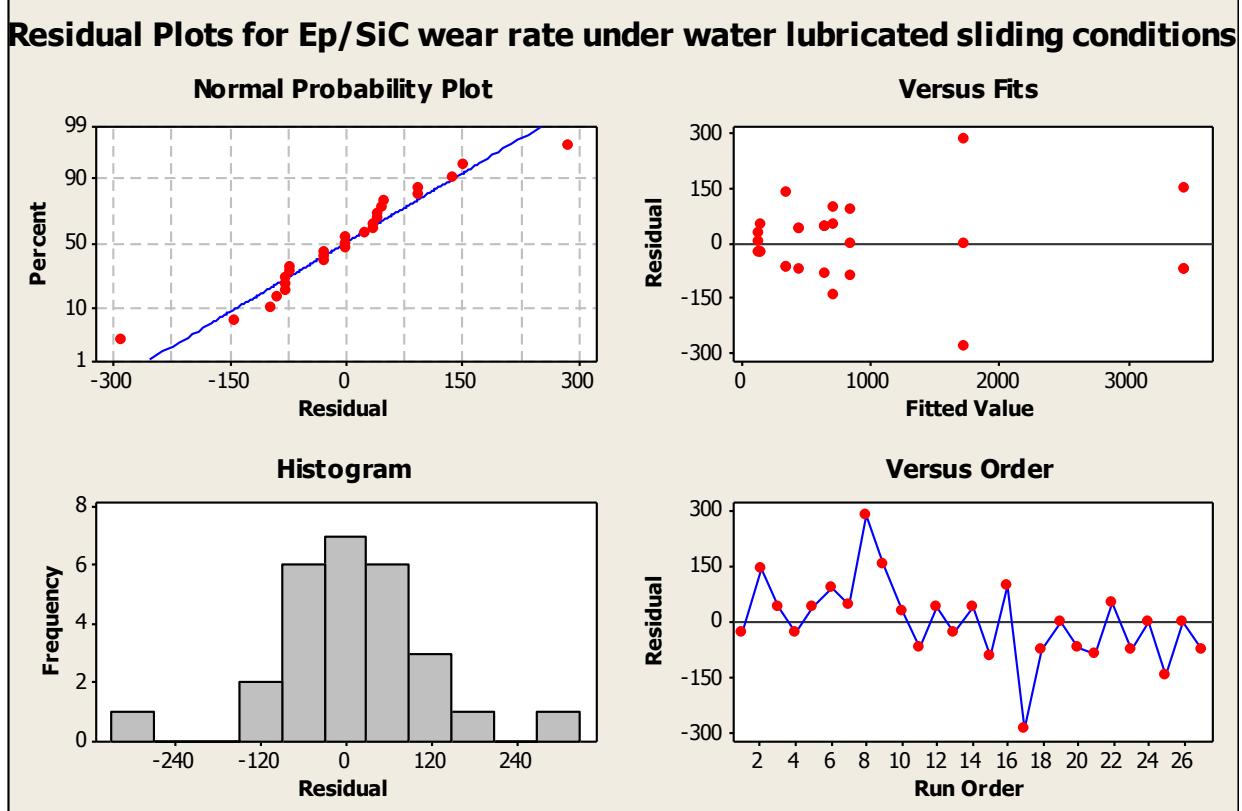Figure 3-32: ANOVA results of epoxy/Al₂O₃ wear rate under water lubricated sliding conditions
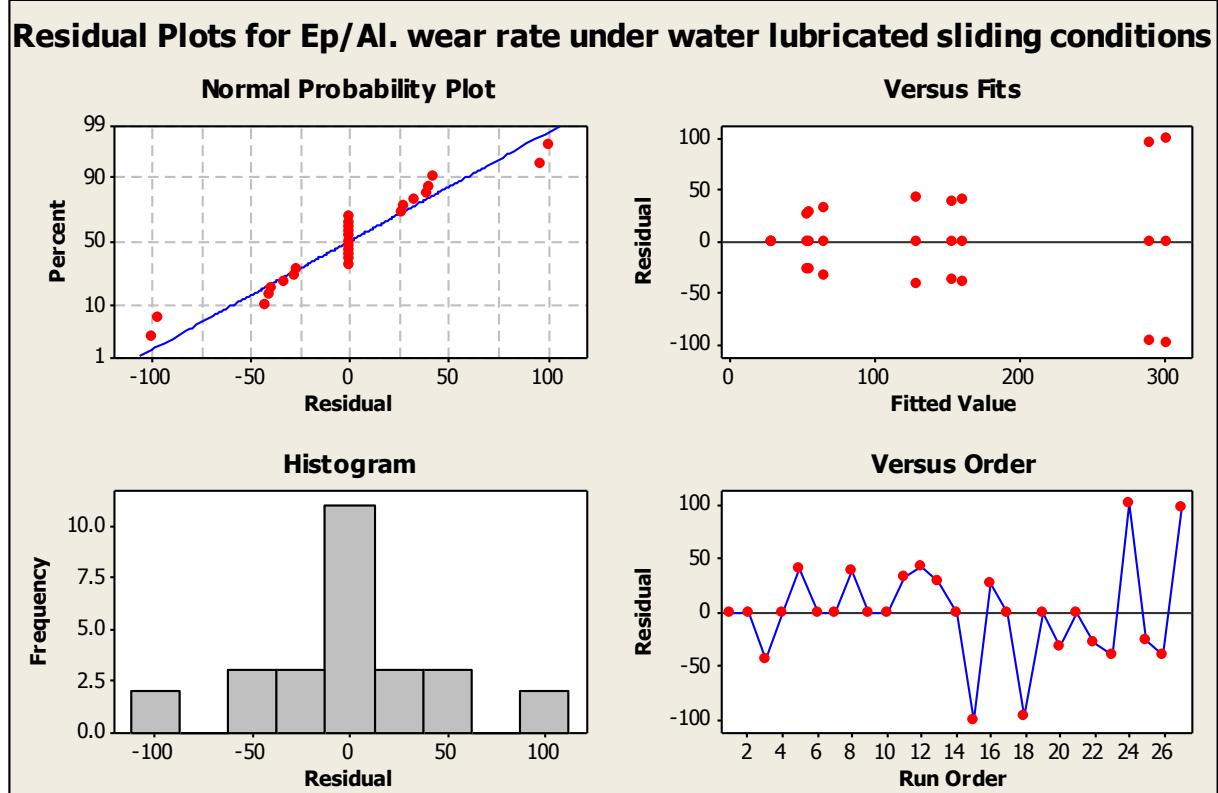
Figure 3-33: residual plots for epoxy/Al₂O₃ wear rate under water lubricated sliding conditions

ANOVA carried out on wear rate results of epoxy/SiC composites under water lubricated sliding conditions indicated that all factors have a strong effect on wear rate (p value is lower than 0.05). F-test indicated that the effect of particle volume fraction is more significant than that of load and the combined effect of both factors showed the lowest significance (Figure 3-34).

The regression equation of wear rate of epoxy/SiC composites under water lubricated sliding conditions is:

$$q = 28425\text{-}3973\,f\text{-}3004\,N + 411\,f\,N + 134\,f^2 + 81\,N^2\text{-}14.0\,f^2\,N\text{-}10.8\,f\,N^2 + 0.371\,f^2\,N^2$$
(3.5)

This equation is fitted to data with adjusted R^2 value of 98.37% and standard deviation of 130×10^{-6} mm^3/m. Observations no 8 and 17 showed the maximum residual. Figure 3-35 shows percent and frequency of residuals and residual plots versus fitted values and run order. From normal probability plot, it was noticed that the percent of zero-value residual is about 50 %, and its occurrence was noticed from the histogram to be 7 times.

```
General Linear Model: Ep/SiC wear rate versus particle volume fraction and load
under water lubricated sliding conditions

Factor              Type    Levels  Values
volume fraction     fixed        3  10, 20, 30
load                fixed        3  20, 22, 24

Analysis of Variance for Ep/SiC wear rate, using Adjusted SS for Tests

Source                DF     Seq SS     Adj SS    Adj MS       F      P
volume fraction        2   14141943   14141943   7070972  418.13  0.000
load                   2    7858674    7858674   3929337  232.36  0.000
volume fraction*load   4    4694082    4694082   1173521   69.39  0.000
Error                 18     304393     304393     16911
Total                 26   26999093

S = 130.041   R-Sq = 98.87%   R-Sq(adj) = 98.37%

Unusual Observations for Ep/SiC wear rate

        Ep/SiC
Obs   wear rate      Fit   SE Fit   Residual  St Resid
  8     2011.00  1724.00    75.08     287.00      2.70 R
 17     1437.00  1724.00    75.08    -287.00     -2.70 R

R denotes an observation with a large standardized residual.
```

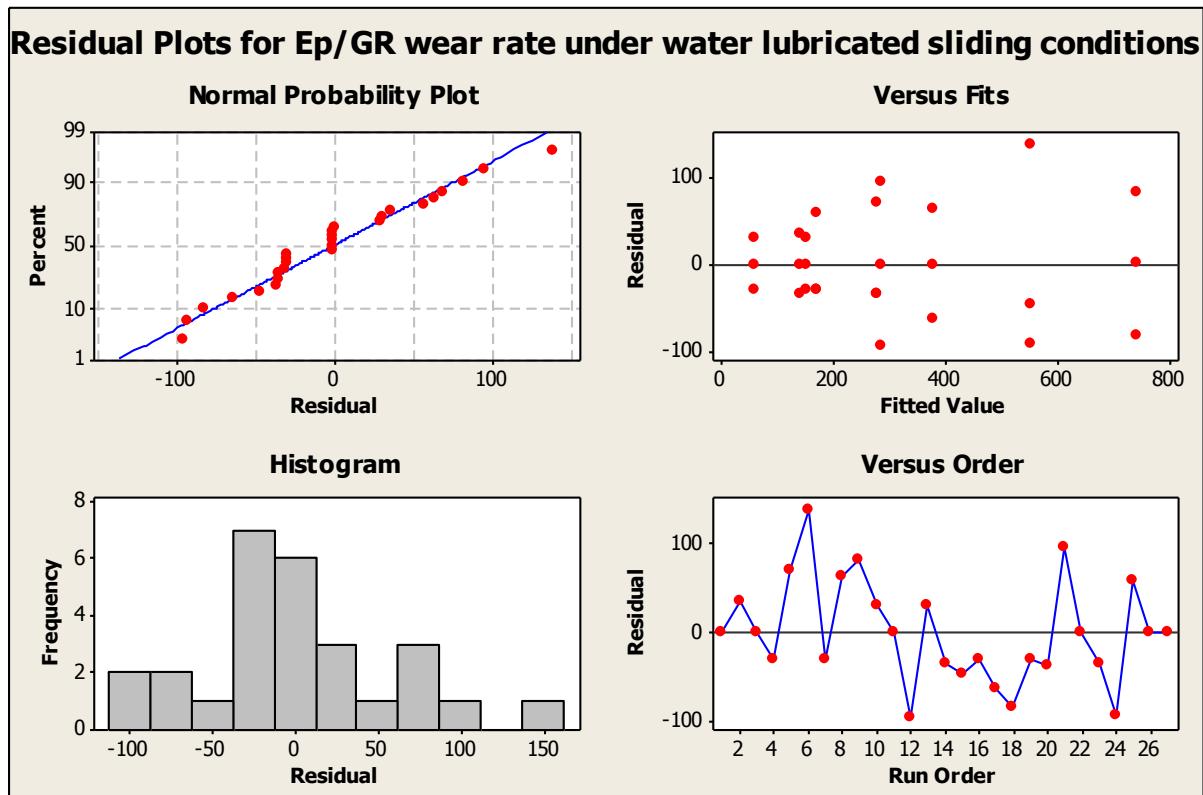Figure 3-34: ANOVA results of epoxy/SiC wear rate under water lubricated sliding conditions

Figure 3-35: residual plots for epoxy/SiC wear rate under water lubricated sliding conditions

ANOVA carried out on wear rate results of epoxy/graphite composites under water lubricated sliding conditions indicated that all factors have a strong effect on wear rate (p value is lower than 0.05). F-test indicated that the effect of load is more significant than that of particle volume fraction and the combined effect of both factors showed the lowest significance (Figure 3-36).

The regression equation of wear rate of epoxy/graphite composites under water lubricated sliding conditions is:

$$q = -6523 + 1134\,f + 578\,N - 106\,fN - 23.3\,f^2 - 12.9\,N^2 + 2.11\,f^2\,N + 2.51\,f\,N^2 - 0.048\,f^2\,N^2$$
$$(3.6)$$

This equation is fitted to data with adjusted R^2 value of 89.8% and standard deviation of 70×10^{-6} mm^3/m. Observations no 6 showed the maximum residual. Figure 3-37 shows percent and frequency of residuals and residual plots versus fitted values and run order. From normal probability plot, it was noticed that the percent of zero-value residual is about 50 %, and its occurrence was noticed from the histogram to be 6 times.

```
General Linear Model: Ep/graphite wear rate versus particle volume fraction and
load under water lubricated sliding conditions

Factor            Type    Levels  Values
volume fraction   fixed        3  10, 20, 30
load              fixed        3  20, 22, 24

Analysis of Variance for Ep/graphite wear rate, using Adjusted SS for Tests

Source                DF    Seq SS   Adj SS   Adj MS      F      P
volume fraction        2    330378   330378   165189  33.61  0.000
load                   2    742442   742442   371221  75.53  0.000
volume fraction*load   4     91618    91618    22904   4.66  0.009
Error                 18     88467    88467     4915
Total                 26   1252905

S = 70.1060   R-Sq = 92.94%   R-Sq(adj) = 89.80%

Unusual Observations for Ep/geaphite wear rate

         Ep/graphite
Obs   wear rate      Fit  SE Fit  Residual  St Resid
  6     693.000  554.333  40.476   138.667      2.42 R

R denotes an observation with a large standardized residual.
```

Figure 3-36: ANOVA results of epoxy/graphite wear rate under water lubricated sliding conditions

Figure 3-37: residual plots for epoxy/graphite wear rate under water lubricated sliding conditions

3.7   SCANNING ELECTRON MICROSCOPY (SEM)

The worn surfaces of the epoxy matrix and the composites were examined by scanning electron microscope (SEM) of type Joel-GXA-840A electron probe micro-analyzer. Figure 3-38 shows SEM micrographs of the worn surface of the epoxy matrix specimens under dry sliding conditions. Small ploughs and debris were formed on the worn surface of the base matrix under dry sliding conditions at 2.4 kg load (Figure 3-38 c) due to the effect of load on breaking bonds between polymeric chains. Thus, wear rate is low. At higher applied load of 4.4 kg, large fragments were observed on the surface (Figure 3-38 f) and consequently, the wear rate increased. It can be supposed that, during rubbing action, an epoxy-based material may behave in a brittle manner. And small cracks may be introduced perpendicular to the sliding direction producing a wavy surface. And therefore, wear debris and ploughs can be formed. It is clear that the presence of ploughs within surface of the specimen indicates that an abrasive wear mechanism occurred. Wear debris were noticed to be attached to the wear grooves.

Ploughs and separated particles were observed on the worn surface of the epoxy+10%vol. Al_2O_3 composites under dry sliding conditions at 2.4 kg load (Figure 3-39 c) and the wear rate is low. But at load of 4.4 kg, large ploughs and fragments were observed on the surface with the separated particles of Al_2O_3 (Figure 3-39 f). Therefore, the wear rate is significantly increased. Wear rate of epoxy+10%vol. Al_2O_3 composites is generally higher than that of the base matrix.

Small ploughs and immersed particles were observed on the worn surface of the epoxy+10%vol. SiC composites under dry sliding conditions at 2.4 kg load (Figure 3-40 c) and the wear rate is lower than that of Al_2O_3 composites. The attached particles of SiC indicated that the interfacial bonding between particles and the base epoxy matrix is fairly strong. But at load of 4.4 kg, large ploughs and fragments were observed on the surface due to the detachment of particles (Figure 3-40 f). Therefore, the wear rate is considerably increased. Wear rate of epoxy+10%vol. SiC composites is higher than that of epoxy+10%vol. Al_2O_3 composites at 4.4 kg load.

Large ploughs and separated particles were observed on the worn surface of the epoxy+10%vol. graphite composites under dry sliding conditions at 2.4 kg load (Figure 3-41 c) and the wear rate is low. But at load of 4.4 kg load, large amounts of small fragments were observed on the surface with the separated particles of graphite (Figure 3-41 f). Therefore, the wear rate is extensively increased. Wear rate of epoxy+10%vol. graphite composites is generally higher than that of the base epoxy matrix and its composites with Al_2O_3 and SiC particulates.

(a)

(d)

(b)

(e)

(c)

(f)
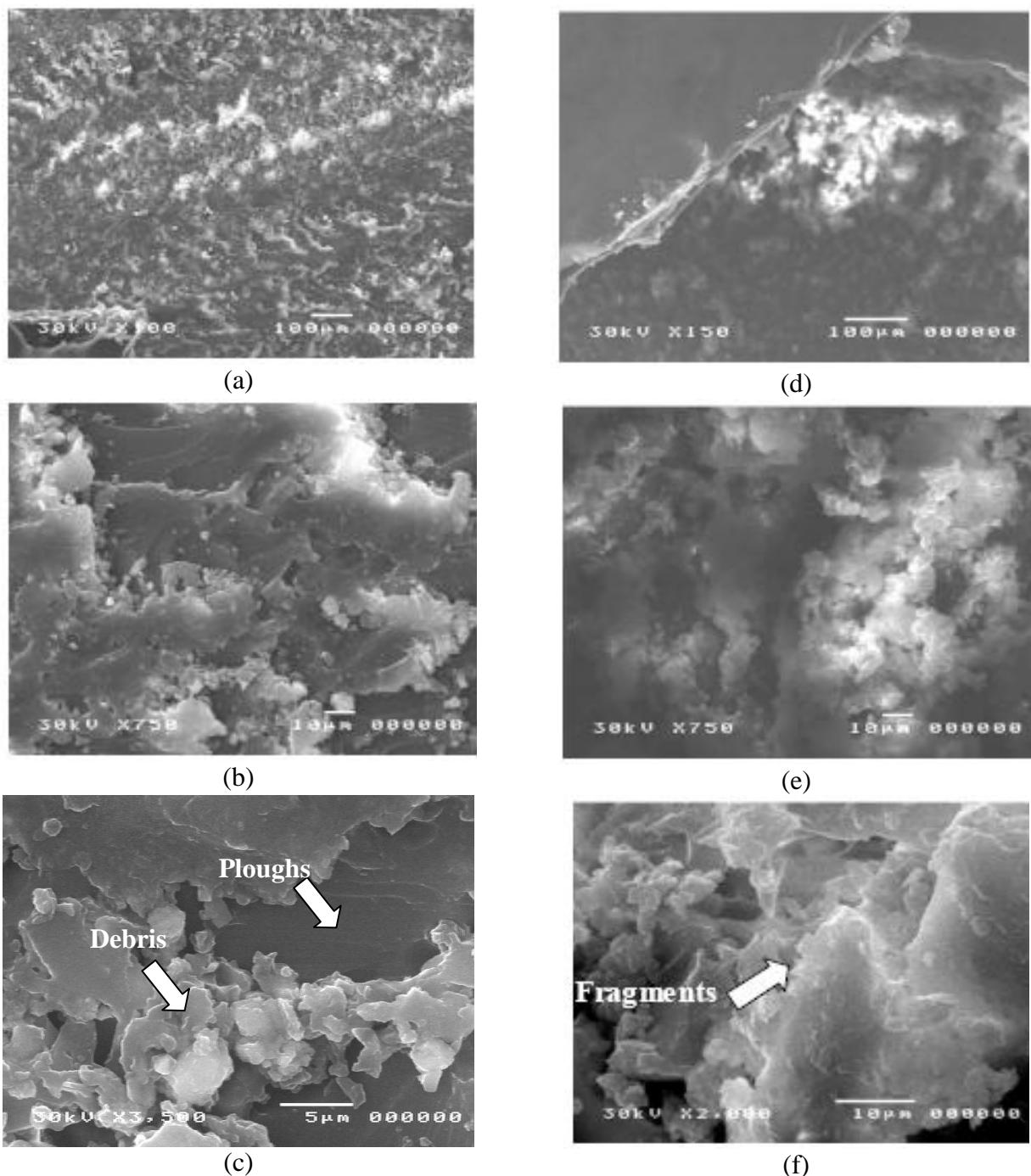
Figure 3-38: SEM of epoxy matrix worn surface under dry sliding conditions (a,b,c) at 2.4 kg load (d,e,f) at 4.4 kg load with different magnifications
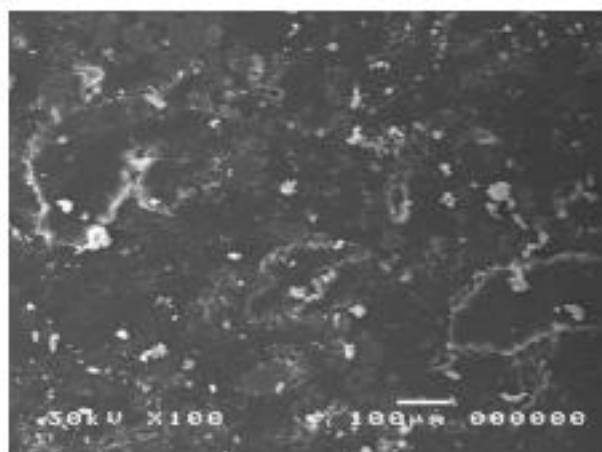
(a)
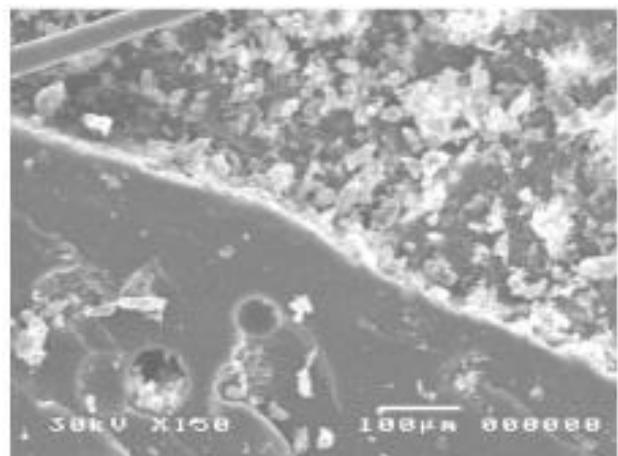
(d)
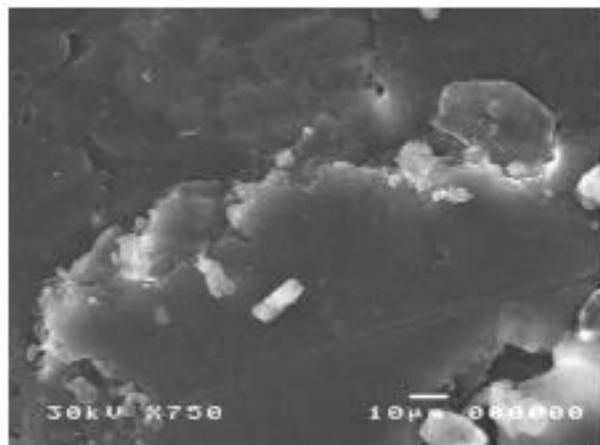
(b)
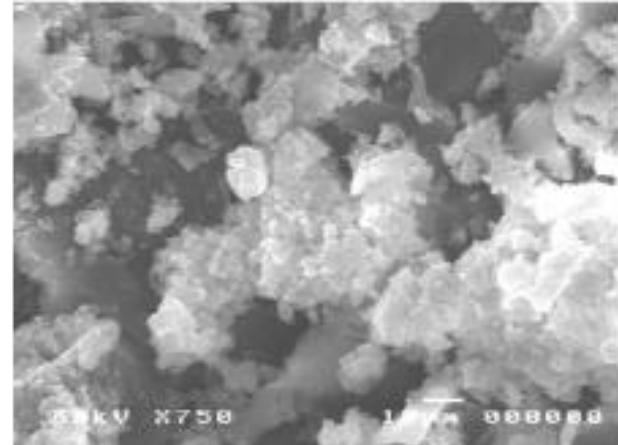
(e)
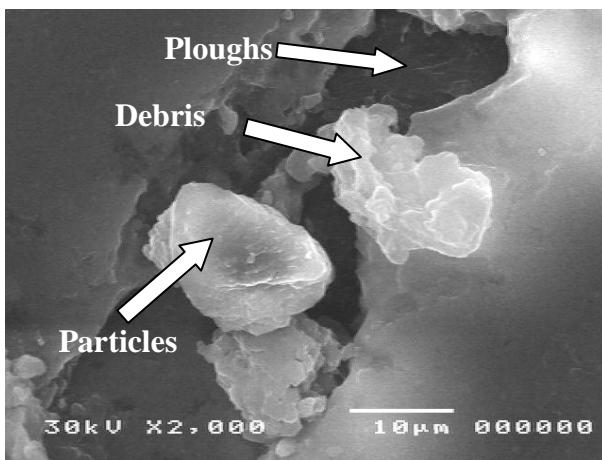
(c)
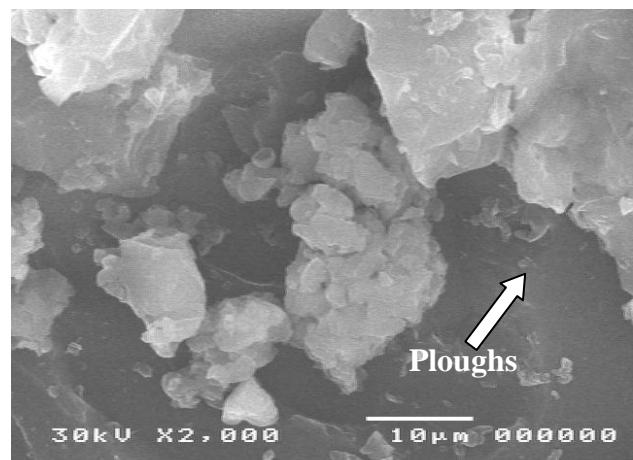
(f)

Figure 3-39: SEM of worn surfaces of epoxy +10% vol. Al_2O_3 composites under dry sliding conditions (a,b,c) at 2.4 kg load (d,e,f) at 4.4 kg load with different magnifications
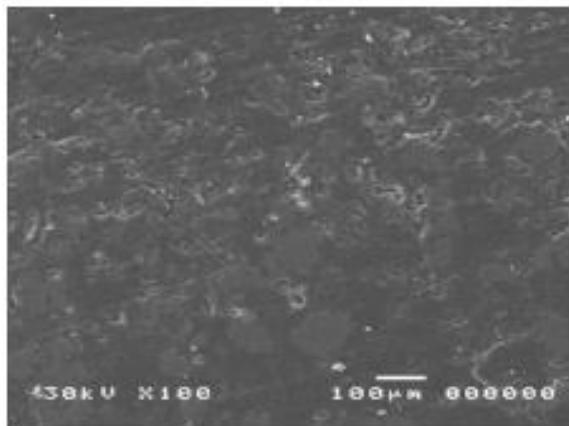
(a)
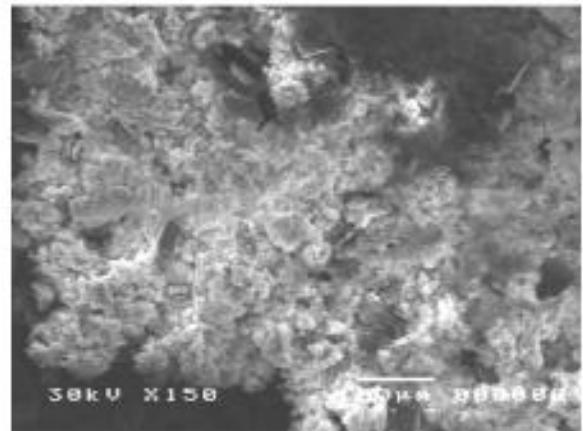
(d)
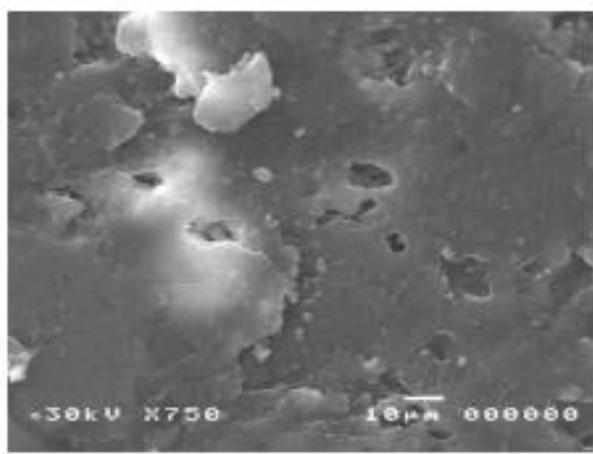
(b)
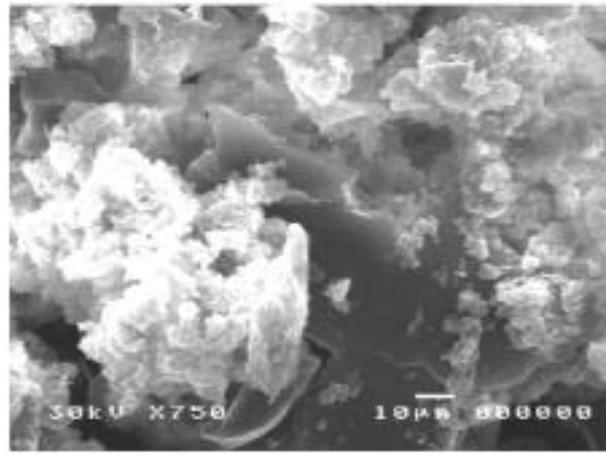
(e)
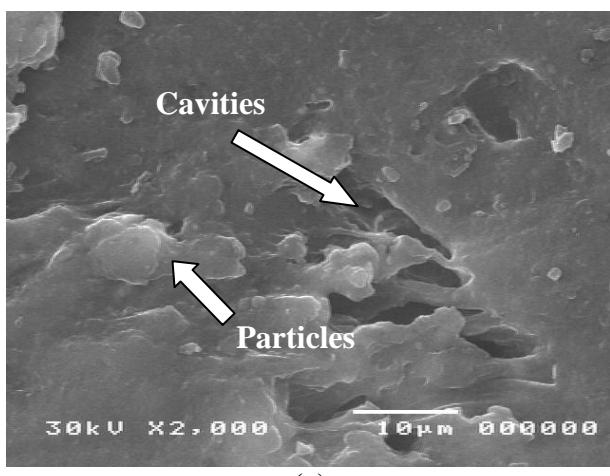
(c)
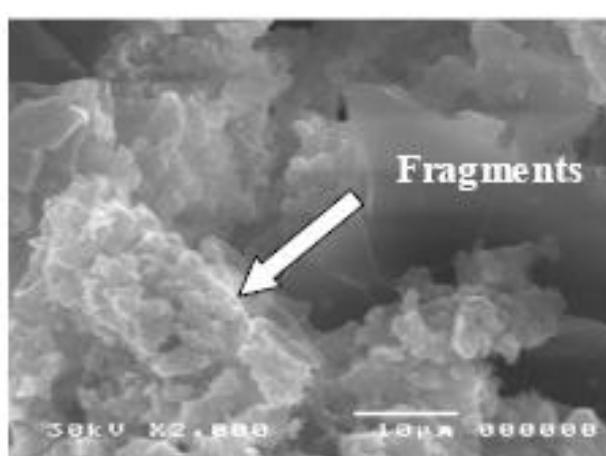
(f)

Figure 3-40: SEM of worn surfaces of epoxy + 10% vol. SiC composites under dry sliding conditions (a,b,c) at 2.4 kg load (d,e,f) at 4.4 kg load with different magnifications
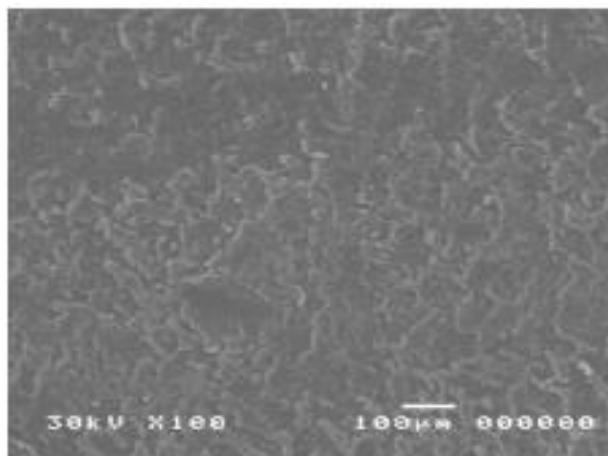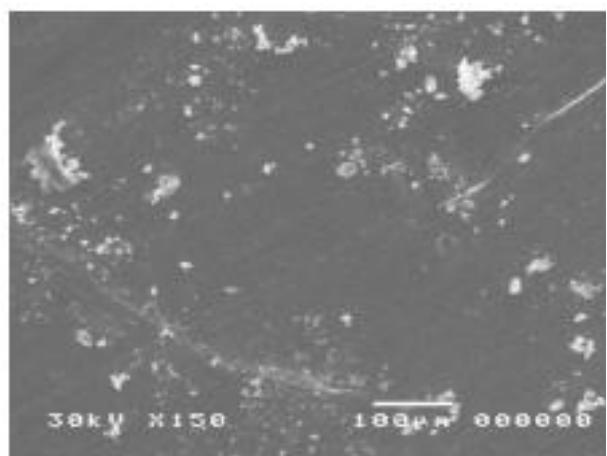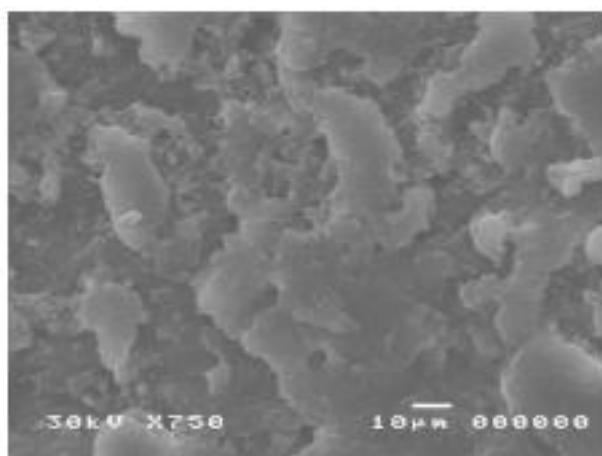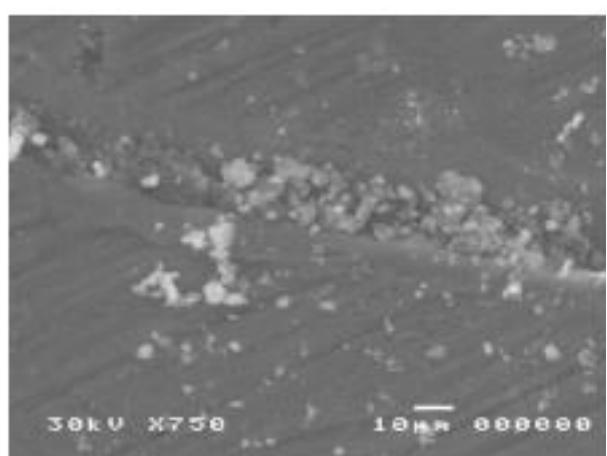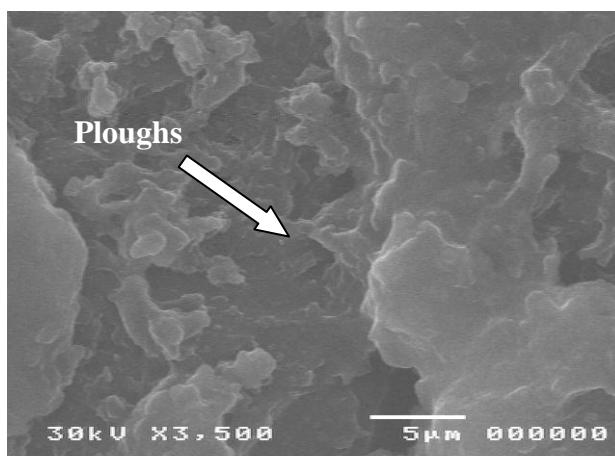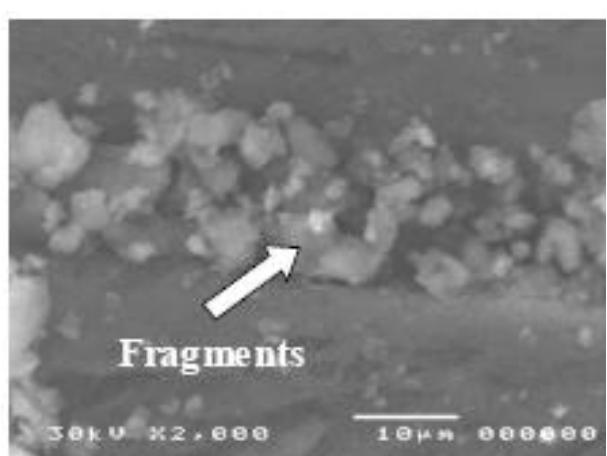
Figure 3-41: SEM of worn surfaces of epoxy+10% vol. graphite composites under dry sliding
conditions (a,b,c) at 2.4 kg load (d,e,f) at 4.4 kg load with different magnifications

Under water lubricated sliding conditions, no fragments were noticed on the specimen surface. Figure 3-42 shows the appearance of polished grains of the base epoxy matrix. Thus, a very low wear rate was being recognized.

In case of epoxy+10%vol. Al_2O_3 composites under water lubricated sliding conditions, fine cracks were observed on the worn surface (Figure 3-43). But Al_2O_3 particles were appeared to be impeded into the base matrix reducing the wear rate.

Surface fractures and separated SiC particles were noticed on the worn surface of epoxy+10%vol. SiC composites (Figure 3-44), and the wear rate was increased.

In case of epoxy+10%vol. graphite composites under water lubricated sliding conditions, thin layers of the material upon limited areas were taken off out of the worn surface but not fractured (Figure 3-45), and thus, wear rate is approximately the same as that of the base matrix.
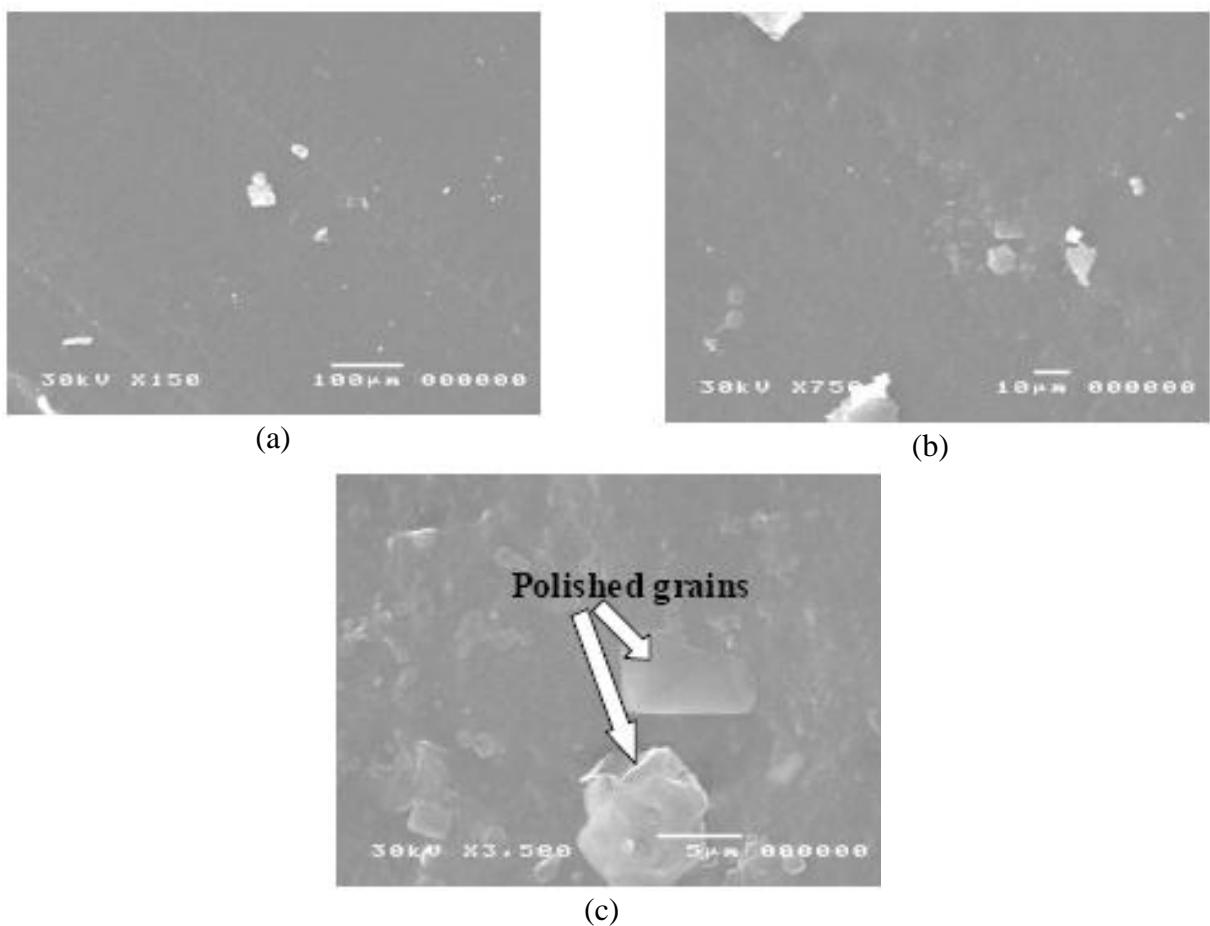

(a)

(b)

(c)

Figure 3-42: SEM of worn surfaces of epoxy matrix under wet sliding conditions and 20kg load with different magnifications

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(a)

(b)

(c)
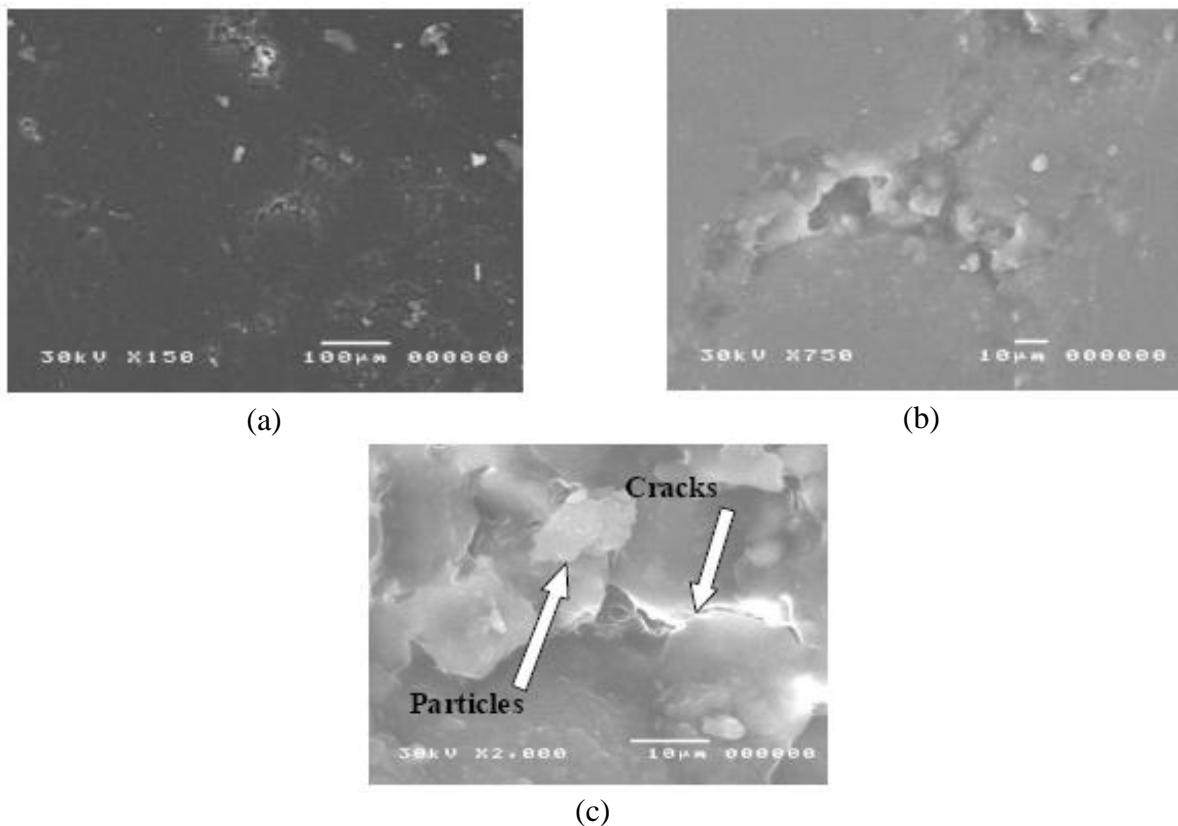
Figure 3-43: SEM of worn surfaces of epoxy+10% vol. Al$_2$O$_3$ composites under wet sliding conditions and 20kg load with different magnification
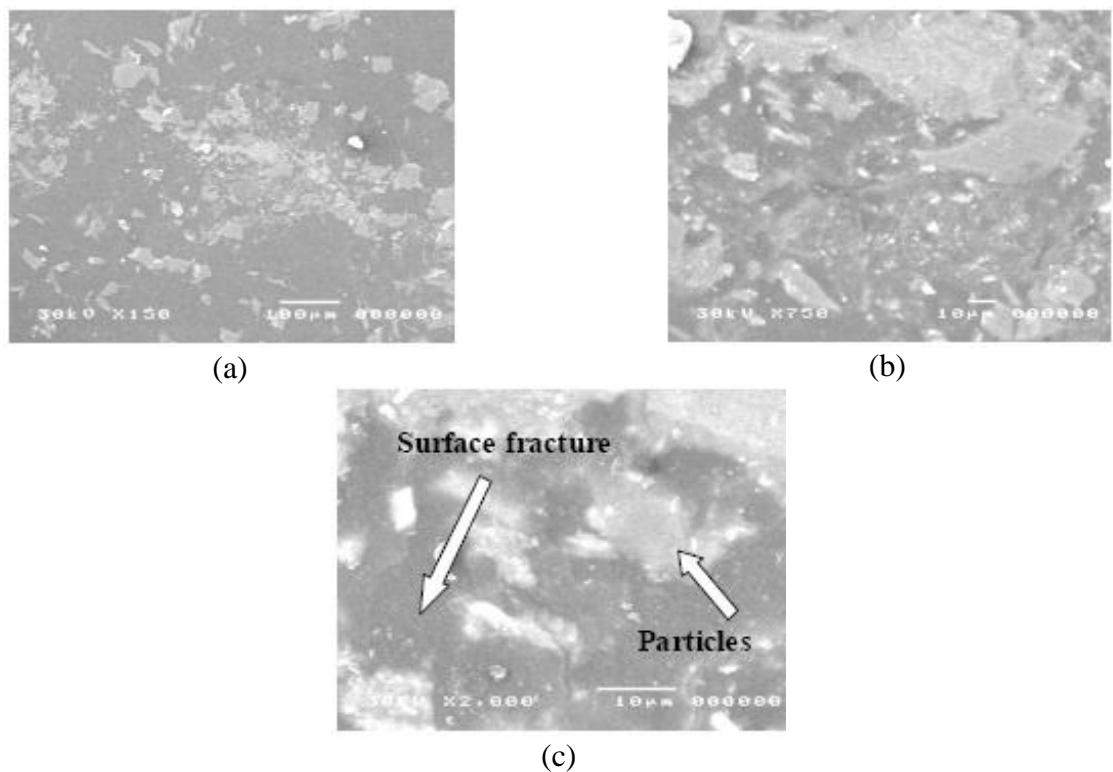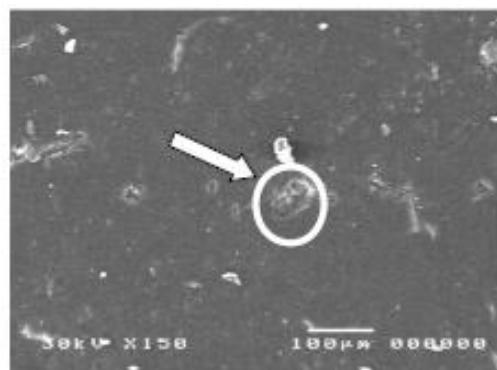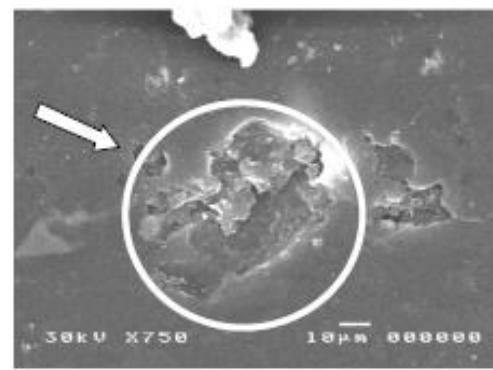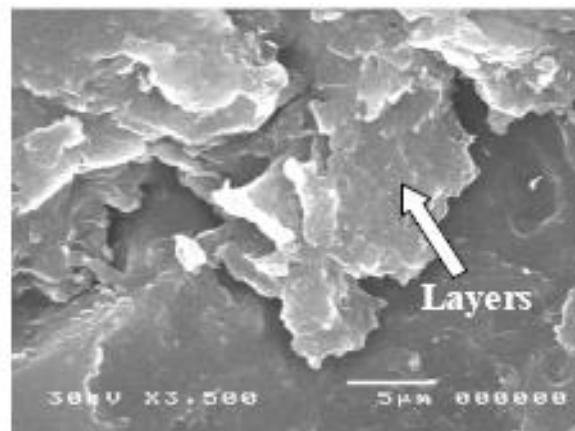

(a)

(b)

(c)

Figure 3-44: SEM of worn surfaces of epoxy+10% vol. SiC composites under wet sliding conditions and 20kg load with different magnification

(a)

(b)

(c)

Figure 3-45: SEM of worn surfaces of epoxy+10% vol. graphite composites under wet sliding conditions and 20kg load with different magnification