

Evaluation of Wear Characteristics of PMMA-Nano SiO₂ and Nano ZnO Composites using L9 (3⁴) Orthogonal Array of Experimental Design by Pin-On Disc Tribometer

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Abstract

Results of dry wear testing of PMMA composites reinforced with nano SiO₂ and nano ZnO and their micron sized counter parts using pin-on disc tribometer are presented. L9 (3⁴) orthogonal array of experimental design was chosen to study the specific wear rate. Nano SiO₂ and nano ZnO were characterized by SEM-EDAX and XRD. PMMA nano composites exhibited lowest wear rate compared to PMMA composites reinforced with their micron counter parts and neat PMMA. 3D surface profilometric examination of worn out sample surfaces corroborated the wear rates.

Keywords: Wear rate of PMMA-nano SiO₂ and nano ZnO composites, L9 (3⁴) OA Design, pin-on disc tribometer, surface profilometry.

Introduction

Poly methyl methacrylate (PMMA), widely used as a prosthodontic denture base. The denture base materials should exhibit good mechanical properties and dimensional stability in moist environment (Salih, Olewi, and Alaa Mohammed 2016). One of the important properties of PMMA is excellent wear resistance.

Canché(G.Canché-EscamillaS., Duarte-Arandaa, and M.Toledano n.d.) et al evaluated the effect of hybrid silica/poly(methylmethacrylate) (PMMA) nanoparticles on the properties of for dental restoration. They prepared hybrid nanoparticles with silica as core and PMMA as shell were obtained by a seeded emulsion polymerization process. They further reported, that composites with low amounts of PMMA shell had higher modulus than those in which silica was used as the filler.

Akinci (Akinci, Sen, and Sen 2014) reported the friction and wear performance of pure poly (methyl methacrylate) [PMMA] and zirconium oxide (ZrO₂) filled PMMA composites were realized under dry sliding conditions. Wear tests were carried out at room temperature under the loads of 5N, 10N and 20N at the sliding speed of 0.5m/s, 1.0m/s and 1.5m/s. The coefficients of friction of the composites were significantly influenced with ZrO₂ content. Results for testing

materials showed that the friction coefficient and the wear rate are sensitive to the applied loads and sliding speeds. The wear rates of the PMMA composite are changing between 3.01×10⁻⁷mm³/m and 5.50×10⁻⁶mm³/m, depending on ZrO₂ content, applied load and sliding speeds. It was further noted by the authors, that, higher applied loads resulted in increased coefficient of friction and wear rate.

The friction and wear properties of nano-SiO₂ and -TiO₂ particle-reinforced PMMA composites was studied by Zhenhua (Zhenhua n.d.). The composite exhibits excellent tribological properties. The wear mechanisms change from micro cutting wear, multiplastic deformation wear and adhesive wear into abrasive wear and brittle fracture wear.

Farhan and co-authors (Farhan et al. 2017) reported the friction and wear of PMMA-nano TiO₂-ZnO (particle loading at (0, 2, 3, 4 and 5 vol%) composites. It was concluded that the The wear and friction coefficient decreased with increase the time and percentage [TiO₂ - ZnO].

Fabrication of PMMA- nano silica composites with neat and triethoxyvinylsilane-modified silica nanoparticles (with loading content in the 0.25%, 0.50%, 0.75%, 1% wt range), and determination of fracture toughness was reported by (Topouzi et al. 2017) et al. They reported increase the fracture toughness, the elastic modulus and the Glass Transition Temperature of PMMA resins.

Prasad (A.J.K Prasad 2013) reported systematic studies on the dry wear and wet wear of PMMA composites reinforced with nano Al₂O₃, mixture of nano ZnO-SiO₂ and nano mixed metal oxides and their counter parts. The present study is taken up to evaluate the effects of the nano SiO₂ and nano ZnO on the specific wear rate of PMMA composites reinforced with these fillers and to compare that with PMMA composites with their micron counterparts and unfilled (neat) PMMA. It has been a known phenomenon that by increasing applied load, sliding distance and test duration, results in higher wear of materials for example, (Akinci, Sen, and Sen 2014) (Stachowiak and Batchelor 1993) (Bhushan 2013). To study the combined effects of these wear test parameters and composition of PMMA composites simultaneously, L9 (3⁴) orthogonal array of experimental design was chosen to study the specific wear rate.

Experimental

Preparation of Nano Silica by Acid Catalyzed Method

Tetraethyl Orthosilicate (TEOS) was used as the precursor for preparing nano silica by (a) acid catalyzed (acetic acid as catalyst and distilled water as hydrolyzing agent) and (b) base catalyzed routes following those reported by Azlina et al (Azlina et al. 2016) and Dabbaghian (Dabbaghian and Babalou 2010). In figures 1 and 2 the respective process steps adapted are presented.

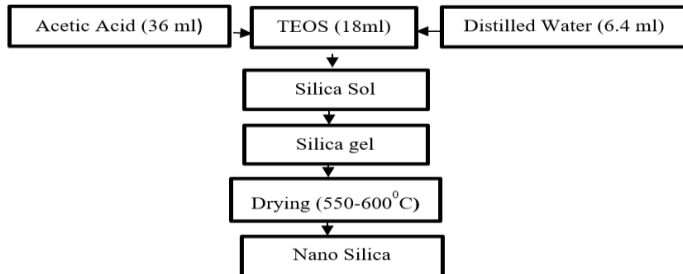


Fig. 1 Process steps for preparing Nano SiO₂ acid catalyzed [(Azlina et al. 2016)].

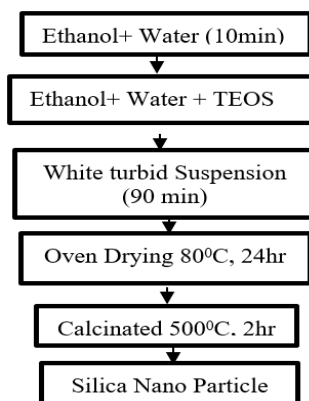


Fig. 2 Process steps for preparing Nano SiO₂ base catalyzed (Dabbaghian and Babalou 2010).

Preparation of Nano ZnO (Prasad et al. 2018)

Nano zinc oxide was prepared following the procedure adapted by (Wahab et al. 2010) (Prasad et al. 2018) process steps for nano ZnO preparation given in the figure 3.

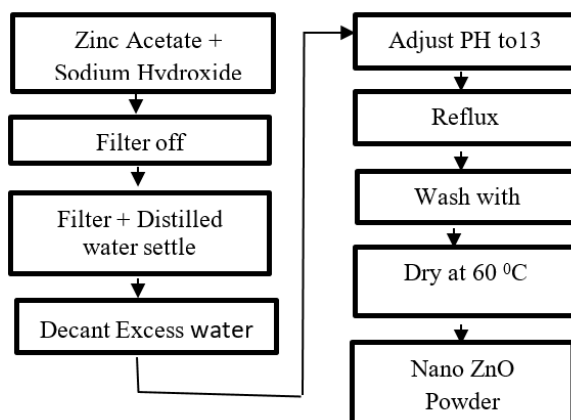


Fig. 3 Process Steps for preparation of Nano ZnO (Wahab et al. 2010) (Prasad et al. 2018).

Photographs of various stages observed in nano ZnO formation are presented in figure 4.

Characterization of Nano SiO₂ and Nano ZnO

Nano SiO₂ and Nano ZnO were characterized to assess the particle size and morphology by SEM-EDAX and XRD.

Micron SiO₂ and Micron ZnO

Purchased micron SiO₂ and micron ZnO were used as received. Micron SiO₂ and micron ZnO were not characterized by SEM and XRD.



(a) Nano ZnOH (b) Nano ZnO Gel (c) Dried Nano ZnO Powder

Fig. 4 Photographs of various stages observed in nano ZnO formation.

Preparation of PMMA Nano and Micron Composites

Brass split dies with multiple cavities (figure 5) were used to prepare cylindrical samples (180 mm long with 14 mm diameter). The reinforcement materials used were (a) nano SiO₂, (b) micron SiO₂, (c) nano ZnO and (d) micron ZnO. The matrix material used was dental grade PMMA (DPI make acrylic resin). Typical PMMA sample shown in figure 6.

PMMA composites were prepared by taking appropriate quantities of both polymer powder and the reinforcement, in an agate mortar and thoroughly mixing for 15 minutes. Polymerization was done by adding the specified quantity of the monomer. The mixture was allowed to go to the dough stage. The dough was thoroughly mixed and then evenly packed in the two halves of the split die, quickly closing the die and scraping excess material from the exteriors of the die. After a lapse of 10-15 minutes of time, the die was opened. PMMA composite samples were ejected from the die (packed and stored in a desiccator). Neat PMMA (without any reinforcement) samples were prepared following the same procedure.

Wear Testing of PMMA composites

Wear testing of neat PMMA and PMMA composites (reinforced with both nano and micron SiO₂ and ZnO) were carried out as per ASTM G99 standard using Pin-on Disc tribometer (Ducom make, model TR201 LE). To study the combined effects of wear testing parameters namely, applied normal load, speed of disc rotation, wear track diameter and composition of PMMA composites, simultaneously, L9 (3⁴) orthogonal array of experimental design was chosen. MINITAB (version 19) software to arrive at optimum set conditions.

Each sample, before testing, was subjected to a run in period testing (P. J. Blau 2013) (P. Blau 2005) for 10 minutes with a load of 8kg, speed of 900 rpm and wear track of 80mm. Sample for testing was selected as per the L9 (3⁴) (table 2.1) and wear test carried out (test duration of 15 minutes) on a randomly selected experimental run (column 1 of table 1).

Wear tests were conducted choosing a random experiment number (column 1 of table 1) not as per the serial number of column 1 of table 1.

Table 1 L₉ (3⁴) Orthogonal Array of Experimental Design.

| L ₉ (3 ⁴) Orthogonal array | | | | | Weight Loss | Specific wear rate | |
|---|-----------------|------------------|--------------------------------|------------------------|-----------------------------|--------------------|----|
| Independent Variables | | | | | Performance Parameter Value | P1 | P2 |
| Experiment Number | Variable-1 Load | Variable-2 Speed | Variable-3 Wear Track Diameter | Variable 4 Composition | | | |
| 1 | 1 | 1 | 1 | 1 | p1 | | |
| 2 | 1 | 2 | 2 | 2 | p2 | | |
| 3 | 1 | 3 | 3 | 3 | p3 | | |
| 4 | 2 | 1 | 2 | 3 | p4 | | |
| 5 | 2 | 2 | 3 | 1 | p5 | | |
| 6 | 2 | 3 | 1 | 2 | p6 | | |
| 7 | 3 | 1 | 3 | 2 | p7 | | |
| 8 | 3 | 2 | 1 | 3 | p8 | | |
| 9 | 3 | 3 | 2 | 1 | p9 | | |

1= low, 2= medium 3=high Load=4,6,8 kg Wear track diameter= 40,60,80 mm speed=500, 750, 900 rpm, Composition=of reinforcement (either nano or micron SiO₂ or ZnO as the case may be).



(a) Brass Split Die



(b) PMMA+5% Nano ZnO Samples

Fig. 5 Photographs of the process steps in preparing PMMA Composites.



(c) Neat PMMA Samples

Fig. 6 Photographs of neat PMMA sample.

Surface Roughness Measurement

Surface roughness parameters of the worn-out tested samples were measured using Confocal Microscope (Olympus make LEXT OLS4000).

Results and Discussion

Characterization of Nano Oxides by Scanning Electron Microscopy

Characterization Nano SiO₂

From the figure 7, it is seen that the nano SiO₂ particles are spherical in shape. There is a size distribution of the spherical particles. The smallest particle diameter ~100 nm while majority of the spherical SiO₂ particles have diameter greater than 200nm. The spherical particles would have grown to micron size during the calcination (Ashraf et al. 2018) (Garces, Espinal, and Suib 2012) (Deng et al. 2016). EDAX of nano SiO₂ (figure 8) indicate the presence constituent elements Si and O respectively.

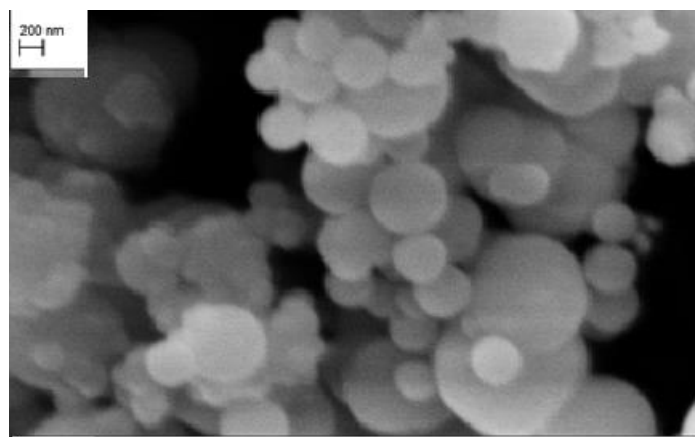


Fig. 7 SEM of nano SiO₂

Nano SiO₂ particles are spherical in shape have different diameters 50nm, 100nm and over 200nm in diameter.

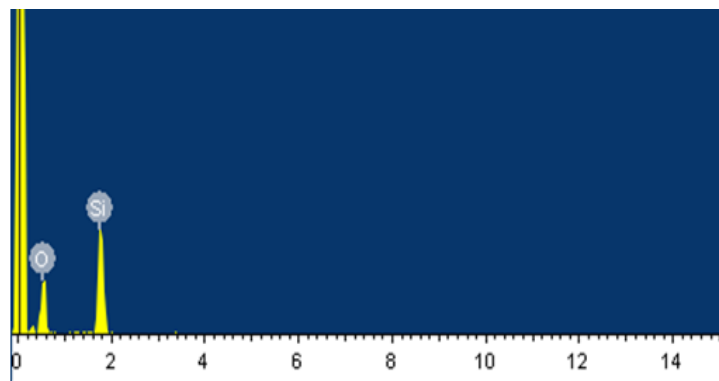


Fig. 8 EDAX of nano SiO₂

Characterization of Nano ZnO.

From the SEM image shown in figure 9 of nano ZnO, it may be observed that, the particles are irregular in form in the initial sol to gel stage. Subsequent calcination could have resulted in the agglomeration. Thickness of the platelet is around 20 nm. Several researchers reported the platelet morphology for nano ZnO is particles shape. ZnO particles have spherical, agglomerated irregular and platelet morphologies. Size of these particles range from nano to several hundred nano meters (3-500nm). Platelet morphology was reported by (Xu et al. 2013). Pariona, Nicolaza et al(Pariona et al. 2020) reported various morphologies. They

reported a platelet morphology. In the present studies, too, it is interesting to note that the platelet nano ZnO were obtained.

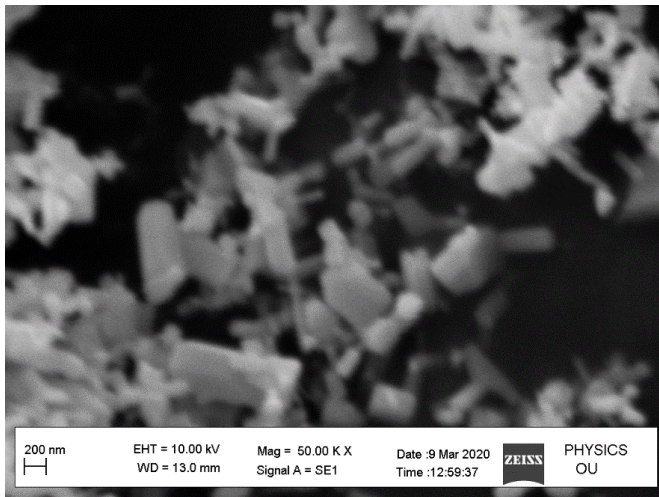


Fig. 9 SEM Image of Nano ZnO

EDAX of nano ZnO (figure 10) reveals the presence of constituent elements Zn and O respectively. (Carbon peak observed was from the conducting tape of carbon on which the nano ZnO was placed before gold sputtering prior to SEM examination)

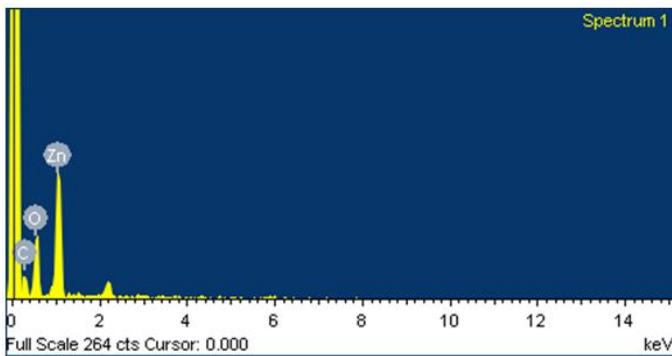


Fig. 10 EDAX of Nano ZnO

Characterization of Nano SiO₂ and Nano ZnO by X-Ray Diffraction

Characterization Nano SiO₂: XRD pattern of nano SiO₂ is shown in figure 11. This pattern reveals no sharp peaks with one broad peak (at 2 theta 23°). Similar XRD pattern was reported in literature (Choolaei et al. 2012).

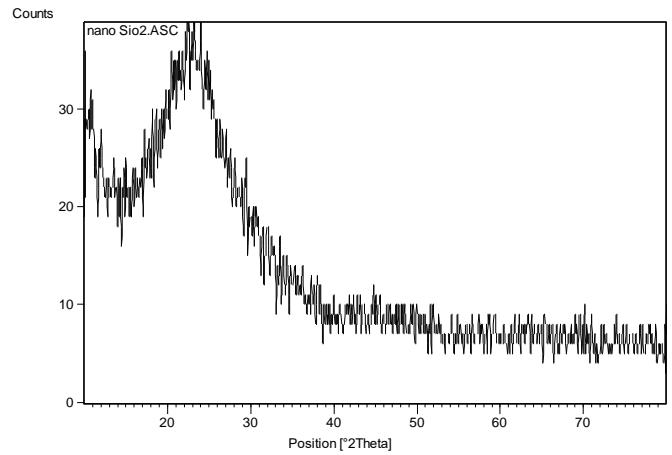


Fig. 11 XRD pattern of Nano SiO₂

Characterization Nano ZnO

XRD pattern of nano ZnO (figure 12) indicating the sharp peaks with a single with highest intensity at 23°. It is interesting to note that the pattern consists of sharp peaks with 100 % intensity occurring at 31.84°, 34.52°, 36.33°, 47.63°, 56.71°, 62.96°, 68.13°, and 69.18°. The platelet morphology of the nano ZnO corresponds to nano crystals of ZnO, the thickness of each platelet being 20 nm. Similar XRD pattern was reported in literature (Tarat et al. 2012) (Talam, Karumuri, and Gunnam 2012)

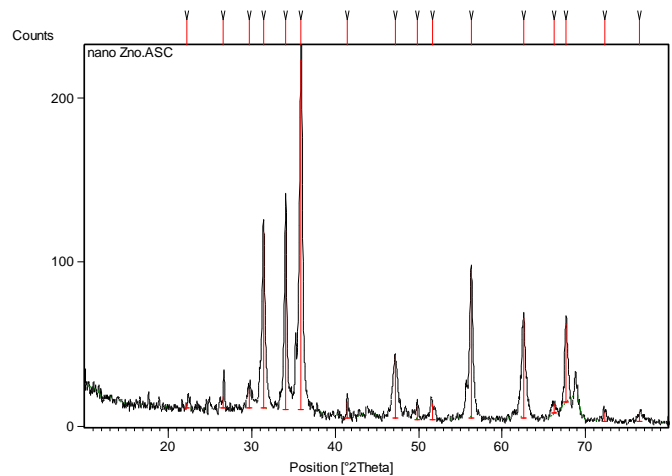


Fig. 12 XRD pattern of Nano ZnO

From the XRD pattern of nano ZnO we understand that prepared nano ZnO has nano crystalline structure having platelets morphology. In tables 2 and 3 we present a comparison of SEM and XRD nano SiO₂ and ZnO prepared.

Table 2 Comparison of SEM Analysis of Nano Oxides Prepared

| Material | Particle size | | | EDAX | | |
|-----------------------|--------------------------------|--------------------------------|---------------------------------------|----------|------------------------|---|
| | Our result Morphology and size | Literature Morphology and size | Reference | Present | Report from Literature | Reference |
| Nano SiO ₂ | Spherical ~100nm dia | 79.68 to 87.35nm. | (Azlina et al. 2016) | Si and O | Si O | (Rabizadeh and Allahkaram 2011) (Sekhar, Diwakar, and Madhavi 2019) |
| Nano ZnO | Platelets Thickness in 20 nm | platelet | (Xu et al. 2013)(Pariona et al. 2020) | | | |

Table 3 Comparison of XRD Patterns of Nano Oxides Prepared

| Material | No of Peaks | Peak with 100% intensity and 2θ | Reference |
|-----------------------|--------------------|---------------------------------|-----------|
| Nano SiO ₂ | 23 Max | - | - |
| Nano ZnO | 8 Peaks 36-100% | 34-50% | 31.5-45 |

In table 4 and 5 wear rates obtained from weight loss method as reported by Al- masoody[1] and as quoted by Bharat Bhusan [2] are presented for PMMA+ SiO₂, and PMMA+ ZnO composites respectively These values are the outcomes measured as per the planned L₉(3⁴) Orthogonal array of experimental design.

Table 4 Specific Wear Rate of Neat PMMA

| S N O | SPECIMEN COMPOSITION | WEAR TRACK DIA | LOADS (N) | SPEED | VOLUME | SLIDING DISTANCE | WEAR RATE (p1) | SPECIFIC WEAR RATE (m3/nm) (p2) |
|-------|----------------------|----------------|-----------|-------|--------|------------------|----------------|---------------------------------|
| 1 | NEAT PMMA | 0.04 | 4 | 500 | 0.08 | 400 | 15.7588 | 98.5 x10 ⁻⁵ |
| 2 | NEAT PMMA | 0.08 | 6 | 750 | 0.04 | 1200 | 3.9797 | 55.2 x10 ⁻⁵ |
| 3 | NEAT PMMA | 0.06 | 8 | 900 | 0.02 | 1080 | 1 | 11.6 x10 ⁻⁵ |

Table 5 Specific Wear Rate of PMMA+ SiO₂ Composites

| S N O | SPECIMEN COMPOSITION | WEAR TRACK DIA | LOADS (N) | SPEED | VOLUME | SLIDING DISTANCE | WEAR RATE (p1) | SPECIFIC WEAR RATE (m3/nm) (p2) |
|-------|----------------------|----------------|-----------|-------|--------|------------------|----------------|---------------------------------|
| 1 | NEAT PMMA | 0.04 | 4 | 500 | 0.08 | 400 | 15.7588 | 98.5x10 ⁻⁵ |
| 2 | NEAT PMMA | 0.08 | 6 | 750 | 0.04 | 1200 | 3.9797 | 55.2 x10 ⁻⁵ |
| 3 | NEAT PMMA | 0.06 | 8 | 900 | 0.02 | 1080 | 1 | 11.6 x10 ⁻⁵ |

| Nano SiO ₂ | | | | | | |
|-------------------------|----------------------|--------------|-------|---------|----------------|---------------------------------|
| Experiment No | SPECIMEN COMPOSITION | WEIGHT (Kgs) | | | WEAR RATE (p1) | SPECIFIC WEAR RATE (m3/nm) (p2) |
| | | BEFORE | AFTER | LOSS | | |
| 1 | 5% | 4.08 | 4.06 | 0.02 | 0.482759 | 3.0172E-07 |
| 2 | 10% | 3.95 | 3.94 | 0.01 | 0.497462 | 1.3818E-07 |
| 3 | 15% | 4.07 | 4.06 | 0.01 | 1.206897 | 2.0953E-07 |
| 4 | 15% | 4.06 | 4.04 | 0.02 | 0.970297 | 2.6953E-07 |
| 5 | 5% | 4.06 | 4.04 | 0.02 | 1.940594 | 2.6953E-07 |
| 6 | 10% | 3.94 | 3.92 | 0.02 | 1.5 | 3.4722E-07 |
| 7 | 10% | 3.92 | 3.9 | 0.02 | 2.010256 | 3.141E-07 |
| 8 | 15% | 4.04 | 4.01 | 0.03 | 2.199501 | 4.5823E-07 |
| 9 | 5% | 4.04 | 4.02 | 0.02 | 0.975124 | 1.1286E-07 |
| Micron SiO ₂ | | | | | | |
| Experiment No | SPECIMEN COMPOSITION | WEIGHT (g) | | | WEAR RATE (p1) | SPECIFIC WEAR RATE (m3/nm) (p2) |
| | | BEFORE | AFTER | LOSS | | |
| 1 | 5% | 3.95 | 3.94 | 0.00253 | 0.06297 | 3.93562E-08 |
| 2 | 10% | 4.39 | 4.37 | 0.00456 | 0.204334 | 5.67594E-08 |
| 3 | 15% | 4.28 | 4.23 | 0.01168 | 1.353262 | 2.34941E-07 |

| | | | | | | |
|---|-----|------|------|---------|----------|-------------|
| 4 | 15% | 4.23 | 4.21 | 0.00473 | 0.220122 | 6.1145E-08 |
| 5 | 5% | 3.94 | 3.9 | 0.01015 | 1.020435 | 1.41727E-07 |
| 6 | 10% | 4.37 | 4.34 | 0.00686 | 0.465048 | 1.0765E-07 |
| 7 | 10% | 4.34 | 4.3 | 0.00922 | 0.84021 | 1.31283E-07 |
| 8 | 15% | 4.21 | 4.18 | 0.00713 | 0.501199 | 1.04416E-07 |
| 9 | 5% | 3.9 | 3.88 | 0.00513 | 0.259054 | 2.99831E-08 |

Observation of specific wear rate values of p2 of neat PMMA and PMMA SiO₂ composites, it is noticeable that there is a 2-3-fold reduction in the specific wear rate.

Table 6 Specific Wear Rate of PMMA+ ZnO Composites

| Nano ZnO | | | | | | |
|---------------|----------------------|------------|-------|------|----------------|---------------------------------|
| Experiment No | SPECIMEN COMPOSITION | WEIGHT (g) | | | WEAR RATE (p1) | SPECIFIC WEAR RATE (m3/nm) (p2) |
| | | BEFORE | AFTER | LOSS | | |
| 1 | 5% | 3.94 | 3.88 | 0.06 | 1.515464 | 9.4716E-07 |
| 2 | 10% | 4.05 | 4 | 0.05 | 2.45 | 6.8056E-07 |
| 3 | 15% | 4.26 | 4.23 | 0.03 | 3.475177 | 6.0333E-07 |
| 4 | 15% | 4.23 | 4.21 | 0.02 | 0.931116 | 2.5864E-07 |
| 5 | 5% | 3.88 | 3.84 | 0.04 | 4.083333 | 5.6713E-07 |
| 6 | 10% | 4 | 3.99 | 0.01 | 0.736842 | 1.7057E-07 |
| 7 | 10% | 3.99 | 3.9 | 0.09 | 9.046 | 1.41 |

| | | | | | 154 | 35E-06 |
|---------------|----------------------|------------|-------|---------|----------------|---------------------------------|
| 8 | 15% | 4.21 | 4.12 | 0.09 | 6.42233 | 1.338E-06 |
| 9 | 5% | 3.84 | 3.8 | 0.04 | 2.063158 | 2.3879E-07 |
| Micron ZnO | | | | | | |
| Experiment No | SPECIMEN COMPOSITION | WEIGHT (g) | | | WEAR RATE (p1) | SPECIFIC WEAR RATE (m3/nm) (p2) |
| | | BEFORE | AFTER | LOSS | | |
| 1 | 5% | 4.25 | 4.24 | 0.00235 | 0.054384 | 3.399E-08 |
| 2 | 10% | 4.49 | 4.48 | 0.00223 | 0.048719 | 1.35332E-08 |
| 3 | 15% | 4.52 | 4.51 | 0.00221 | 0.048074 | 8.3462E-09 |
| 4 | 15% | 4.51 | 4.44 | 0.01552 | 2.398074 | 6.66132E-07 |
| 5 | 5% | 4.24 | 4.19 | 0.01179 | 1.37907 | 1.91537E-07 |
| 6 | 10% | 4.48 | 4.43 | 0.01116 | 1.234481 | 2.85759E-07 |
| 7 | 10% | 4.43 | 4.38 | 0.01129 | 1.262665 | 1.97291E-07 |
| 8 | 15% | 4.44 | 4.42 | 0.0045 | 0.199747 | 4.1614E-08 |
| 9 | 5% | 4.19 | 4.17 | 0.00477 | 0.224355 | 2.5967E-08 |

Similar observation on specific wear rate values of p2 of neat PMMA and PMMA ZnO composites, it is noticeable namely a 2-3-fold reduction.

In table 7 and 8 the response variable for the PMMA+nanoSiO₂ and PMMA+micronSiO₂ composites are present respectively. Response graphs of PMMA+nanoSiO₂ and PMMA+micronSiO₂ composites are presented in figures 13 and 14 respectively

Table 7 Response Table for Means PMMA+Nano SiO₂

| Level | Load | Speed | Wear Track | Composition |
|--------|---------|---------|------------|-------------|
| 1 | 0.01333 | 0.02000 | 0.02333 | 0.02000 |
| 2 | 0.02000 | 0.02000 | 0.01667 | 0.01667 |
| 3 | 0.02333 | 0.01667 | 0.01667 | 0.02000 |
| Delta* | 0.01000 | 0.00333 | 0.00667 | 0.00333 |
| Rank+ | 1 | 3.5 | 2 | 3.5 |

*Delta Measures the size of the effect by taking the difference between the highest and lowest characteristic average for a factor.

+Rank The ranks in a response table help you quickly identify which factors have the largest effect. The factor with the largest delta value is given rank 1, the factor with the second largest delta is given rank 2, and so on.

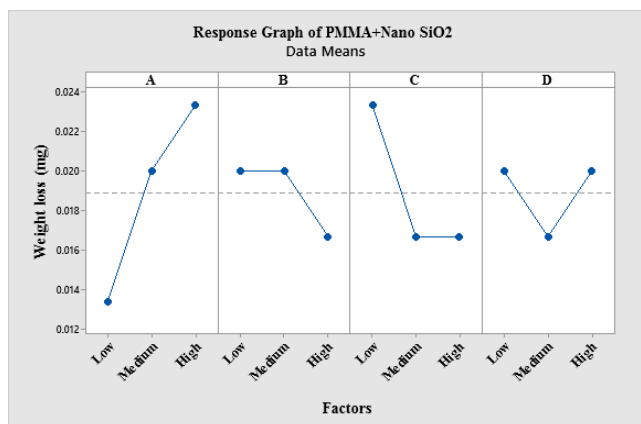


Fig. 13 Response Graph of PMMA+Nano SiO₂ L9(34) OA

For PMMA+ nano SiO₂, (From the figure 3.1) the lowest weight loss in the pin-on-disc wear test method with the factors and their levels studied in the present investigation (refer to table 3.1) is obtainable by running experiment with 4 kg load, speed of 750 or 900 rpm with track diameter 40 or 60 mm and composition 5% or 15 % nano SiO₂

Table 8 Response Table for Means PMMA+Micron SiO₂

| Level | Load | Speed | Wear Track | Composition |
|-------|----------|----------|------------|-------------|
| 1 | 0.006257 | 0.005493 | 0.005507 | 0.005937 |
| 2 | 0.007247 | 0.007280 | 0.004807 | 0.006880 |
| 3 | 0.007160 | 0.007890 | 0.010350 | 0.007847 |
| Delta | 0.000990 | 0.002397 | 0.005543 | 0.001910 |
| Rank | 4 | 2 | 1 | 3 |

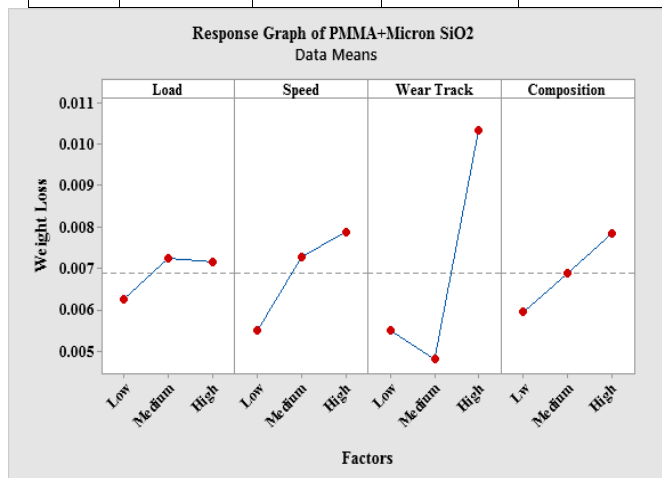


Fig. 14 Response Graph of PMMA+Micron SiO₂ L9(34) OA Experimental Data (reference table 7)

For PMMA+ micron SiO₂, (From the figure 14) the lowest weight loss in the pin-on-disc wear test method with the factors and their levels studied in the present investigation (refer to table 1.) is obtainable by running experiment with 4 kg load, speed of 750 rpm with track diameter 60 mm and composition 5% micron SiO₂

In table 9 and 10 are present the response variable for the PMMA+nanoSiO₂ and PMMA+micronSiO₂ composites respectively. Response graphs of PMMA+nano ZnO₂ and PMMA+micronZnO composites are presented in figures 15 and 16 respectively

Table 9 Response Table for Means PMMA+Nano ZnO

| Level | Load | Speed | Wear Track | Composition |
|-------|---------|---------|------------|-------------|
| 1 | 0.04667 | 0.05667 | 0.05333 | 0.04667 |
| 2 | 0.02333 | 0.06000 | 0.03667 | 0.05000 |
| 3 | 0.07333 | 0.02667 | 0.05333 | 0.04667 |
| Delta | 0.05000 | 0.03333 | 0.01667 | 0.00333 |
| Rank | 1 | 2 | 3 | 4 |

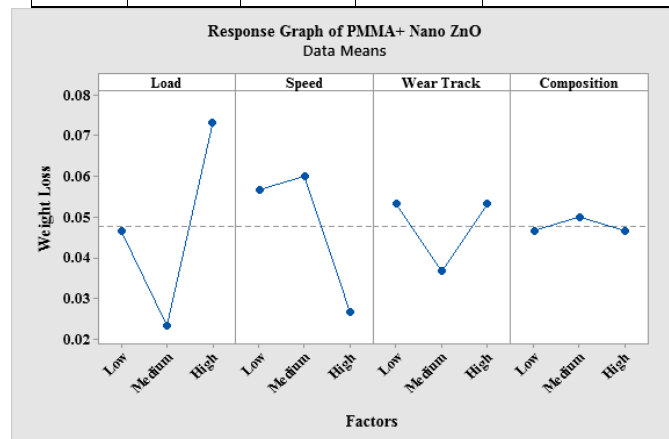


Fig. 15 Response Graph of PMMA+Nano ZnO L9(34) OA Experimental Data (reference table 3.4)

For PMMA+ nano ZnO,(From the figure 3.3) the lowest weight loss in the pin-on-disc wear test method with the factors and their levels studied in the present investigation (refer to table 3.1) is obtainable by running experiment with 6 kg load, speed of 900 rpm with track diameter of 60 mm and composition 5% or 15 % nano ZnO.

Table 10 Response Table for Means PMMA+Micron

| Level | Load | Speed | Wear Track | Composition |
|-------|---------|---------|------------|-------------|
| 1 | 0.05039 | 1.23837 | 0.49620 | 0.55260 |
| 2 | 1.67054 | 0.54251 | 0.89038 | 0.84862 |
| 3 | 0.56226 | 0.50230 | 0.89660 | 0.88196 |
| Delta | 1.62015 | 0.73607 | 0.40040 | 0.32936 |
| Rank | 1 | 2 | 3 | 4 |

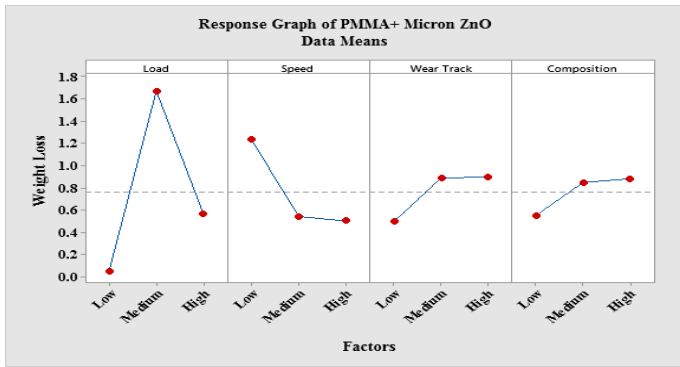


Fig. 16 Response Graph of PMMA+Micron ZnO L9(34) OA Experimental Data (reference table 3.4)

For PMMA+ micron ZnO, (From the figure 3.3) the lowest weight loss in the pin-on-disc wear test method with the factors and their levels studied in the present investigation (refer to tabel 3.1) is obtainable by running experiment with 4 kg load, speed of 900 rpm with track diameter of 40 mm and composition 5% micron ZnO.

From the above discussions on the minimum weight loss of PMMA composites reinforced with SiO₂ and ZnO, the following observations can be drawn as given in table 11

Table 11 Factors and levels that yield minimum weight loss

| Weight (N) | Speed (RPM) | Wear Track Diameter (mm) | Composition (wt %) | Weight loss (mg) |
|-------------------------------------|-------------|--------------------------|--------------------|------------------|
| PMMA+ Nano SiO₂ | | | | |
| 4 | 750 or 900 | 40 or 60 | 5 or 15 | 0.04-0.06 |
| PMMA+ Micron SiO₂ | | | | |
| 4 | 750 | 60 | 5 | 0.004-0.006 |
| PMMA+ Nano ZnO | | | | |
| 6 | 900 | 60 | 5 or 15 | 0.02-0.04 |
| PMMA+ Micron ZnO | | | | |
| 4 | 900 | 60 | 5 or 15 | 0.01-0.06 |

Probable reason for the observed lowest wear rate for PMMA nano composites may be ascribed to the morphology of the reinforcements, which could have acted as nano balls in 3 body tribocontact between polymer matrix and the steel substrate of the tribometer in case of Nano SiO₂. In case of PMMA-nano ZnO, the platelets would have formed a hard wear resistant layer at the specimen-substrate interface

locally, thereby preventing the further exposure of the fresh composite surface from further wear.

Surface Roughness Evaluation of Worn-out PMMA Composites

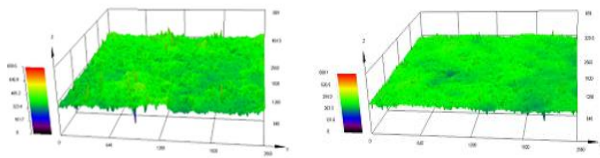
In tables 12 Ra and Rq, Sa and Sq values of worn out surfaces of PMMA+SiO₂ PMMA+ZnO Composites is present [(Trial no 5 of the L9 matrix (table no 3.1) load 6 N, speed 750 rpm, wear track 80 mm and 5 wt% of either SiO₂ or ZnO]

A roughness value can either be calculated on a profile (line) or on a surface (area). The profile roughness parameter (Ra, Rq,) are more common. The area roughness parameters (Sa, Sq,) give more significant values. There are many different roughness parameters in use, but Ra is by far the most common, though this is often for historical reasons and not for particular merit, as the early roughness meters could only measure Ra. Other common parameters include {Rz}, {Rq}, and { Rsk}. Some parameters are used only in certain industries or within certain countries. For example, the {Rk}{ Rk} family of parameters is used mainly for cylinder bore linings, and the Motif parameters are used primarily in the French automotive industry.[6] The MOTIF method provides a graphical evaluation of a surface profile without filtering waviness from roughness. A motif consists of the portion of a profile between two peaks and the final combinations of these motifs eliminate "insignificant" peaks and retains "significant" ones. Please note that Ra is a dimensional unit that can be micrometer or micro inch (Roughness n.d.).

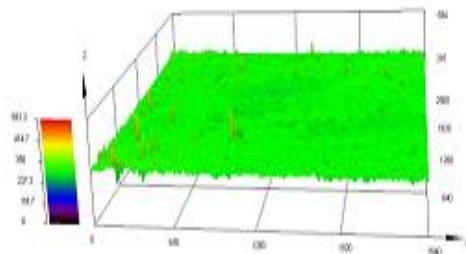
Table 12: Average Surface Roughness Value (Ra) and RMS Value of Surface Roughness (Rq) of components

| Sample | Ra | Rq | Sa | Sq |
|-------------------------------|-------|--------|--------|--------|
| Neat PMMA | 7.565 | 11.769 | 9.672 | 13.218 |
| PMMA+ Nano SiO ₂ | 7.065 | 9.453 | 9.228 | 10.917 |
| PMMA+ Micron SiO ₂ | 8.626 | 10.979 | 10.793 | 10.825 |
| PMMA+ Nano ZnO | 7.146 | 9.158 | 9.128 | 9.317 |
| PMMA+ Micron ZnO | 8.245 | 10.258 | 9.458 | 9.645 |

From the above table, it can be observed that neat PMMA has highest Ra, Rq values and lowest values are observed for PMMA+ nano ZnO composites. The values of Ra Rq for PMMA+SiO₂ are intermediate. Similar observations are noted for Sa and Sq values.

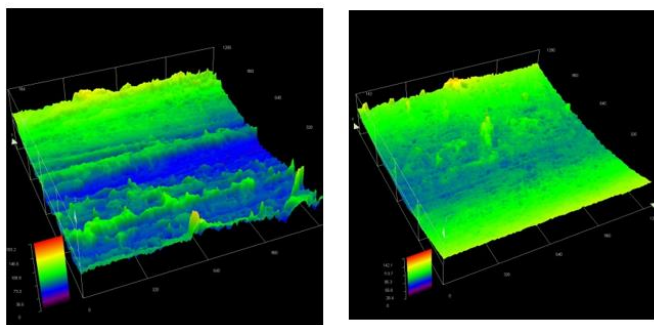


(a) (b)

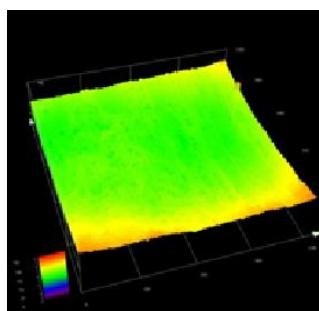


(c)

Fig. 16 (a) neat PMMA, (b) PMMA 5% Micron SiO₂ (c) PMMA-5% Nano SiO₂ composites



(a) (b)



(c)

Fig. 17 (a) neat PMMA, (b) PMMA 5% Micron ZnO (c) PMMA-5% Nano ZnO composites

In figures 3.5 and 3.6 typical areal (3D surface profilometric) roughness graphs of neat PMMA, PMMA+ SiO₂ and PMMA + ZnO composites are presented. The surface roughness values are complementing the surface roughness values (table 3.10)

Conclusions

| Sample | Ra | Rq | Sa | Sq |
|-------------------------------|-------|--------|--------|--------|
| Neat PMMA | 7.565 | 11.769 | 9.672 | 13.218 |
| PMMA+ Nano SiO ₂ | 7.065 | 9.453 | 9.228 | 10.917 |
| PMMA+ Micron SiO ₂ | 8.626 | 10.979 | 10.793 | 10.825 |
| PMMA+ Nano ZnO | 7.146 | 9.158 | 9.128 | 9.317 |
| PMMA+ Micron ZnO | 8.245 | 10.258 | 9.458 | 9.645 |

Nano SiO₂ had spherical morphology with ~100 nm diameter. ZnO had platelet morphology (average platelet thickness 20 nm). specific wear rate of PMMA SiO₂ composites, it is exhibited a 2-3-fold reduction in the specific wear rate over neat PMMA. Similar values for specific wear rate for PMMA-nano ZnO composites, namely 2-3 fold reduction has been noticed. For sample tested with applied normal load 6 N, disc speed 750 rpm, wear track diameter of 80 mm composition 5 wt % either SiO₂ or ZnO, Ra and Rq values PMMA-nano composites were lowest, followed by PMMA micron composites. The highest Ra, Rq were observed with neat PMMA.

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