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Study of Wear Characteristics of Ultra High Molecular Weight Polyethylene Nano Composite reinforced with Nano Al_2O_3

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ABSTRACT

An attempt has been made in the present work to study the wear characteristics along with the effect of parameters on the wear behaviour of UHMWPE nano composite reinforced with nano Al2O3. Also optimization of parameters and the wear values with respect to the parameters is attempted using Response Surface methodology through MINITAB software. Three levels of three different parameters are considered for the study wherein the design of experiments is done using Taguchi technique. Most importantly the effect of different reinforcement percentage of Al2O3 is discussed so as to know the behaviour of composite under dry wear conditions and the corresponding influence of the presence of reinforcement in terms of quantity. The results shows that the addition of nano alumina with increased percentages imparts superior wear properties into the composite which can also be seen through the surface profile. The statistical analysis also shows the importance and effects of parameters on the wear rate during the experimentation

Keywords: UHMWPE, Al2O3, Response surface methodology, Taguchi, MINITAB, Design of Experiments.

1.0 Introduction

UHMWPE were seen to be developed in the early 1950s has now gained much popularity due to their superior bio-compatible property [1-4], self-lubrication, chemically stable [6], and high wear and impact resistance property, which leads of path in biomedical as well as various engineering applications.During the absence of wet lubrication mating parts which are metallic in nature requires some kind of coating to resist wear and friction up to a greater extent and this is done by the researchers by using UHMWPE due to their terrific Tribological characteristics leading to reduced friction and high wear resistance. Researchers adopted a technique wherein nano and hybrid composites were synthesized using UHMWPE with reinforcements in the form of nano fillers like carbon nano tubes [7, 8], graphene [9], nano clay [10] and nano clay/CNT [11].Reinforcing UHMWPE with different wt. % of nano clay and conducting Tribological tests upon them showed that the failure of 1.5% reinforcement did not happen until 100000 cycles with a normal load of 9N with a linear speed of 0.1m/s, but at a load of 15N it failed instantly [10]. To overcome this again UHMWPE with 1.5 wt% nano clay and 1.5wt% CNT were combined to form hybrid composite and observed that the load bearing capacity is increased above 15N [11].Reinforcements are used along with the polymer to produce polyethylene which find their applications in many orthopaedic parts like knee joints and bone joints which leads to the synthesis of ultra-high molecular weight materials which can be used as bio-materials in practice. The first application of UHMWPE was in the joint replacement which has now found a vast variety of applications through different combination or composition with different materials leading to mechanical properties which are improved and superior.One of the application of UHMWPE is in the replacement of a component serving load bearing function in joint and joint surface implants, in particular the acetabular cups in the hip prosthesis [12]. Polyethylene and polypropylene when compared to UHMWPE has high wear rate and friction co efficient leading to lesser bio-compatibility and hence the UHMWPE finds its application due to reduced wear rate and co efficient of friction. Although UHMWPE has got good Tribological properties, its application is restricted to load bearing components and cannot be used in any other non-load bearing components or as a direct replacement for bones due to their low strength compared to other polymers. Even from the past decade the UHMWPE is used in place of fibre sutures. When the family of materials is considered alumina is one such material which finds wide usage [13]. High purity Al2O3 has got superior properties in terms of bio compatibility, wear resistance and corrosion resistance properties and hence can also be used as replacements. Its cyto compatibility is not damaged due to its bio-inert nature. It has got a wide range of properties including high modulus of elasticity, and compressive strength towards higher side. Due to these properties when added with UHMWPE, the mechanical as well as the Tribological properties are enhanced and the material altogether behaves bio-compatible. With the discovery of its benefits different manufacturers combined UHMWPE with Al2O3 to

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produce hip and knee joints for weight bearing function [14].UHMWPE is loaded with 0.5, 3, 5 and 10 wt. % alumina and the application was to protect steel surfaces. After conducting wear tests with sliding against SS ball it was found that the coatings with 3 AND 5% alumina were not failed even at 250000 cycles which shows the improved wear resistance which is more significant as compared to that of pure UHMWPE [15].

Therefore this work is based on developing and Tribological testing the UHMWPE reinforced with nano Al2O3, aiming at a new direction for the present scientific and engineering research on UHMWPE nano composites applied in the fields of bio-medical and industrial bearings.

2.0 Materials and Methods

Alumina (Al2O3) is used as reinforcement and it is synthesized using Sol-gel process in the laboratory environment [16]. UHMWPE composites were prepared along with the ceramic fillers using compression moulding technique which is described in the next section. After synthesis the specimen for wear testing were prepared according to the ASTM standard and pin-on-disc wear testing machine was used for testing wear and frictional force of material. DUCOM make wear monitor model TR201LE was used for acquiring wear data. 3 factors along with their 3 levels were considered for the study and design of experiments based on the taguchi technique is created for the experimentation purpose.

2.1 UHMWPE composite preparation

Initially, 30±2grams of polymer reinforcement mixture are placed in a bowl for dry mixing and then wet mixing with acetone (app 10-15ml) for a period of 30 mins for ensuring proper mixture of matrix and reinforcement. Wet mixture along with the bowl is kept in microwave oven and heated at 180°±2°C for a period of 10±2mins. After which the dried mixture is sealed and labelled in self-sealing plastic cover ready for compression moulding.

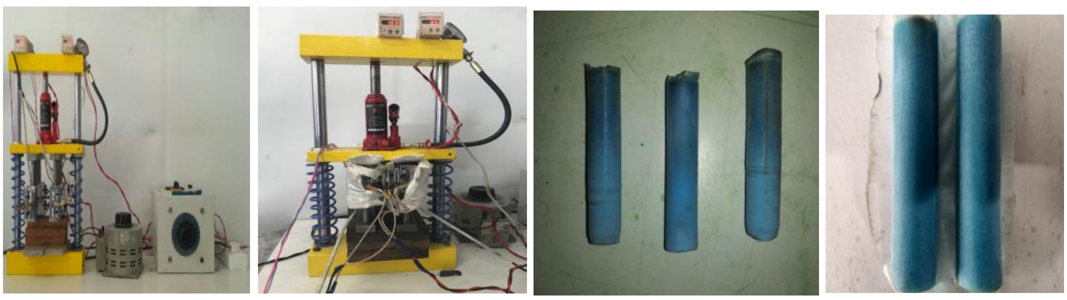
a **b** **c** **d**

Fig. 1(a-d): a. Weighing polymer and reinforcement, **b.** wet mixing, **c.** heating in micro wave oven, **d.** packaging and labelling

Now the dried mixture is transferred to closed multi cavity die. Then the hand pressing of plunger is done along with hammering. Controlled heating is provided with the insulated band heater. Temperature of the setup is measured using iron-constantan type thermocouple. Pressure gauge is used to monitor the pressure due to hand pump pressurized hydraulic jack. Time period was about 90±2mins and the temperature for moulding was about 130°C±2°C with a pressure of 190±5kg/cm^2 and for preventing loss of heat during the process wooden slabs were used to be placed underneath the die. Cera-wool blanket stitched with glass fibre was used to cover the band heater. Once the require temperature and pressure is reached the process is continued for the required time and then the power supply was cut-off. Once the die reaches the room temperature the specimens were removed and bagged with naming for further testing. Once the temperature reaches 90°C the position was reversed.

a **b** **c** **d** **e**

Fig. 2 (a-e): a. multi cavity die **b.** band heater **c.** pressure gauge **d.** cera-wool blanket stiched with glass fibre **e.** temperature indicator



a **b**

c **d**

Fig. 3(a-d): a. Compression moulding setup **b.** reversed die **c.** specimen without reversing the die **d.** specimen with reversed die

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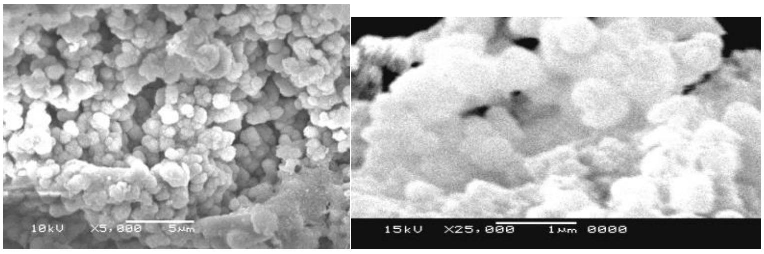
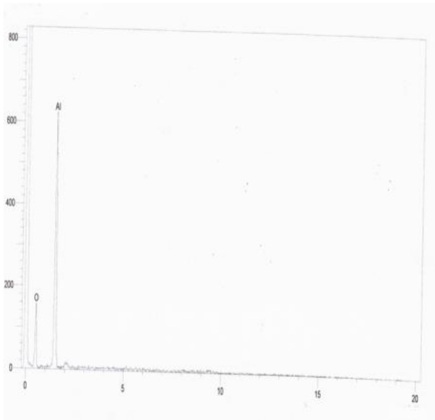
2.2 Microstructural characterization of nano Alumina powder produced by Sol-gel method



a b

Fig. 4(a-b): a. taken at a magnification of 5,000X, **b.** taken at a magnification of 25,000X

The micrographs of nano alumina were observed under SEM at 5000X and 25000X magnification. The below figures show the observations made. From a. agglomeration of particles (alumina) can be seen and the particles are observed to be spherical in shape. The agglomerated secondary particle was found to be 100nm approximately. Even at a higher magnification (25000X) small primary particles are observed. The size of these small primary particles were observed to be 2nm-5nm which then agglomerates into secondary particles. The size of the secondary particles were observed to be 70nm-100nm following the observations made by the other researchers which satisfies the definition of nano particles.



Energy, kilo electron volts (Kev)

Fig. 5: EDAX Pattern of Nano Alumina (Al_2O_3) Powder

For exploring the chemical purity of alumina particles produced using sol-gel process Energy Dispersive X-ray Analysis was conducted and the result photograph of analysis is showed above. The pattern shows the presence of Al and O and clearly we can observe that there are no impurities present. Hence the sol-gel method of alumina synthesis can be applied to get alumina with no impurities.

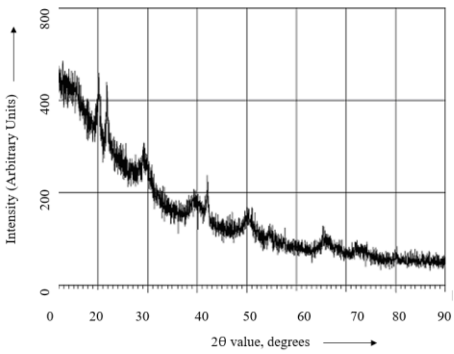


Fig. 6: XRD Pattern of Nano Alumina Powder

The X-ray diffraction pattern of alumina as shown above shows only broad diffuse peaks and no sharp peaks (as in crystalline solid materials). The particles here are amorphous to the incidence of X-rays results in the observation of broad diffuse peak and not the sharp peaks in line with the literature review done and satisfies the XRD analysis of nano particles as well.

2.3 Wear Testing

After the preparation of specimens the wear teat is conducted using pin-on-disc wear testing machine for testing wear and frictional force of material. DUCOM make wear monitor model TR201LE was used for acquiring wear data. Specimens were made for 45mm x 3mm cylindrical rod for the test according to the ASTM standards. For conduction of experiments taguchi method of DOE is used. The factors and their levels are shown in the below table.

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Table 1: Factors and their levels considered for wear test

Factors	Level 1	Level 2	Level 3
Load (kg)	5	7.5	10
Track radius (mm)	40	60	70
Composition (%)	2	6	10

2.4 Procedure

Pin-on-Disc operates on the following principle: a flat, pin or ball is loaded onto a test sample with a precisely known weight and at a specific position from the center to create a circular wear track as the bottom plate rotates. Wear rates for the pin and the disk are calculated from the volume of material lost during the test.

In the present investigation, pin is the UHMWPE composite cylinder and the disc is Φ100 mm x 4 ± 0.5mm thick EN31 steel (having hardness of 63 HRc disc).

a **b** **c** **d** **e**

Fig. 7(a-e): a. sample **b.** Sample holder of wear testing machine **c.** Wear testing monitor **d.** Top view of wear testing machine **e.** Weighing machine

2.4.1 Test Parameters for Pin-on-disc Dry wear test

Table 2: Test parameters for Pin-on-disc Dry wear test

Sl No	Test Parameter	Value
1	Diameter of the pin specimen, mm	13
2	Length of the pin specimen, mm	50±0.5
3	Track radius, mm	40, 60, 70
4	Revolutions per minute of the disc, rpm	1000
5	Applied normal load (kg)	5.00 (49.05N) 7.50 (73.575) 10.00 (98.1N)
6	Duration of test (minutes)	30

3.0 Results and Discussions

The influence of factors (parameters) in wear test and their effects are studied using MINITAB software wherein the factors with their levels are randomized and sequence is obtained for the experiments to be carried out. L27 array is selected and 27 number of experiments are conducted with the given set of factors and their levels. The below table shows the factorial design and experimental data in terms of factors and their corresponding results in terms of wear rate.

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Table 3: L27 factorial design

Applied normal load Factor-1 (N)	Track radius Factor-2 (mm)	Compositio n Factor-3 (%)	Weight of sample before test (g)	Weight of sample after test (g)	Wear rate (µm)
10	40	2	2.43	2.40	69
10	60	6	7.32	7.01	2026
10	70	2	2.40	2.19	1704
5	40	2	7.79	7.70	24
7.5	60	10	7.49	7.33	27
10	60	10	7.89	7.67	2499
10	70	10	7.40	7.21	1345
5	70	6	6.64	6.64	21
7.5	40	2	2.21	2.10	515
7.5	40	10	7.52	7.50	423
7.5	60	2	2.53	2.42	646
7.5	60	6	7.22	6.99	189
10	60	2	2.48	2.38	571
7.5	70	6	6.88	6.80	35
7.5	70	10	7.40	7.36	269
7.5	70	2	2.50	2.30	1868
5	60	10	7.99	7.96	202
5	60	6	6.97	6.93	20
5	70	2	2.21	2.21	31
10	70	6	6.92	6.90	599
10	40	10	7.93	7.92	412
5	60	2	2.18	2.16	64
5	70	10	7.26	7.26	43
7.5	40	6	7.32	7.30	96
5	40	10	7.50	7.44	263
10	40	6	7.12	7.00	1314
5	40	6	7.14	7.13	63

For the obtained values of wear rate the effects of parameter is studied using S/N graph as shown below. The response table as well as the graph clearly show that the major influencing is the load and as the load is increased the wear rate also increases. The second parameter responsible for the variation in wear rate is the composition and then the track radius.

Table 4: Response Table for Signal to Noise Ratios

Level	L	TR	C
1	34.35	45.54	47.33
2	46.85	47.54	43.17
3	58.16	46.29	48.86
Delta	23.80	2.00	5.69
Rank	1	3	2

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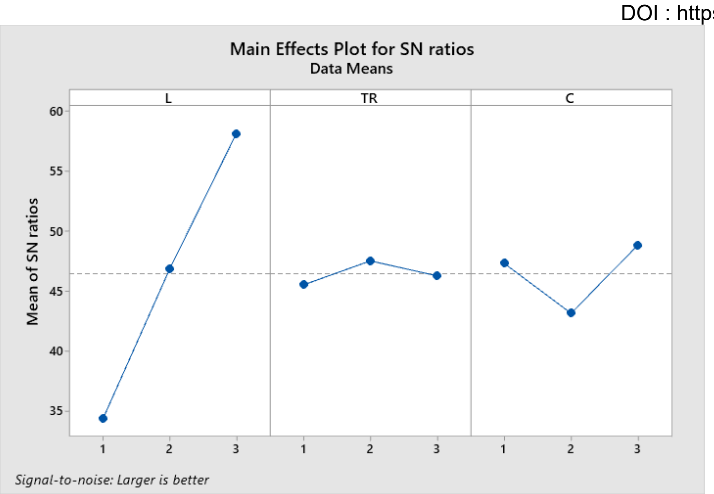

Fig. 8: SN graph for the parameters v/s response

When the dry sliding wear rate of these composites are considered, when the load is increased a hard layer is formed due to an increase in the temperature. It is observed that the nano alumina particles are deep rooted into the matrix because of which a thin film is formed on the surface which is obviously affected by the increase in load and the same can be viewed as the debris attached to the disc after a period of time. As in the graph shown above the wear rate reduces due to the increase in the reinforcements as they tend to reduce temperature due to the reduction in friction which in turn reduces recrystallization at the worn out surfaces.

The adequacy as well as significance of the model can be proven using the ANOVA with 5% significance level.

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	5025975	558442	1.12	0.402
Linear	3	296422	98807	0.20	0.897
A	1	124069	124069	0.25	0.625
B	1	7775	7775	0.02	0.902
C	1	179693	179693	0.36	0.557
Square	3	1665572	555191	1.11	0.372
A*A	1	1189506	1189506	2.38	0.141
B*B	1	428271	428271	0.86	0.368
C*C	1	114785	114785	0.23	0.638
2-Way Interaction	3	1348951	449650	0.90	0.462
A*B	1	220795	220795	0.44	0.515
A*C	1	20907	20907	0.04	0.840
B*C	1	1083523	1083523	2.17	0.159
Error	17	8501577	500093		
Lack-of-Fit	6	680407	113401	0.16	0.983
Pure Error	11	7821170	711015		
Total	26	13527552			

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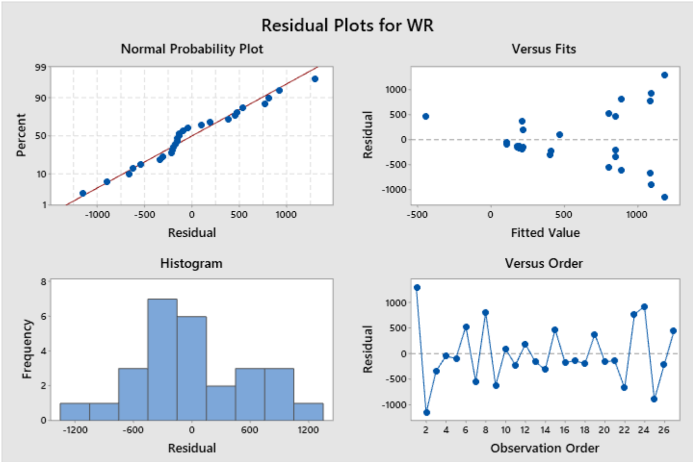


Fig. 9: Residual plots for wear rate

From the results above it is seen that the load is a major factor effecting the wear rate and it is because of the increased friction due to which there is an increase in the material loss as well and due to the increased friction the wear rate is also increased. The interaction of the parameters can also be seen in the table. By seeing the residual plots, there is no deviation seen in the values and a particular pattern is followed without any abnormality in the distribution of errors with any unusual structure or even the pattern. This proves the competency of the model generated.

Worn-out surfaces after wear test were subjected to examination under 3D surface profilometry. The specimen was cut in to 2 pieces and a spot for analysis (1mm x 1mm) is marked after which the measurements for surface roughness were done using Confocal Microscope (Olympus make LEXT OLS4000). A sample for surface roughness measurement has been given below. Once the scanning of the marked part is completed and the images are saved, the further measurement of surface roughness parameters is done and tabulated as in the below table.

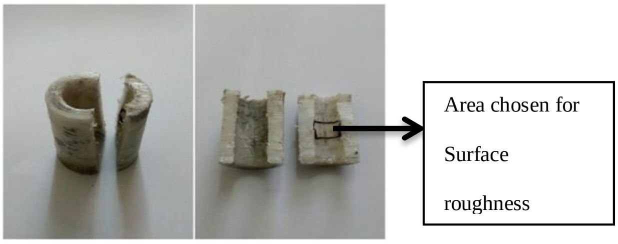
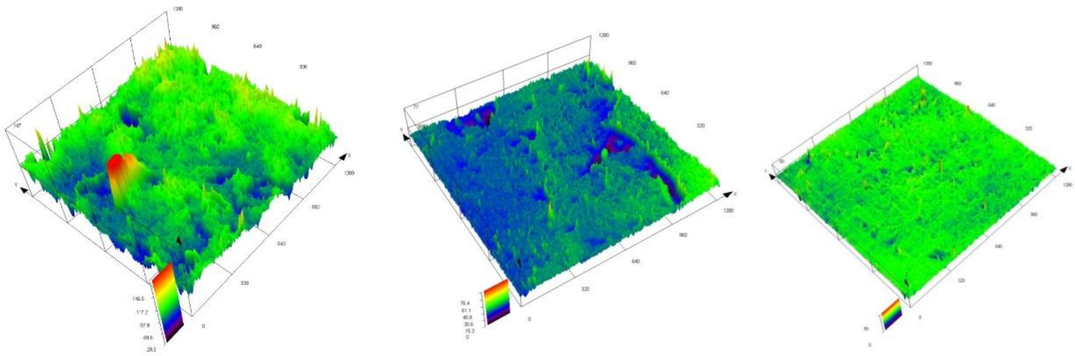


Fig. 10: Surface roughness measurement



a b c

Fig. 10 (a-c): a. 2% nano Al2O3+UHMWPE **b.** 6% nano Al2O3+UHMWPE **c.** 10% nanoAl2O3+UHMWPE

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Table 5: Surface roughness measurement of UHMWPE and its composites

Composites	Surface profile			
	Sa (µm)		Sq (µm)	
	Without testing	With testing	Without testing	With testing
Pure UHMWPE	2.693	7.935	3.269	9.546
UHMWPE + 2% Al2O3	4.276	4.569	5.276	5.287
UHMWPE + 6% Al2O3	4.098	4.819	5.983	5.812
UHMWPE + 10% Al2O3	3.162	3.459	4.166	4.227

The profiles and the table of values for surface roughness show that the smooth profile is obtained for 10wt% addition of alumina and which may be the result of wear debris dislodgment which in turn may act as lubricant and not the abrasive particles.

5.0 CONCLUSION

An attempt of studying the wear behaviour and the parameters responsible for the increase in wear is made in this study with surface roughness measurement with different composition. The following conclusions are made with respect to the study conducted:

The addition of alumina into the UHMWPE has led to a superior wear property inculcated in the composite which can be seen through the results yielding less wear rate with higher composition and so the reinforcement in terms of nano alumina becomes major factor in reducing the wear rate. Wear loss and wear rate can be seen minimum when the load, track radius and composition are 5kg, 70mm and 6wt% respectively with a minimum wear value of 21µm. With the analysis using response surface methodology it is seen that the major factor influencing the wear rate is the load followed by the wt% of reinforcement. Hence by knowing this the design and application area of the proposed composite can be established. For even better results in terms of wear properties any other lubricating agents like MoS2, WS2, graphite, PTFE, Ag, hBN can be used as other reinforcement along with Al2O3.

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