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Dry Sliding Wear Behavior of Hydroxyapatite Reinforced Az91d Mg Alloy Metal Matrix Composites

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The present work aims to increase the tribological performance of magnesium alloy matrix material reinforced with hydroxyapatite ceramic particulates. Reinforcement of hydroxyapatite particulate effects on wear behavior of AZ91D mg alloy was analyzed using Taguchi technique have been reported. The average grain size of 63μm reinforcement particulates were used to fabricate the composites by stir casting process. Dry sliding wear behaviour of various weight percentages of (2, 3 & 4 wt%) hydroxyapatite reinforced composites were tested on pin-on-disk machine using different loads of 5, 10, 15 and 20N, sliding velocity of 0.5, 1, 1.5 and 2.0 m/s at a sliding distance of 500, 750,1000, 1250 mm respectively. DOE approach using Taguchi technique was engaged to analyze the wear behavior and coefficient of frication of the composites. Analysis of variance and Signal-to-noise ratio was used to inspect the influence parameters for wear rate and coefficient of Friction.

1. Introduction:

Over the conventional metals and alloys, the metal matrix composites were achieving their importance to increase the strength in various applications like automobile, aerospace and biomedical applications. The tribological and mechanical properties were increased by reinforce the hard ceramic particulates in matrix material which were uniformly distributed in the matrix material. The composite materials are the emerging material that gives an opportunity to develop the tailor materials as per the required applications. Fundamentally composite materials are different from the conventional materials with their homogeneity point of view. Metal matrix composites were manufactured by reinforcing the ceramic particulates in matrix melt by stir casting technique. Moreover, mechanical properties like specific strength and modulus of elasticity had shown them as good materials for various engineering applications where in sliding contact predictable.

Magnesium metal matrix composites shows potential material for industrial, structural as well as biomedical applications owed to their properties like high specific strength, hardness, damping capacity with low density. AZ91D Mg alloy is one of the extensively used materials, which posse's excellent mechanical as well as physical properties. Formerly, if a material is selected for suitable application the degradation testing was required to examine the effects of material in predictable environment. The degradation of material was occurred due to wear, fatigue and corrosion [1-3]. Significant studies have been carried out to identify friction and wear behavior of AZ91D alloy [4-6]. New era for development of composite material this increases the mechanical properties as compared to alloy material. The present work made an attempt to address the wear behavior of AZ91D/ hydroxyapatite metal matrix composite material.

Wear is a major concern of fabricated composites subjected to sliding action in automobile and biomedical applications. The friction factor may possibly influence the tribological behavior of sliding parts [7-9]. Based on the temperature developed between contact surfaces and series of vibrant changes like abrasion, adhesion, delamination, oxidation and melting may occur. Lot of research work was gone on SiC particulate reinforced metal matrix composites via experimental design methods. Basavarajappa et al, has been investigated on Al2219-SiC and Al2219-SiC graphite materials by using Taguchi and ANOVA techniques to characterize the significant factors. The results present significant effects on wear by the factors of sliding velocity, sliding distance and load [10]. Y. Sahin et al was reported a set of experiment values done by combination of orthogonal arrays and ANOVA method to analyze the tribological behaviour of Al 2014 with 10 wt% of SiC composite [11]. Kavimani et al was conducted the experimental investigation on SiC reinforced with AZ31 magnesium matrix material and reported tribological behavior also studied the dry sliding wear behavior of the developed composite materials by using Taguchi technique [12].

A very few studies have carried out to understand the sliding wear and friction behavior of the AZ91D matrix material but significant amount of ambiguity still exists. Furthermore, comparatively very limited work was done on AZ91D alloy by using Taguchi method. For this reason, an attempt was made in the present work to investigate the influence of friction factor and wear rate of the composites on dry sliding wear by using Taguchi technique. This is the sequential paper published by the authors where the experimental analysis was already carried out.

2. Materials and Methods

2.1 Materials

AZ91D magnesium alloy was selected as base matrix material. It has good mechanical properties and wear resistance so as it is preferred in automobile, aerospace and biomedical applications. The chemical composition of AZ91D magnesium alloy used as matrix material in the current research work and chemical composition was presented in table 1. Hydroxyapatite particulates used as reinforcement were purchased from Aman scientific chemicals supplier and having particle size of 63 μm.

Table 1: Chemical composition of AZ91D Mg alloy

Elements	Al	Zn	Mn	Cu	Fe	Ni	Si	Mg
Wt%	8.89	0.78	0.20	0.002	0.002	<0.001	<0.01	Rest

2.2 Taguchi Technique

Taguchi technique entails the decline of dissimilarity in process through robust design of experiment and it is a controlling tool for the design of high-quality system. It minimizes the experimentation time period and cost of the experimentation to the other conventional models. In Design of experiments Orthogonal arrays and signal to noise ratio (S/N ratio) are the essential tools to optimize the experimentation. S/N ratios exhibit less quality attribute variation due to uncontrollable factors. Taguchi technique generates typical orthogonal arrays to find the optimum results of several factors at yield response.

2.3 Fabrication of composites

Stir casting technique was used to fabricate the AZ91D/ hydroxyapatite metal matrix composite. Figure 1 shows the stir casting equipment used for the study. AZ91D magnesium alloy was taken as matrix material while 2, 3&4 Wt% of Hydroxyapatite ceramic particles was taken as reinforcement. Additional 10 Wt% of matrix material was chosen for reducing material losses such as slag removal and oxidation. Reinforcement was preheated up to 400 °C to remove perspiration substance [13] and was incorporated in molten metal in suitable proportions as per calculated hierarchy. Vortex was created by using conventional stirrer at 700 rpm the pre heated reinforcement particulates were introduced in the vortex for uniform distribution of particulates in the matrix material. Inert environment was created by using argon gas to shield the molten metal to prevent oxidation and covered flux is used to remove the unwanted gases in the molten metal.

Figure 1. Stirr casting set up and composite fingers

2.4 Wear test

The wear test specimens were machined by using wire cut EDM and tested under dry sliding condition as per ASTM G99 standards using pin on disc sliding wear testing machine [14]. EN8 steel disk of diameter 200 mm was used as workface on which the test specimen slides. Wear volume loss method was adopted in the present research in which the specimens of 3mm diameter and 40mm length were used for conducting the test. Sliding disk was cleaned with ethanol to remove the unwanted traces on the surface at every test before and after conduction. The specimens were metallographically polished, cleaned with ethanol on work surface for effective results. Heist loss of wear rate was calculated by multiplying the area of cross section of test specimen with LVDT reading. The wear rate and coefficient of friction of AZ91D/ hydroxyapatite composites were continuously observed and recorded throughout sliding test.

The wear rate was calculated by using the following formula [15,16]:

$$Wear\ rate = \frac{Volume\ loss\ (\Delta V)}{Sliding\ distance\ (SD)}$$

Sliding distance (km) = 2πr x rpm x time in minutes, where r is radius of the disk.

Figure 2 Wear samples Machined by Wire EDM

2.5 Experimental Design

In Taguchi technique L16 orthogonal array was selected in Design of experiments technique. Design of experiments technique was engaged for planning the number of experimental trails and moreover to optimize the response variables. Four levels selected for four controlling factors such as % of reinforcement (0, 2, 3 &4 wt %), load (5, 10, 15&20N), Sliding velocity (0.5, 1, 1.5 & 2 m/s) and sliding distance (500, 750, 1000 & 1250) were considered. Table 2 represents the selected controlling factors and levels. Generally, the quality option is separated in three types such as Larger the better, smaller the better and nominal better [17, 18]. In this work smaller the better was adopted to investigate the wear performance and coefficient of friction [19]. The objective of the design is to minimize the coefficient of friction and wear rate.

Table 2: Controlling factors and their levels.

Factors	Symbol	Level 1	Level 2	Level 3	Level 4
Hydroxyapatite (wt %)	A	0	2	3	4
Load (N)	B	5	10	15	20
Sliding velocity (m/s)	C	0.5	1	1.5	2.0
Sliding distance (m)	D	500	750	1000	1250

3. Results and Discussion:

Minitab 18 evaluated the eminent characteristics and optimizes the experimental value into Signal to Noise ratio [20]. Table 3 presents the results of experimental values analyze in signal to noise ratio for wear rate and coefficient of friction by using L16 orthogonal array.

Table 3: Experimental values as per L16 orthogonal array and S/N ratio for wear rate and COF

Exp no	% Re	Load N	SV m/s	SD mm	Wear rate µm	COF	S/N ratio of wear	S/N ratio of COF
1	0	5	0.5	500	0.00365	0.854	-18.5194	1.37084
2	0	10	1.0	750	0.00384	0.795	-18.3134	1.99266
3	0	15	1.5	1000	0.00391	0.912	-18.1565	0.80010
4	0	20	2.0	1250	0.00462	0.915	-16.7072	0.77158

5	2	5	1.0	1000	0.00362	0.732	-18.6595	2.70978
6	2	10	0.5	1250	0.00343	0.705	-18.6125	3.03622
7	2	15	2.0	500	0.00412	0.812	-18.6830	1.80888
8	2	20	1.5	750	0.00431	0.766	-18.7304	2.31542
9	3	5	1.5	1250	0.00373	0.512	-18.3587	5.81460
10	3	10	2.0	1000	0.00531	0.495	-18.8019	6.10790
11	3	15	0.5	750	0.00361	0.363	-17.7021	8.80187
12	3	20	1.0	500	0.00391	0.420	-17.3105	7.53501
13	4	5	2.0	750	0.00484	0.666	-17.7021	3.53052
14	4	10	1.5	500	0.00491	0.631	-17.3105	3.99941
15	4	15	1.0	1250	0.00435	0.712	-16.3031	2.95040
16	4	20	0.5	1000	0.00384	0.674	-16.1784	3.42680

The controlling factors play a vital role in various procedures to influence the composite performance. Taguchi technique assesses the experimentation along with signal to noise ratio by means of numerical method with the intention of effect of reinforcement that visually recognized process parameter [21]. Wear rate of material is usually influenced by various parameters like reinforcement, load, sliding velocity and sliding distance. The above table gives the mean wear rate and means of COF of hydroxyapatite reinforced composites. It gives the mean of mean estimation of composites affected by various controlling factors [22].

The significance of controlled factors such as reinforcement, load, sliding velocity and sliding distance were investigate the optimum parameters of wear rate and COF along with signal to noise ratio reactions was presented in figure 3&4. Process parameters commencing that typically wear lose control by reinforcement, sliding velocity, and load in the falling order. The signal to noise ratio mean effect plot for wear rate was presented in figure 3 and indicated that addition of tricalcium phosphate reinforcement particles at 2 wt% shows the less wear rate. Increasing the reinforcement percentages in the composites reduces the bonding strength of matrix material caused to reduce the wear resistance. hydroxyapatite at 2wt % particle shows high optimum value at a load of 5N, sliding velocity of 1.0 m/s and sliding distance of 1000 m.

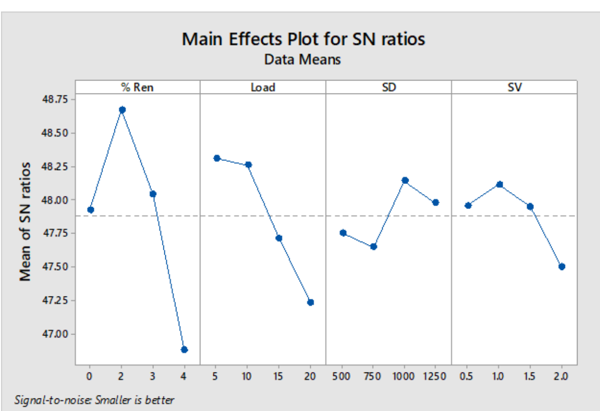


Figure 3: S/N ratio response curve for wear rate

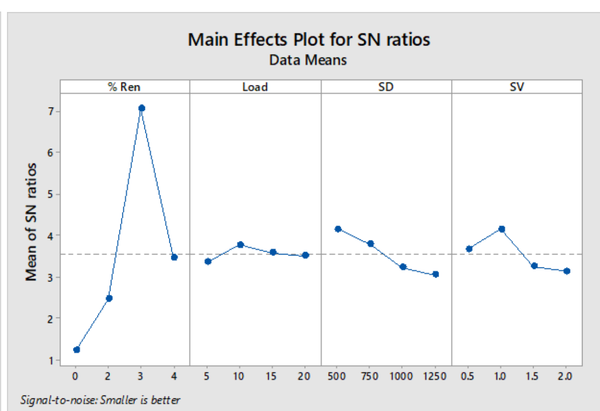


Figure 4: S/N ratio response curve for COF

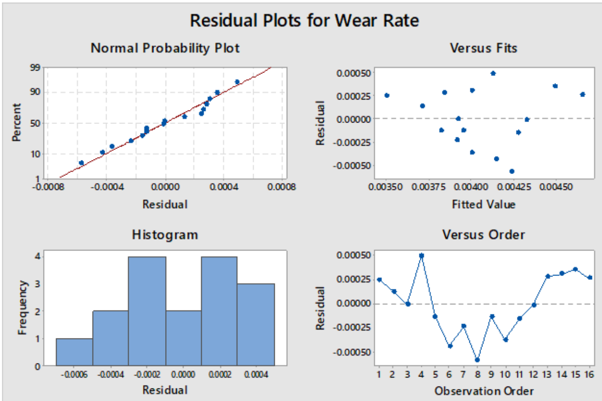


Figure 5: Residual plot for wear rate

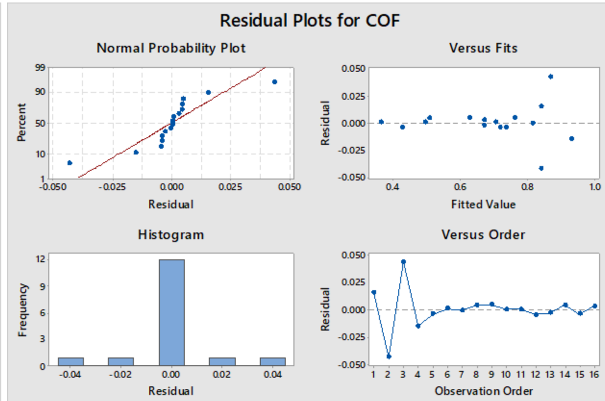


Figure 6: Residual plot for COF

Figure 4 presents the levels and optimum factors for COF. Which includes the hydroxyapatite reinforcement at 2 wt%, load 10N, sliding velocity of 1.0m/s and sliding distance 500m gives the optimum results. It is found that the high coefficient of friction was achieved at base material and 3, 4 wt% of composites. The signal to noise ratio response was determined by using smaller is better option. Figure 5&6 exhibits the fitted values in residual plots for wear rate and coefficient of friction.

3.1 Analysis of Variance:

ANOVA analysis is used to identify the importance of distinct process parameters. Table 4 presents the ANOVA responses for wear rate. The F column in table 4&5 indicates the process parameter and degrees of freedom for each variable. F-test is conducted to find out the significant parameter which affects the wear rate of magnesium matrix tricalcium phosphate composite [23]. The 96% R-square value obtained because of the P value is 0.044. The predictable reaction fits well by means of tentatively obtained reactions. If the P-value above 0.05 indicates that the model is statistically remarkable. By using ANOVA analysis along with S/N ratio, which is presents the hydroxyapatite wt% had a maximum influence to reduce the wear rate and COF.

Table 4**:** ANOVA response for Wear Rate

Source	DF	Seq SS	Adj SS	Adj MS	F-Value	P-Value
Hydroxyapatite wt%	3	2.498	9.248	9.7494	3.06	0.044
Load (N)	3	4.221	4.220	1.407	5.19	0.108
Sliding Distance (m)	3	5.106	5.106	1.7020	0.50	0.495
Sliding Velocity (m/s)	3	13.472	13.742	4.508	0.98	0.344
Error	3	2.335	2.335	0.7848		
Total	15					

Table 5: ANOVA response for COF

Source	DF	Seq SS	Adj SS	Adj MS	F-Value	P-Value
Hydroxyapatite wt%	3	0.3965	0.3968	0.1322	92.46	0.001
Load (N)	3	0.0054	0.0056	0.0018	1.32	0.416

Sliding Distance (m)	3	0.0151	0.0154	0.0050	3.65	0.162
Sliding Velocity (m/s)	3	0.0131	0.0131	0.0043	3.07	0.202
Error	3	0.0044	0.0043	0.0014		
Total	15	0.4348				

3.2 Multiple linear regression models:

The linear regression equation (1) showing that the factors allied with hydroxyapatite wt% (A3, A4), load (B1, B4), sliding velocity (C2, C4) and sliding distance (D1, D3) are negative. Wear rate of hydroxyapatite reinforced composites decreased with decreased load and sliding distance.

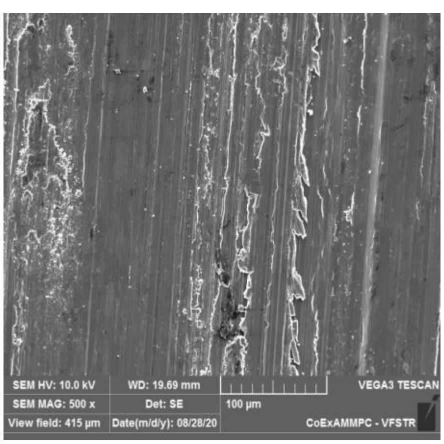
$$\text{Wear Rate} = 5.602+1.425A_1+0.324A_2-0.201A_3-1.505A_4-0.116B_1+0.224B_2+0.336B_3-0.106B_4+0.210C_1-0.321C_2+0.138C_3-0.106C_4-0.602D_1+0.306D_2-0.168D_3+0.248D_4 \quad \ldots\ldots\ldots\ldots\ldots(1)$$

$$\text{COF} = 4.12+0.782A1-1.312A2+0.231A3-0.385A4+0.410B1+0.285B2-0.038B3+0.842B4-2.119C1+0.425C2-0.218C3+0.841C4+0.058D1-0.170D2+0.381D3-0.167D4 \quad \ldots\ldots\ldots\ldots\ldots(2)$$

Increased wear rate by increasing the hydroxyapatite reinforcement particulates at more than 2 wt%. From regression equation (2) shows the coefficients related to hydroxyapatite wt% (A2, A4), load (B3, B4), sliding velocity (C1, C3) and sliding distance (D2, D4) are negative. The parameters in the regression equation present the negative indication is less in the value and positive indication is high value of wear rate and COF.

3.3 Surface Response:

Figure 6 presents the worn surface of AZ91D alloy and hydroxyapatite reinforced composites obtained by Scanning electron microscopy. The microstructure images present the worn of reinforcement particles in the wear track. Abrasive wear obtained owed to yield bulging point of steel disc body as it presses the hydroxyapatite reinforced composites. Moreover, the wear loss was high due to increased weight percentage of reinforcement particulates in the composite materials. The rigorously scratched regions shown in figure 6(b) also it indicate the abrasion and patches in worn surface. Material heavy flow and thin grooves occurred in sliding direction, the morphologies of wear surface specify the reality of adhesive and delaminating wear mechanism in the composite samples.



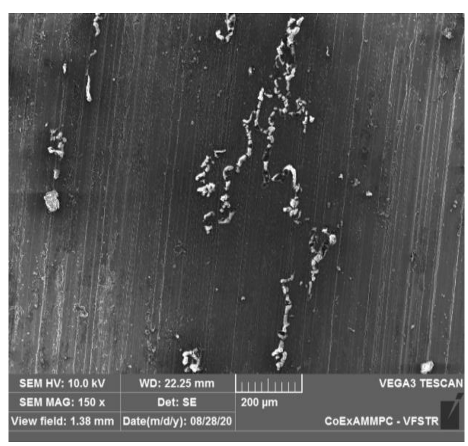


Fig 6(a) Worn surface of AZ91D mg alloy Fig 6(b) Worn surface of AZ91D/ hydroxyapatite composite.

Figure 7 presents the microstructure image of composite materials after conduction of wear test, which exhibits the worn out of reinforcement particle in the matrix material. Wear loss reduced in the composite samples by the addition of 2wt% of hydroxyapatite particles. hydroxyapatite particulates in the matrix material will decrease the grain boundary and matrix plastic deformation to increase the strength of material. Present wear study deformation resistance offered by hydroxyapatite, fractured into tiny pieces and formed wear debris particles. These debris prevent the sliding disk from stabbing of metal, which protects the magnesium matrix here by reduce the wear rate of composite material. Figure 8d shows the borders of worn out of reinforcement material due to adhesive wear mechanism. In optimization analysis, the structure of fine layer and smaller rubbles were observed. From this study the hydroxyapatite reinforced composite materials had excellent potential which resists to tribological wear behavior compared to AZ91D magnesium matrix material.

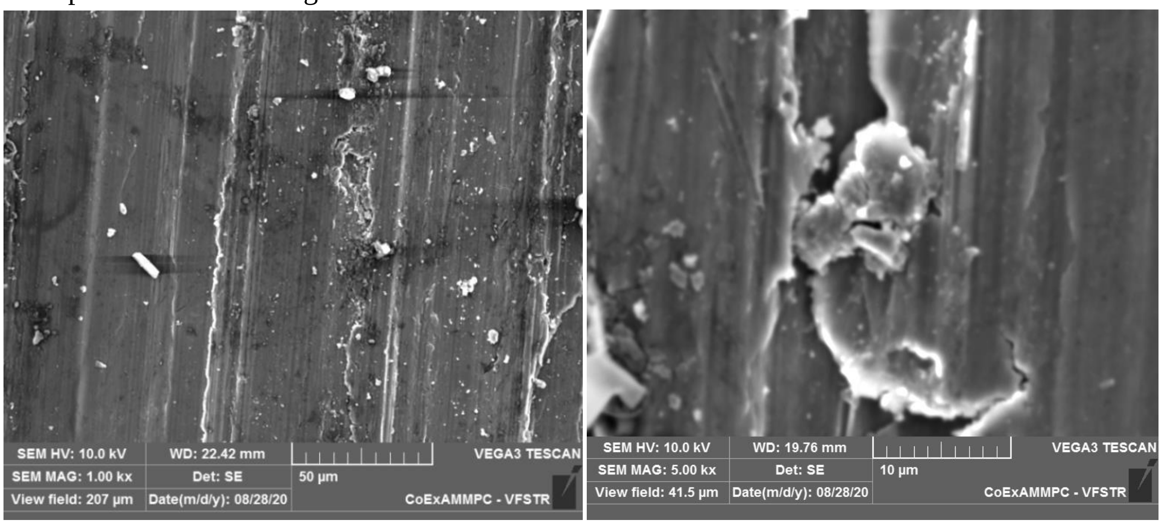


Figure 7 microstructure images of reinforcement particulates worn out from the matrix material.

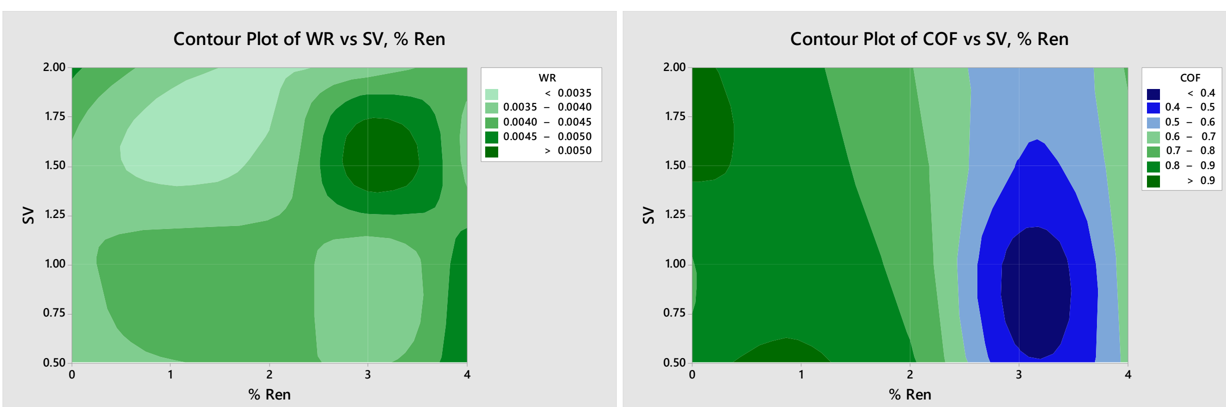


Figure 8 Contour plots for wear and COF

Figure 8 represents the contour plots obtained by using ANOVA analysis and it indicates the worn surfaces by influencing of composites by sliding velocity with increasing of percentage of reinforcement. Figure 9 shows the surface plots occurred by influencing of composite materials by applying loads and increasing of reinforcement.

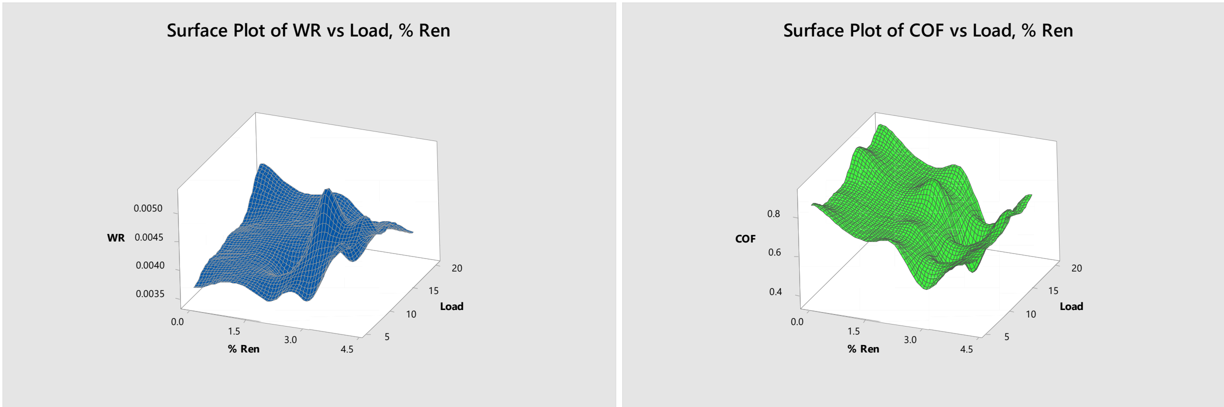


Figure 9 Surface plots Wear rate and COF

Conclusions:

AZ91D magnesium metal matrix composite reinforced with various weight percentages of Hydroxyapatite particulates were effectively fabricated by using stir casting technique. Taguchi technique was used to optimize and analyze the controlling factors influence the wear rate and coefficient of friction. The analysis of parameters is observed the following.

- hydroxyapatite reinforcement shows the supreme contribution to reduce the wear rate and coefficient of friction.
- The sliding distance is the most influencing factor for increasing the wear rate.
- Optimized graphical and analytical results of A2,B2,C1 and D2 corresponds to hydroxyapatite 2 wt%, load 10N, sliding velocity 0.5m/s and sliding distance of 750 m respectively.
- Conclusion of Microstructural analysis is that reinforcement debris forms a protective layer between sliding disk to work sample to reduce the Coefficient of friction.
- The mathematical regression equation developed by ANOVA analysis, coefficient of correlation was obtained 96% for wear rate and 98% for COF.
- At 2 wt% hydroxyapatite particulates decreases the wear rate and coefficient of friction of the composite material.

Therefore, it can be concluded that prepared AZ91D+2% hydroxyapatite stands as the best suitable material for the biomedical applications.

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