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Design of experiment analysis of elevated temperature wear of Mg-WC nano-composites

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ABSTRACT

Current study explores the effect of selected process parameters i.e. wt.% of reinforcement (A), elevated temperature (B) and load (C) on wear characteristics of Mg-WC nanocomposites using Taguchi robust design concept. Ultrasonic treated stir casting is employed to synthesize nanocomposites. Three levels for every factor are taken into consideration and accordingly L_{27} orthogonal array (OA) is used for minimization of wear rate. Main effect plot is generated to investigate the important parameters and optimality is also predicted from the main effect plot. Optimal condition for minimum wear rate is 2wt.% of WC, 100°C temperature and 20N load (A3B1C1). Interaction plots are generated to scrutinize the interaction outcome between selected parameters. ANOVA study is executed to evaluate significant parameters and their effective handout on output. Current investigation reveals, Wt.% of WC is the most significant factor while temperature and load are moderately significant. Among the interacting parameters, interaction between wt.% of WC & temperature (A×B) has moderate significance. Wt.% of WC (A) has 43.135% contribution while temperature (B), load (C) and interaction between wt.% of WC & temperature (A×B) have 26.623%, 19.037% and 5.639% contribution respectively. Residual plots for wear rate are discussed and confirmation test finally helps to validate present experimental model. S/N ratio is improved by 4.411 dB (48.60%) than the initial condition.

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1. Introduction

Need of lightweight materials for aviation and automotive industries are continuously increasing due to the concern of fuel consumption and hazardous emissions (Hirsch & Al-Samman, 2013). Particularly magnesium alloys are becoming suitable choice of aviation and automotive industries due to their light weight (two third of Al), good machinability (<50% than Al) and better manufacturability (<25-50% than Al) (Avedesian & Baker, 1999). But typical applications of magnesium alloys are restricted due to low modulus, inherent brittleness, lower strength at elevated temperature and poor high temperature tribological performance. Hence applications of Mg alloys are restricted in certain specific elevated temperature applications like engine parts (~ 200°C), bearing of aerospace, IC engines (~ 175°C) and cylinder liners (Pai et al., 2012). Hence researchers are focusing towards improvement of properties of magnesium alloys so that their application areas become wider. Presently, scientific community is considering several methods like use of rare earth (RE) element as alloying element, use of ceramic particles as reinforcement (Banerjee et al., 2019). Zafari et al. (2012) have examined wear characteristics of AZ91 magnesium alloy at room and high temperatures. Zafari et al. disclose that, increase of load and speed results in severe changes in wear. Zafari et al. (2014) also scrutinized the role of rare earth additions (lanthanum) in high temperature wear behavior. At temperature range 100-200°C, AZ91 alloy shows severe wear while RE containing alloy shows much lower wear rate. Labib et al. (2016) have used SiC particle as reinforcing phase in magnesium matrix and studied wear behavior at room and high temperature. Labib et al. discloses that use of ceramic based SiC particle possess significant less wear rate compared to pure magnesium at high temperatures (100-200°C). Accordingly in the current study tungsten carbide (WC) nano-particles are considered because of its high hardness (1400 HV), good shock absorption capability, high melting point, noteworthy oxidation resistance etc (Pal et al., 2018). Recently, Karuppusamy et al. (2019) have examined magnesium based nanocomposites having WC as reinforcement and studied wear behavior at cryogenic domain. Banerjee et al. (2019) have studied tribological behavior of Mg-WC nanocomposites at different temperatures. Praveenkumar et al. (2019) fabricated Mg-WC composites and evaluated mechanical as well as tribological properties at room temperature.

It is well-known that nano-particles are susceptible to agglomeration; accordingly, cautious selection of synthesis technique is solicited. Recent literatures show that metal matrix nanocomposites (MMNC) are efficiently and economically synthesized by ultrasonic treated stir-casting technique. Several ceramic particles i.e. Al_2O_3, CNT, SiC, ZnO are used to develop Mg-MMNC using ultrasonic treated stir-casting technique (Nguyen et al., 2015; Erman et al., 2012; Selvam et al., 2014; Aung et al., 2010). In this process electrical energy having high frequency is employed to form mechanical vibrations in melt and small microscopic bubbles are generated. Further these bubbles implode and produce shock waves as well as intense heating. These shock waves and intense heating helps to break clusters of nanoparticles (Banerjee et al., 2019).

Recently, Nguyen et al. (2015) synthesized Mg-Al_2O_3 and found excellent wear behavior at high load and speeds. Habibnejad-Korayem et al. (2010) have reported superior wear behavior of Mg-Al_2O_3. Kaviti et al. (2018) fabricated Mg-BN nanocomposites and examined wear behaviour at different load and speed. Labib et al. (2016) synthesized Mg-SiC composites and investigated wear behavior at temperature range of 25-250°C at different loads. Significantly lower wear rate is reported for composites at elevated temperatures. Gopal et al. (2017) have developed Mg-CRT-BN composites and reported the contribution of size & wt. % on tribological characteristics. Banerjee and Sutradhar (2018) have statistically optimized tribological process parameters of Mg-Gr-WC using RSM. Girish et al. (2016) have used Taguchi methodology to investigate wear behavior Mg-SiC-Gr composites. Available literature discloses that studies on optimization of elevated temperature wear behaviour of Mg-WC nanocomposites are not available. Present study uses Taguchi method based L_{27} OA to find optimal parameter condition of minimized wear rate. For this purpose wt.% of reinforcement, applied load and elevated temperatures are taken as input factors while three levels of every factor are considered. Significant parameters and interactions are found by ANOVA analysis. Finally adequacy of the model is successfully verified by confirmation test.

2. Experimental Scheme

In current investigation, AZ31 alloy is observed as base material because of excellent castability, good formability, low density and high elastic modulus. Elemental details of AZ31 is presented in Table 1. Tungsten carbide nanoparticles (HW Nano, K510) with average size 80 nm are chosen as reinforcement. Details of WC are given in Table 2. For this study 1, 1.5 and 2wt.% of reinforcements are reinforced with AZ31 magnesium alloy.

Table 1. Elemental details of AZ31

Element	Si	Mn	Al	Fe	Zn	Mg
Wt. %	0.10	0.28	3.20	0.22	1.20	Balance

An expressly designed stir casting furnace (SWAMEQUIP, Chennai) is used to develop magnesium metal matrix nanocomposites (MMNC). Illustrated representation of the main furnace and other particulars are furnished in Figure 1. This furnace has several attachments like particle preheating attachment, mechanical stirrer, inert gas supply, bottom pouring system, die pre-heating set up, ultrasonic vibrator. Primarily, required extent of AZ31 alloy is inserted into the main crucible and heated at 750°C. Meanwhile, desired proportion of tungsten carbide nanoparticles are weighted and brought into the particle heating chamber and die heating rod is put down inside the split type die to foreheat it. After a certain time a drop in melt temperature is noticed which yields that magnesium alloy inside the crucible is fully melted. Then the stirrer is dip into the melt and stirring is started at high speed. As a result vortex is created. Preheated nanoparticles are then injected continuously into the melt. Mechanical stirring is stopped after completion of particle injection. Very high frequency ultrasonic cavitation (20 KHz) is produced inside the melt by use of ultrasonic horn. Ultrasonic vibration breaks the particle cluster and helps to achieve uniform distribution of reinforced particles. Finally the die is kept into vacuum arrangement which is put under bottom pouring hole and composite slurry is drained in vacuum condition (10^{-2} mbar). Total experiment is conducted under argon gas environment to protect the system from oxidation. After a certain time the melt is solidified and solidified bar is pull out from the die. Then parting, milling, shaping, turning and grinding is done to develop desired sample (6 mm dia., 30 mm length). Parameteric arrangements of fabrication are tabulated in Table 3.

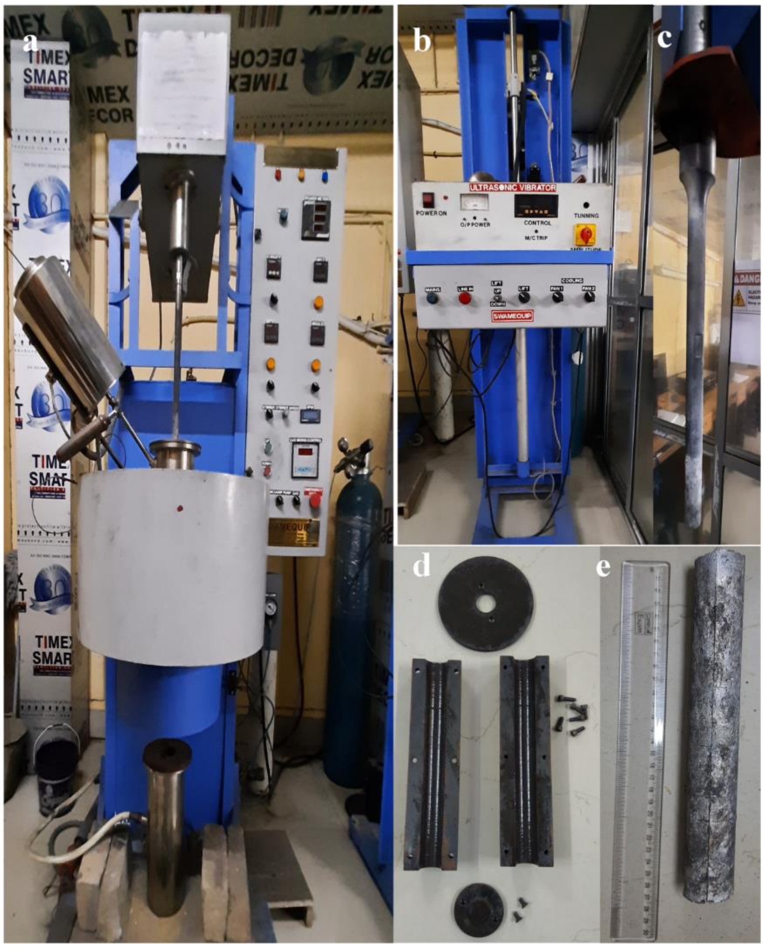

Figure 1. Pictorial view of the furnace (a) Main furnace, (b) Ultrasonic vibrator, (c) Ultrasonic horn, (d) split-type die, (e) As-cast bar

Table 2. Details of WC powder

Parameter	Details
Particle Size	80 nm
Purity of WC	99.9%
Form	Hexagonal
Specific surface area (SSA)	3-8 m^2/g
Color	Black
Composition of WC	W-93.8%
	C-6.1%

Table 3. Parameter context during experimentation

Parameters	Value
Temperature of Furnace	750°C
Material of Stirrer	Stainless steel
Mechanical stirring time	8-10 minutes
Blade Angle	45°
Pre-heating temperature of reinforcement	300°C
Ultrasonic frequency	20 KHz
Duration of Ultrasonic vibration	3-5 minutes
Die Dimension	Φ50 mm × 300 mm (Split type)

In current study hypothesis of Taguchi robust design based parameter outline is employed so that optimality for minimized wear rate of fabricated materials can be engineered. Parametric design helps to decide requisite orthogonal array. Concept of orthogonal array decides preferable quantity of experiments relying on quantity of input factors and their levels. Accordingly, L_{27} OA is chosen to cut down total amount of experiments. Idea of signal to noise ratio (S/N ratio) is employed to distinguish role of input factors on output factor. Aim of this investigation is to minimize wear rate hence lower the better principle of S/N ratio is employed. Significant process parameters and interactions of this study are found by performing analysis of variance (ANOVA). In this study, three input parameters having three levels each are taken. Considered factors and levels are tabulated in Table 4.

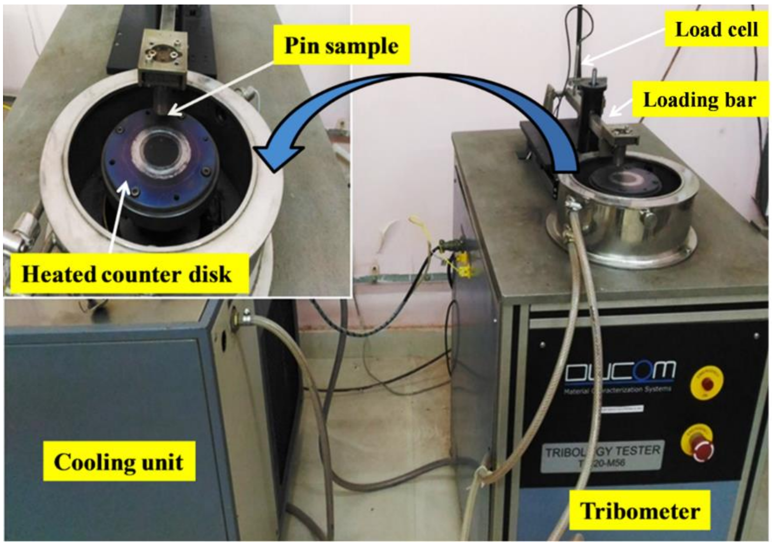

Figure 2. Pictorial view of tribotester

Table 4. Input factors and levels

Factors	Units	Level 1	Level 2	Level 3
Wt.%	%	1	1.5	2
Temperature	°C	100	150	200
Load	N	20	30	40

Experimental results of elevated temperature wear behavior of Mg-WC nanocomposites are achieved by using a pin-on-disc tribotester (TR-20-M56, DUCOM). Pictorial view of trobotester is shown in Figure 2. Hardened EN 31 disc (Φ = 115 mm, t = 8 mm) having high hardness (58-62 HRC) is considered as counter-face system to assure happening of wear only in pin surface. Pin samples are vertically placed on the disc using stationary fixture. Counter disc has rotary motion and the pin surface rub against the counter disc. The counter face disc is heated by 15 KVA induction heating system at the time of experimentation. Wear tests are conducted according to ASTM G99-05. Experiments are carried out for 10 min at different temperature and load while sliding speed is fixed at 0.4 m/s. Pin samples are weighted before and after test to find the mass loss using digital weighing balance (Afcoset, 182A) to calculate wear rate.

3. Results & Discussion

Elevated temperature wear behavior of produced nanocomposites is statistically analyzed by performing S/N ratio analysis, ANOVA analysis and validation test. Genichi Taguchi, founder of Taguchi methodology, suggested choosing S/N ratio analysis in lieu of simple average method while studying experimental results to predict optimal condition. Main advantage of S/N ratio analysis is that it is capable of studying variability of experimental results. Accordingly S/N ratio analysis of experimental data is carried out in this study. As wear rate is needed to be minimized lower the better criterion of S/N ratio analysis is considered in this study. S/N ratios of experimental data are calculated by following formula (n = no. of observations, y = examined data):

$$S / N = -10log(1 / n\sum y^2) \tag{1}$$

Design of experiment and interrelated S/N ratios are noted in Table 5. Mean value of the S/N ratio of all level of each factor is presented in response table (Table 6). Total mean value of S/N ratio is found to be -8.693 dB. Table 6 also presents delta value of every process parameter. Depending on the delta value, process parameters are ranked which helps to decide the influence of that process parameter on the response variable. Table 6 shows that wt.% of WC (A) is ranked as 1, temperature (B) is ranked as 2 and applied load (C) is ranked as 3. Hence, A is the most influencing parameter followed by B and C.

Main effect curve endorses to analyze the influence of input factors on output variable. It also stimulates to find the optimal condition for output parameter. Figure 3 shows the main effect plot of this study. It is well known that nature of the plots explain the effect of the parameter and their levels. Parameter showing highest inclination proves to be of greater importance while parameter having gentle slope considered to be of less importance. Figure 3 shows that plot of parameter A has highest inclination followed by slope of parameter B and parameter C. Hence parameter A has highest importance while B and C have moderate importance. It is evident from Figure 3 that at level 3, parameter A has maximal S/N ratio while parameter B and parameter C both has maximal S/N ratio value at level 1. Thus, optimality for minimum wear rate is A3B1C1, i.e. 2wt.% of WC, 100°C temperature and 20N load. Figure 4 presents the both way interaction plot of this study. In interaction plot, non-parallel lines yields occurence of interaction and intersecting lines yields occurence of strong interaction. In Figure 4, non-parallel lines are present for all three cases which disclose that interaction is present for all three cases of this study.

Analysis of variance is a statistical model to figure out the significant process parameters and interactions which majorly influence total variance of experimental data. Table 7 shows the results of ANOVA analysis of this study. ANOVA table consists of sum of square (SS), mean square (MS), F-ratio, P factor and percentage contribution of each factor. F-ratio values justify the significance of the parameter or interaction. Parameter having higher F-ratio value has higher possibility to be significant. In this study, A has highest F-ratio value followed by B and C which suggest that wt.% of WC (A) is utmost significant fator while temperature (B) and load (C) are moderately significant. Among the interacting parameters, (A×B) has moderate significance. P-values of A, B, C and (A×B) are below 0.005, which yields significance of these parameters. Table 7 also shows that A has 43.135% contribution while B, C and (A×B) have 26.623%, 19.037% and 5.639% contribution respectively.

Coefficient of determination value for the present study is 98.97%. Adequacy of the present model is verified by the normal probability plot (NPP) which is presented in Figure 5. NPP correlate the forecasted values of the system with experimental data. Figure 5 yields that experimental dossier approximately follows a straight line that verifies the adequacy of the system. Similar result was seen in the study of Karuppusamy et al. (2019).

Finally confirmation test is implemented to compare the initial condition with optimal condition so that improvement in final optimal result can be understood. Optimal level is predicted using following equation:

$$\overline{\eta} = \eta_m + \sum_{i=1}^{0} (\overline{\eta_i} - \eta_m) \tag{2}$$

where, η_m = total mean data, η_i = mean data at optimal level, o = number of selected input factors which significantly affect output response.

Table 5. Design of experiment and corresponding S/N ratios

Run order	Wt.%	Temperature	Load	SNRA
1	1	100	20	-7.47295
2	1	100	30	-7.82223
3	1	100	40	-8.41891
4	1	150	20	-9.02652
5	1	150	30	-9.41703
6	1	150	40	-12.8966
7	1	200	20	-11.0485
8	1	200	30	-11.4598
9	1	200	40	-16.277
10	1.5	100	20	-7.12052
11	1.5	100	30	-7.276
12	1.5	100	40	-7.64394
13	1.5	150	20	-8.47147
14	1.5	150	30	-9.07554
15	1.5	150	40	-11.9605
16	1.5	200	20	-10.5294
17	1.5	200	30	-10.5113
18	1.5	200	40	-14.9187
19	2	100	20	-4.665
20	2	100	30	-5.31993
21	2	100	40	-6.8524
22	2	150	20	-3.23335
23	2	150	30	-4.91519
24	2	150	40	-6.61639
25	2	200	20	-6.08119
26	2	200	30	-6.54309
27	2	200	40	-9.13339

Table 6. Response table

Level	Wt%	Temperature	Load
1	-10.427	-6.955	-7.517
2	-9.723	-8.401	-8.038
3	-5.929	-10.722	-10.524
Delta	4.498	3.768	3.008
Rank	1	2	3

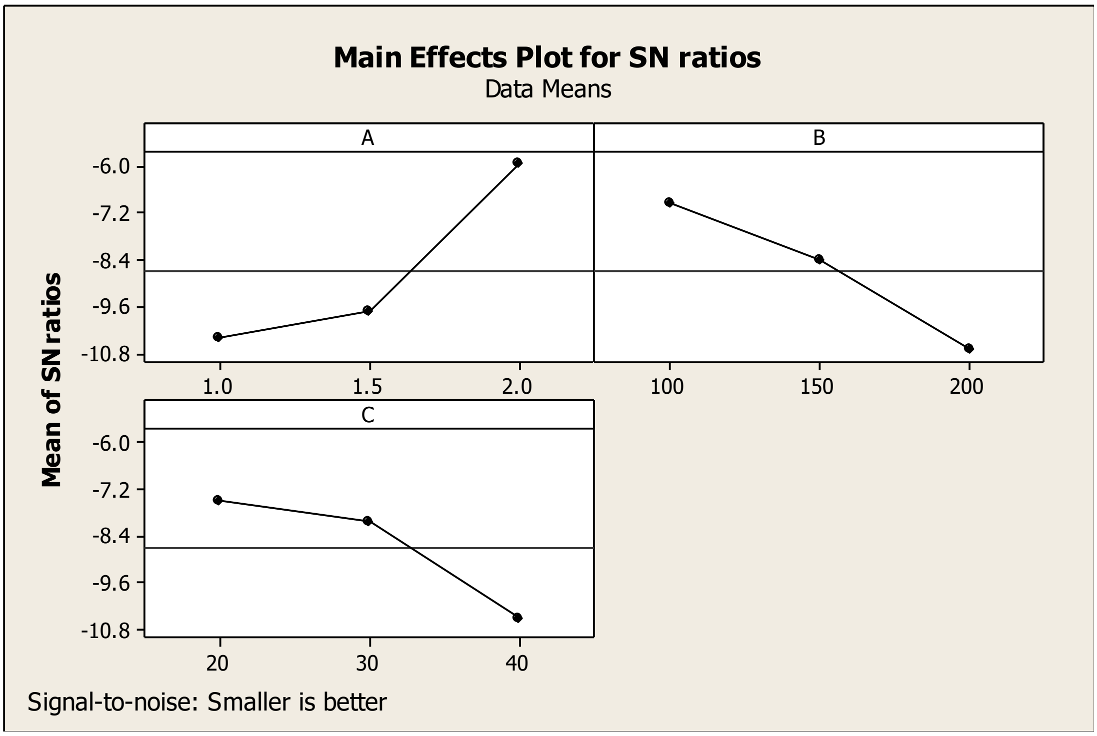

Figure 3. Main effect plot

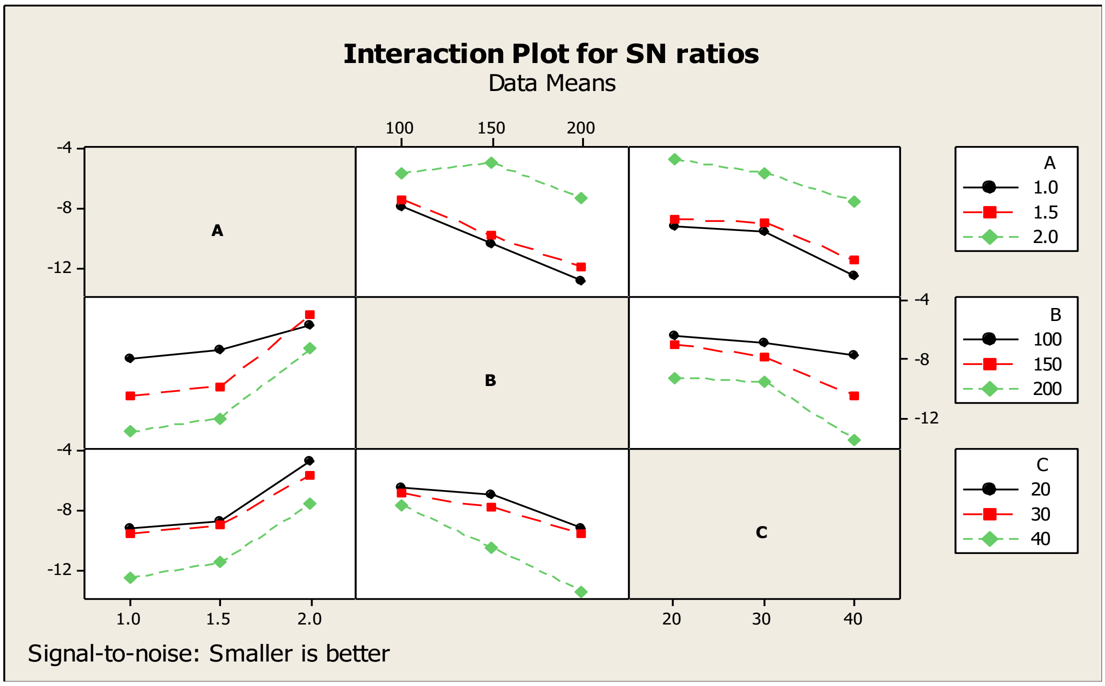

Figure 4. Interaction plot

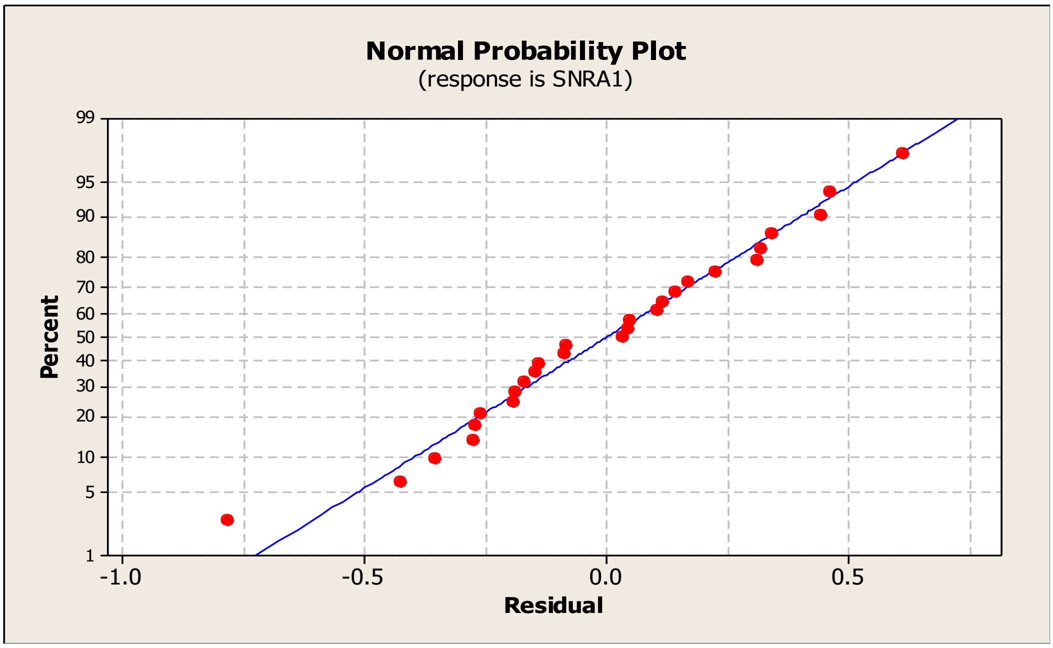

Figure 5. Normal probability plot

Table 7. ANOVA table

Source	DF	Adj SS	Adj MS	F-Value	P-Value	%Contribution
Wt.%	2	105.360	52.680	167.23	0.000	43.135
Temp.	2	65.030	32.515	103.22	0.000	26.623
Load	2	46.500	23.250	73.81	0.000	19.037
Wt.%*Temp.	4	13.774	3.444	10.93	0.003	5.639
Wt.%*Load	4	0.970	0.243	0.77	0.574	0.397
Temp.*Load	4	10.102	2.526	8.02	0.007	4.136
Error	8	2.520	0.315			
Total	26	244.258				100.000

S	R-sq	R-sq(adj.)
0.561256	98.97%	96.65%

Table 8. Confirmation table

	Initial Condition	Optimal condition	
		Prediction	Experimentation
Level	A2B2C2	A3B1C1	A3B1C1
Wear	2.843		1.711
S/N ratio (dB)	-9.076	-3.015	-4.665

Table 8 reveals the details of the confirmation test of this study. It is found that S/N ratio is improved by 4.411 dB (48.60%) than the initial condition. That much improvement is very significant.

4. Conclusion

In this investigation, Mg-WC nanocomposites are synthesized by ultrasonic treatment assisted stir-casting technique. Taguchi robust design is successfully employed to optimize process parameters (wt.% of reinforcement, temperature and load) to minimize wear rate of nanocomposites. Significant process parameters and interactions of this study are found by performing analysis of variance (ANOVA). Adequacy of the present model is verified by the normal probability plot (NPP). Confirmation test is implemented to compare the initial condition with optimal condition so that improvement in final optimal result can be understood. From this statistical investigation, subsequent interpretetion are made:

- Optimality of minimum wear rate is 2wt.% of WC, 100°C temperature and 20N load (A3B1C1).
- Wt.% of WC is utmost notable factor while temperature and load are moderately significant. Among the interacting parameters, interaction between wt.% of WC & temperature (A×B) has moderate significance. Wt.% WC (A) has 43.135% contribution while temperature (B), load (C) and interaction between wt.% of WC & temperature (A×B) have 26.623%, 19.037% and 5.639% contribution respectively.
- Coefficient of determination value for the present study is 98.97%. Adequacy of the present model is verified by the normal probability plot (NPP).
- Confirmation test discloses that S/N ratio at optimality is improved by 4.411 dB (48.60%) than the initial condition.

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