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Investigation and Optimization of Hardness and Tribological Behaviour of Ricehusk Ash Reinforced Aluminium Matrix Composite using Powder Metallurgy Route

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ABSTRACT
A composite material of pure aluminium (Al) reinforced with rice husk ash (RHA) was developed using the powder metallurgy route. The mixing parameters (speed and time) were optimized using optimum mixture design in Design Expert 13 software. The effects of varying aluminium and RHA compositions, as well as mixing time and stirring speed, were investigated. The mechanical and tribological properties, specifically hardness, wear rate and coefficient of friction (COF) were evaluated. Before the RHA reinforcement was added, the pure aluminium exhibited a hardness of 61.36666667 HV, a wear rate of 0.000013889 mm/Nm, and a coefficient of friction of 0.3215. The optimization process resulted in an optimal composition of 12.552% RHA and 87.448% aluminium, with a stirring speed of 169.812rpm and a mixing time of 1 hour. Under these conditions, the composite achieved a hardness of 108.326 HV, a zero wear rate, and a coefficient of friction of 0.307. The optimization led to a percentage improvement of 76% in hardness, a 100% reduction in wear rate, and a 4.1% reduction in coefficient of friction. The reduced coefficient of friction remains within the optimal range for effective braking application.
Keywords: Rice Husk, Powder Metallurgy, Tribology, Optimum Mixture Design

1 INTRODUCTION

Demand for engineering materials with low density, high hardness, improved tribological properties and low cost is important for application in the aerospace and automobile industries. The use of composite materials satisfies this quest. Composites are materials made from two or more constituent materials of significant properties, both physical and chemical that, when combined, produce a new material with characteristics better than that of each individual constituent (Ahamed et al., 2016). It is an important material that offers benefits such as low density and high mechanical strength which serve as an advantage over other existing material like metals and plastics (Tri-Dung, 2019). Metal matrix composites (MMCs) are composites where metals or metallic alloys are used as matrix and other materials are used as reinforcements (Magibalan et al., 2017); it finds applications in various fields such as automotive, powertrain, aerospace, consumer electronics, packaging, and sports (Macke et al., 2012).

The development of low-cost metal matrix composites (MMCs) has been one of the major innovations in the field of materials in the past few decades (Sharma et al., 2020). Over the years, various materials such as aluminium oxide (Al_2O_3), silicon carbide (SiC), barium carbide (B4C), titanium carbide (TiC), have been used to reinforce aluminium to enhance its properties (Wankhade et al., 2021). In recent times, there has been significant interest in producing AMMCs reinforced with rice husk ash (RHA) (Ziyauddin et al., 2022).

Rice husk with high oxide content serves as cost-effective and readily available alternative reinforcing materials in metal matrix composites (MMCs), offering physical and mechanical properties comparable to the conventional particulates (Joseph and Babaremu 2019). It is a significant by-product of rice production, accounting for about 20% of the grains weight and contains about 50 % cellulose, 25–30 % lignin, and 15–20 % of silica (Ramezanianpour,

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2014). It is normally disposed off by burning which contaminates the ecosystem; the burning leads to the elimination of many volatile substances leaving behind ash, which is referred to as rice husk ash (RHA) (Ziyauddin et al., 2022) containing 85 to 90 % amorphous silica (Hossain et al., 2018). Powder metallurgy is one of the most beneficial processing routes to attain uniform distribution (Shaikh et al., 2019); it is a popular metal forming technology used to produce precision components. Different powder and component forming routes can be used to create an end product with specific properties for a particular application or industry (Chang et al., 2013). The research work involved statistical mixture design, characterization, mathematical modelling, analysis and optimization of rice husk ash reinforced aluminium matrix composite. The composite constituents consist of rice husk ash as the reinforcement and pure aluminium as the matrix.

2 EXPERIMENTS

2.1 Materials

Materials and reagent used in this research work include pure aluminium powder (99.99%) purchased from China as the metal matrix. Other materials are rice husk as the reinforcement sourced from Kano state, Nigeria, ethanol as the process control agent and zinc stearate lubricant powder as a lubricant during compaction and ease the ejection of the green compact.

2.2 Preparation

The collected rice husk was washed thoroughly with clean water in order to remove the impurities like dust. The washed rice husk was then dried under the sun for 5 hours. Afterwards, the dried rice husk was burnt in a furnace at 250°C for 1h to remove moisture, oxides and volatile constituents. The burnt rice husk was heated to 700◦C for 12 hours to remove carbonaceous materials present in the rice husk. The obtained RHA particles were ground manually using stone agate and pestle. Then the RHA was sized using Sieve Shaker with average size of 75µm.

2.3 Fabrication of the Composite

The factors considered in this study (Table 1) are mixing time, stirring speed and composition of the mixture. Mixing time varies between 1hour, 2hours, and 3hours, while stirring speed ranges from 100rpm to 300rpm in increments of 100rpm. The composition consists of aluminium and rice husk ash in varying proportions to evaluate their effects on hardness, wear and coefficient of friction. Optimal mixture design was used for this investigation with four factors at different levels. The total number of experimental runs make up to 48 as shown in Table 2. The aluminium metal matrix composite formulations are given below:

$0 \leq RHA \leq 40$
$60 \leq Al \leq 100$
$100 \leq SS \leq 300$
$1 \leq time \leq 3$

Table 1: Factors and levels for the optimal mixture experimental design plan.

Factors	Symbols	Levels
Rice husk ash (mixture) %	A	0, 5, 10, 20, 30, 40
Aluminum (mixture) %	B	100, 95, 90, 80, 70, 60
Stirring speed(rpm)	C	100, 200, 300
Mixing time (hrs)	D	1, 2, 3

2.3.1 Powder Mixing

15 gm of RHA (Rice Husk Ash) and Al (Aluminium) powders was weighed accurately using a high-precision digital weighing balance. To achieve a uniform and homogeneous mixture of the RHA and Al powders, a laboratory planetary ball mill and the milling process lasted for 1, 2 or 3 hour(s). The ball-to-powder ratio (BPR) was set at 5:1, meaning five parts milling balls to one part powder by weight, with a rotational speed of 100, 200 or 300 rpm. To avoid cold welding (where metal particles stick together during milling), 0.5 wt% of ethanol (as a lubricant) was added to the mixture.

2.3.2 Powder Compaction

1gram of the mixture of Aluminium powder (Al) and rice husk ash (RHA) was compacted using a hydraulic powder compacting press at 5 MPa. Zinc stearate powder was used as a lubricant during compaction to facilitate the process and ease the ejection of the green compact.

Table 2: The optimal mixture design.

NO.	RHA (%)	Aluminium (%)	Stirring speed (rpm)	Mixing time (hrs)	Hardness (HV)	Wear rate (mm^3/m)	Coefficient of friction (μ)
1	0	100	300	1	61.36	1.39E-05	0.3215
2	0	100	300	1	61.37	1.39E-05	0.3215
3	5	95	200	3	103.9	3.16E-06	0.251
4	5	95	300	1	103.2	2.51E-06	0.231
5	5	95	100	1	100.8	2.92E-06	0.274
6	5	95	300	2	97.9	3.89E-06	0.2475
7	5	95	200	2	93.9	4.70E-06	0.2435
8	5	95	300	3	63.97	3.40E-06	0.145
9	5	95	100	3	85.67	2.19E-06	0.2405
10	5	95	100	2	110	2.76E-06	0.2595
11	10	90	300	3	122.67	7.06E-06	0.278
12	10	90	100	2	105	8.13E-05	0.307
13	10	90	300	2	106	1.14E-05	0.2625
14	10	90	100	3	96.2	1.21E-05	0.255
15	10	90	200	2	63	3.15E-06	0.2345
16	10	90	300	1	112.33	6.46E-06	0.2535
17	10	90	200	1	99.73	1.69E-05	0.254
18	10	90	100	1	114.67	4.25E-06	0.2345
19	10	90	200	3	87.5	5.50E-05	0.313
20	20	80	300	2	81.07	0.0007649	0.3205
21	20	80	200	1	117.33	0.0001381	0.3405
22	20	80	300	1	55.47	0.0008567	0.429
23	20	80	200	2	132.33	0.0007049	0.408
24	20	80	100	2	70.2	0.0009415	0.277
25	20	80	300	3	90.27	0.0004871	0.3635
26	20	80	200	3	87.07	6.29E-05	0.262
27	20	80	100	1	132.67	0.0006623	0.429
28	20	80	100	3	109.3	0.0007644	0.4605
29	30	70	200	1	88.17	0.0009248	0.2705
30	30	70	200	3	52.17	0.0008334	0.3125
31	30	70	300	2	56.9	0.0010799	0.264
32	30	70	100	3	79.97	0.0001451	0.297
33	30	70	200	2	77.57	0.0002081	0.272
34	30	70	100	1	87.07	0.0005205	0.269
35	30	70	100	2	60.57	8.05E-06	0.252
36	30	70	300	1	86.6	0.0008064	0.3635
37	30	70	300	3	44.23	8.78E-05	0.2695
38	40	60	200	2	53.13	1.09E-06	0.042
39	40	60	300	2	53.67	0.0007622	0.377
40	40	60	200	1	63.23	0.0001977	0.281
41	40	60	200	1	63.23	0.0001977	0.281
42	40	60	200	3	69.9	0.0009303	0.3085
43	40	60	100	2	70.6	1.09E-06	0.2825
44	40	60	100	3	58.43	0.0002751	0.3035
45	40	60	100	3	58.43	0.0002751	0.3035
46	40	60	300	1	47.9	1.09E-06	0.031
47	40	60	300	2	47.9	1.09E-06	0.031
48	40	60	100	3	58.43	0.0002751	0.3035

2.3.3. Sintering Treatment

The sintering process was carried out in an electric muffle furnace and proceeded in three stages, following a specific sintering profile. The first stage is preheating, where the samples were heated to a relatively low temperature of 300^{o}C for 1hour to burn off additives such as ethanol and to strengthen the initial bonds between particles. The second stage is the sintering phase, during which the temperature was increased and maintained between 594^{o}C for 2hours. In the third and final stages, the samples were cooled to room temperature under a controlled atmosphere to prevent oxidation.

3. CHARACTERIZATION OF COMPOSITE SAMPLE

3.1 Microstructural Examination

Microstructural examination was carried out on the composite sample using scanning electron microscope to reveal surface morphology and spatial distribution of RHA (reinforcement particles) in the Aluminium matrix.

3.2. Hardness

The hardness tests were performed using a Vickers micro hardness testing machine. According to the ASTM E18-17e1 standard, a diamond indenter was used with an applied load of 0.3kgf, and a dwell time of 8 seconds.
To minimize potential segregation effects, three measurements was taken for each sample at different locations. The average hardness value was then determined from these measurements.

3.3. Wear and Coefficient of Friction

The wear test was performed on the compacted samples per ASTM 99-95a standard test method for wear testing of materials. Performing the wear test, ball-on-disk tribometer machine of model version R0.01 was used. The load and sliding distance of 8N and 30m respectively were applied and a test duration of 30 minutes for all the samples. The coefficient of friction of the samples was automatically generated by the ball-on-disk tribometer during the wear test.

4 RESULTS AND DISCUSSIONS

4.1 Statistical Analysis, Validation and Modelling of Experimental Results

The summary statistics of the models from Design Expert Package are shown in Tables 3, 4 and 5. For hardness (Table 3), the standard deviation of 16.87 represents the variation in the residuals. The R^2 value of 0.6272 suggests that 62.72% of the variation in hardness is explained by the model. The adjusted R^2 and predicted R^2 is 0.5133 and 0.4114 respectively. The adequate precision of 7.2819, measures the signal-to-noise ratio, it is above the recommended threshold of 4, indicating that the model provides a reasonable signal and can be used for optimization. The adequate precision shows the model has a strong signal for analysis. For the wear rate (Table 4), a standard deviation of 0.0003, the R^2 value of 0.6806 implies that 68.06% of the variation in wear rate is explained by the model, which appears moderately strong. However, the adjusted R^2 drops to 0.3745, The most concerning issue is the predicted R^2 of -0.7425, which being negative suggests that the model performs worse than simply using the mean value to predict wear rate, indicating severe over-fitting. Despite these weaknesses, the adequate precision of 5.8422 is above the recommended threshold of 4, meaning the model has a reasonable signal-to-noise ratio and still captures some meaningful relationships in the data (Nasrollah et al., 2020). For coefficient of friction (Table 5), a standard deviation of 0.0780. The R^2 value of 0.5780 suggests that 57.80% of the variation in the response variable is explained by the model. However, the adjusted R^2 drops significantly to 0.1735. More concerning is the predicted R^2 of -1.5876, which is negative. Negative predicted value may be indicative of over-fitting in the data, which may be as a result of lesser number of data runs. However, the adequate precision of 5.6986 is above the recommended threshold of 4, showing that the model has a reasonable signal-to-noise ratio and still captures some useful information from the data (Nasrollah et al., 2020). Moreover, validation of the hardness (cubic), wear rate (quadratic) and coefficient of friction (quadratic) models could also be observed from Figs. 1, 2 and 3 where the slope of the model (predicted response) against experimental points passing through (or closely) all the points.

Table 3: Model summary statistics for hardness.

Source	Standard deviation	R^2	Adjusted R^2	Predicted R^2	Adequate Precision
Cubic	16.87	0.6272	0.5133	0.4114	7.2819

Table 4: Model summary statistics for wear rate.

Source	Standard deviation	R^2	Adjusted R^2	Predicted R^2	Adequate Precision
Quadratic	0.0003	0.6806	0.3745	-0.7425	5.8422

Table 5: Model summary statistics for coefficient of friction.

Source	Standard deviation	R^2	Adjusted R^2	Predicted R^2	Adequate Precision
Quadratic	0.0780	0.5780	0.1735	-1.5876	5.6986

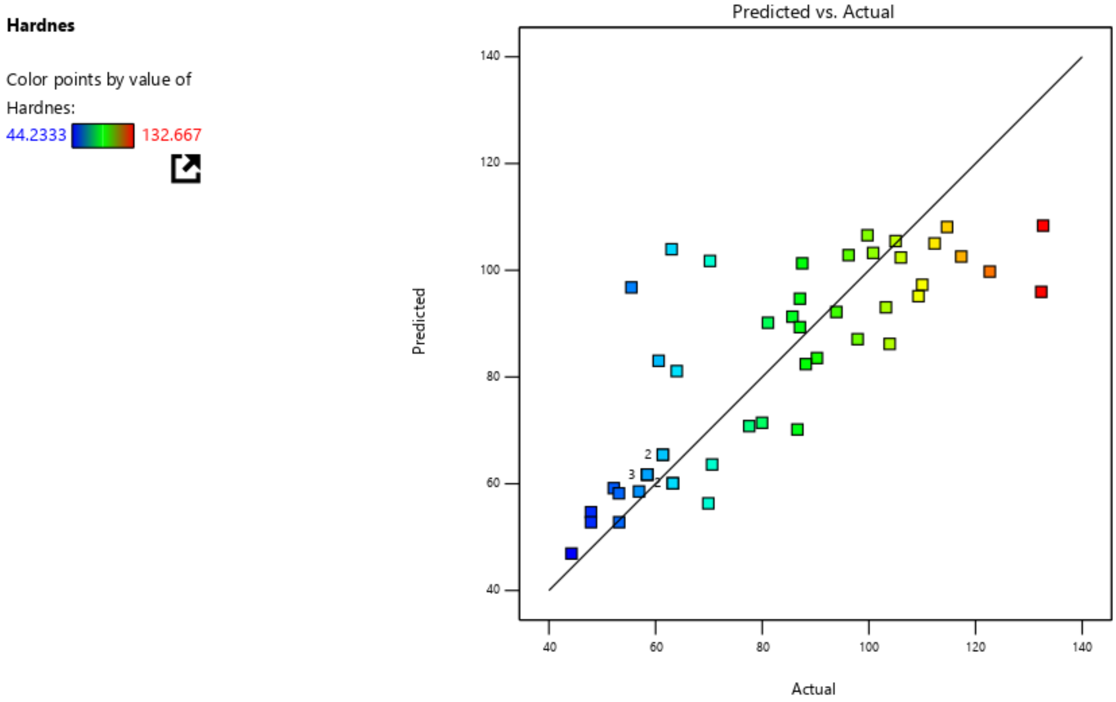

Figure 1: Experimental vs. predicted results of the coefficient of friction (quadratic model).

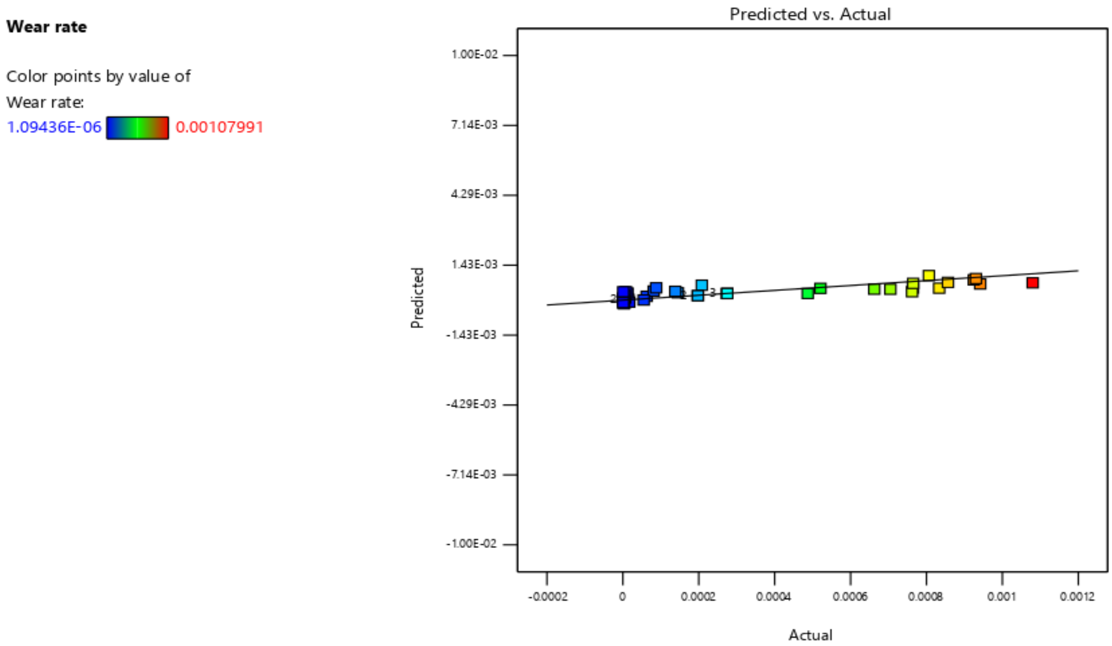

Figure 2: Experimental vs. predicted results of the coefficient of friction (cubic model).

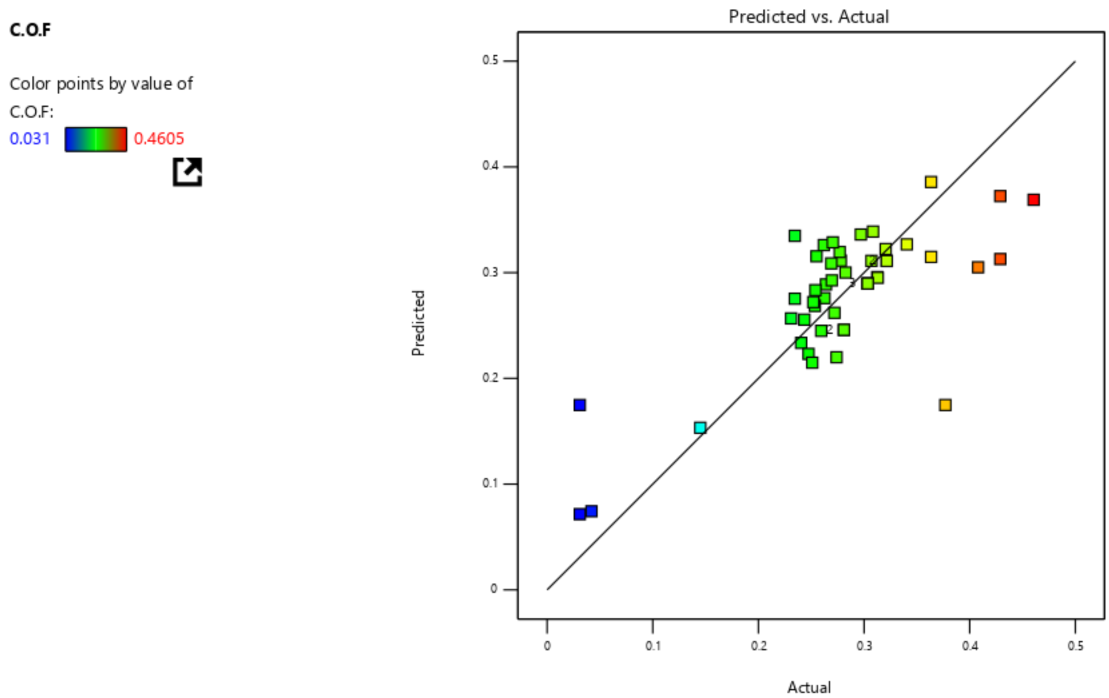

Figure 3: Experimental vs. predicted results of the coefficient of friction (quadratic model).

4.2 Characterization Effects of Two Factor Constituents

The effects of two constituents of the composite were analysed. The hardness effect of an increase in the percentage of rice husk ash and a decrease in the percentage of aluminium (Fig. 4) showed an increase in hardness followed by a decrease. The wear rate effect of an increase in the percentage of rice husk ash and a decrease in the percentage of aluminium (Fig. 5) showed an increase in wear rate followed by a decrease. The coefficient of friction effect of an increase in the percentage of rice husk ash and a decrease in the percentage of aluminium (Fig. 6) showed a non-linear trend.

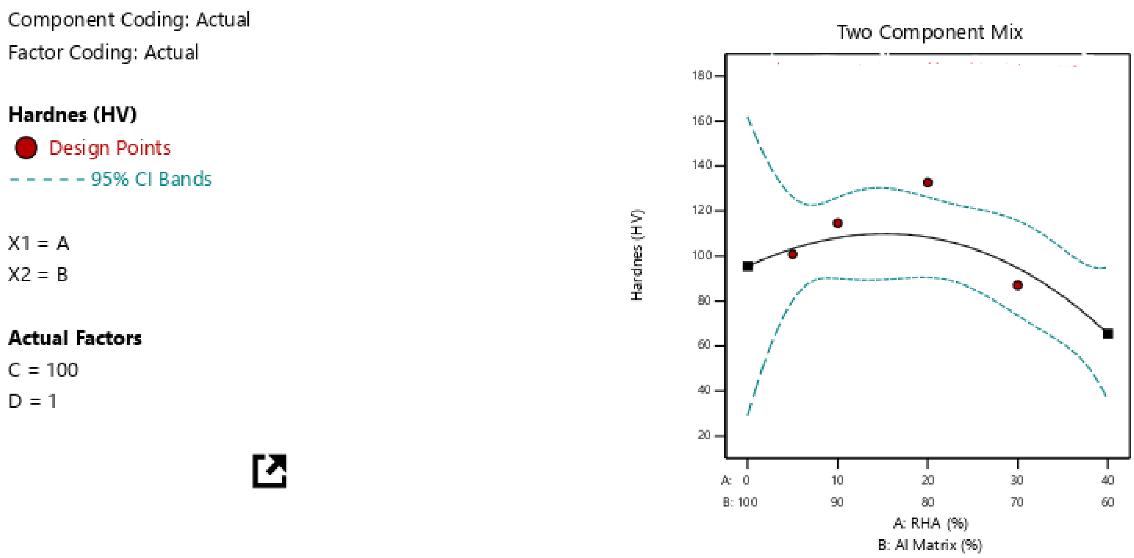

Figure 4: Hardness value effects of aluminium and Rice Husk in the composite.

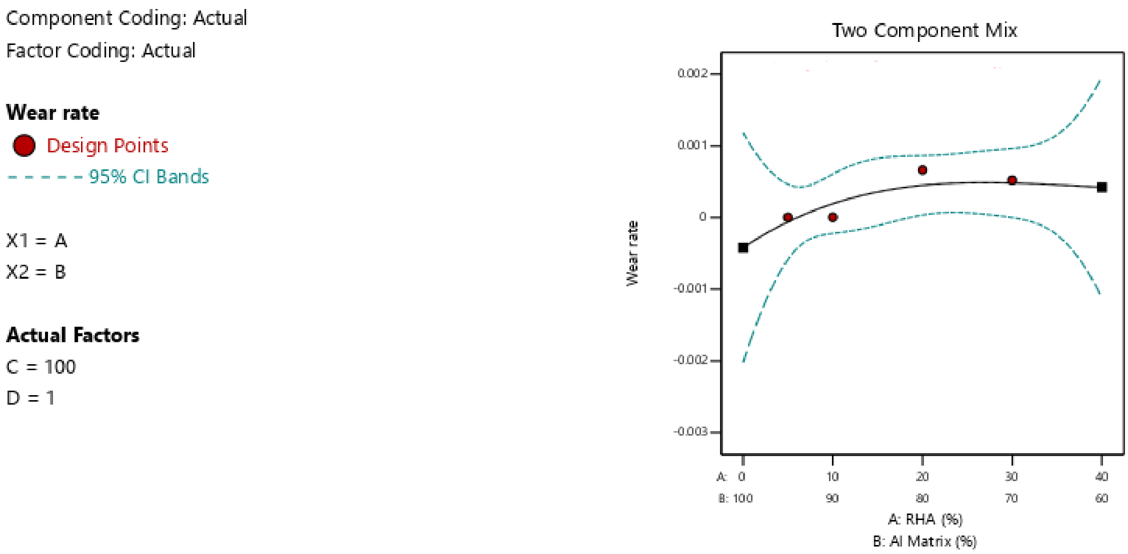

Figure 5: Wear rate value effects of aluminium and rice husk in the composite.

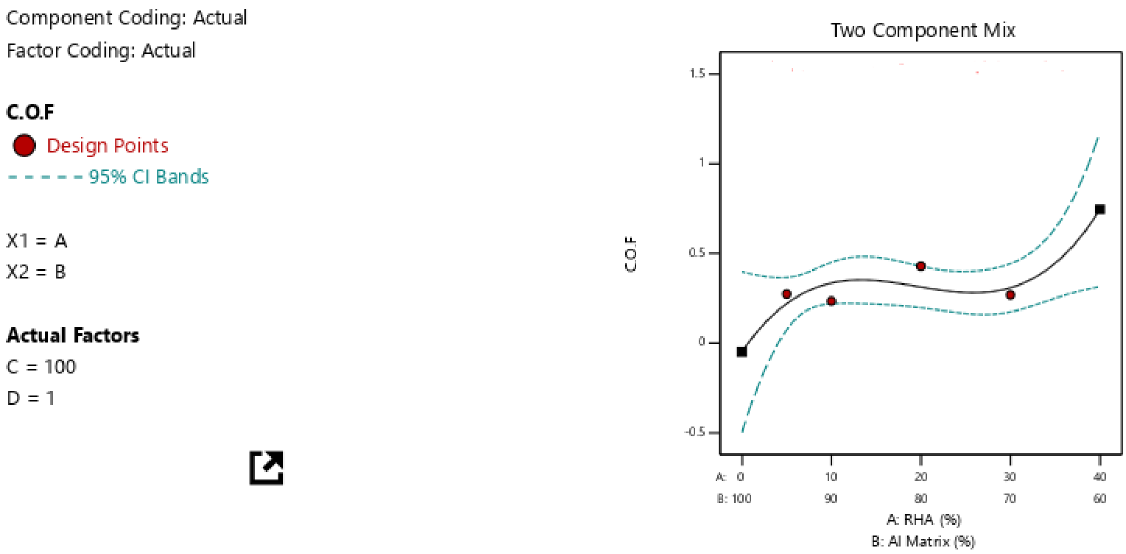

Figure 6: Coefficient of friction value effects of aluminium and rice husk in the composite.

4.3 Response Optimization

Table 6 shows the factors, responses and their respective objective criteria used in this study. RHA content ranges from 0 to 40%, while aluminium content is from 60 to 100% to maintain full composition. Stirring speed (100 to 300 rpm) is applied during compaction and mixing time from 1 to 3hrs. Hardness is maximized, wear rate is minimized, and coefficient of friction is kept within a range of 0.3 to 0.5.

Table 6: Optimization criteria.

Factors/Responses	Objective criteria
Rice husk ash %	Within range 0 to 40
Aluminium %	Within range 60 to 100
Stirring speed (rpm)	100 to 300
Mixing time (hrs)	1 to 3
Hardness (HV)	Maximize
Wear rate	Minimize
Coefficient of friction	Within range 0.3 to 0.5

Table 7: Optimization results.

No.	RHA %	Al Matrix %	Stirring Speed rpm	Mixing Time hr	Hardness HV	Wear Rate	COF	Desirability	
1	12.547	87.453	169.812	1.000	108.326	0.000	0.307	0.851	Selected

The result of the optimization was shown in Table 7. The optimized model compositions plots of the hardness, wear rate and coefficient of friction are respectively shown in Figs. 7, 8 and 9. The result of Figure 7, 8 and 9 showed that the hardness was maximized at 108.326 HV, wear rate minimized at 0.00 and coefficient of friction at 0.3 for a composition of 12.5474% rice husk ash and 87.4526% aluminium.

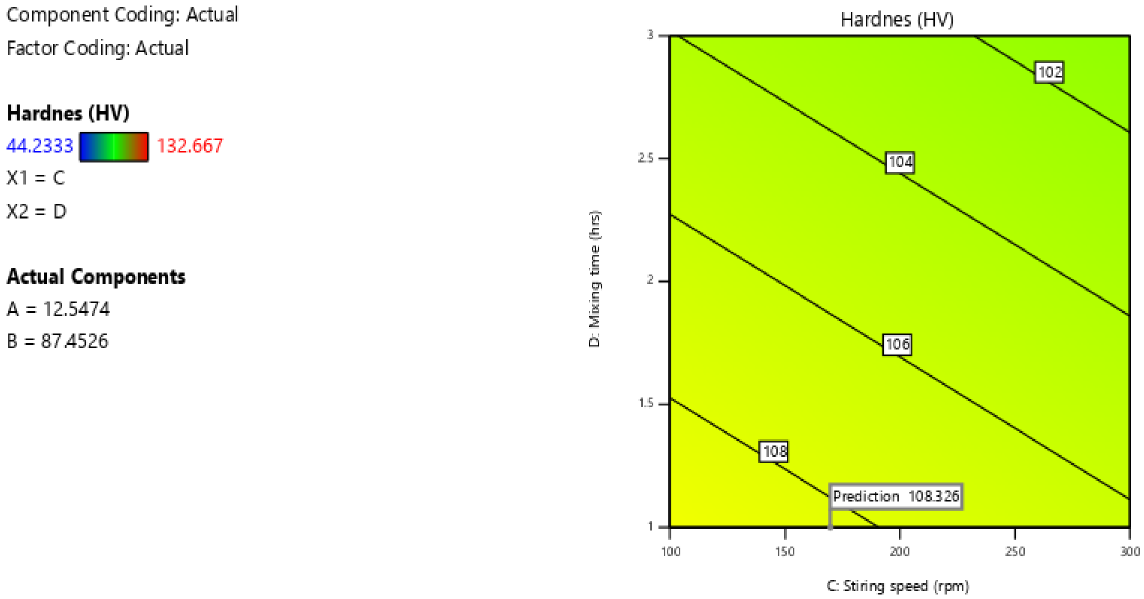

Figure 7: Composition plot of the hardness.

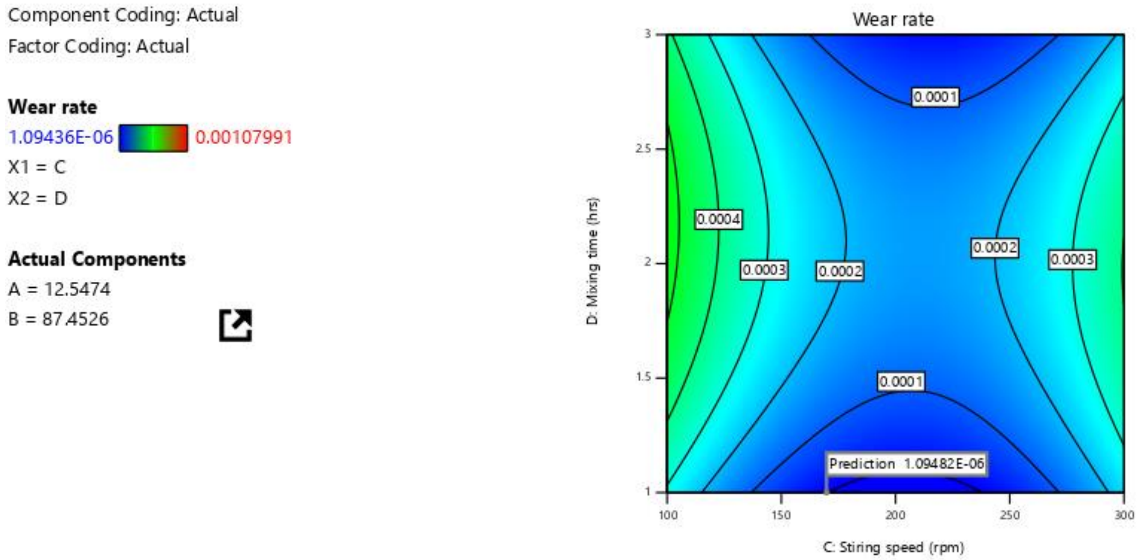

Figure 8: Composition plot of the wear rate.

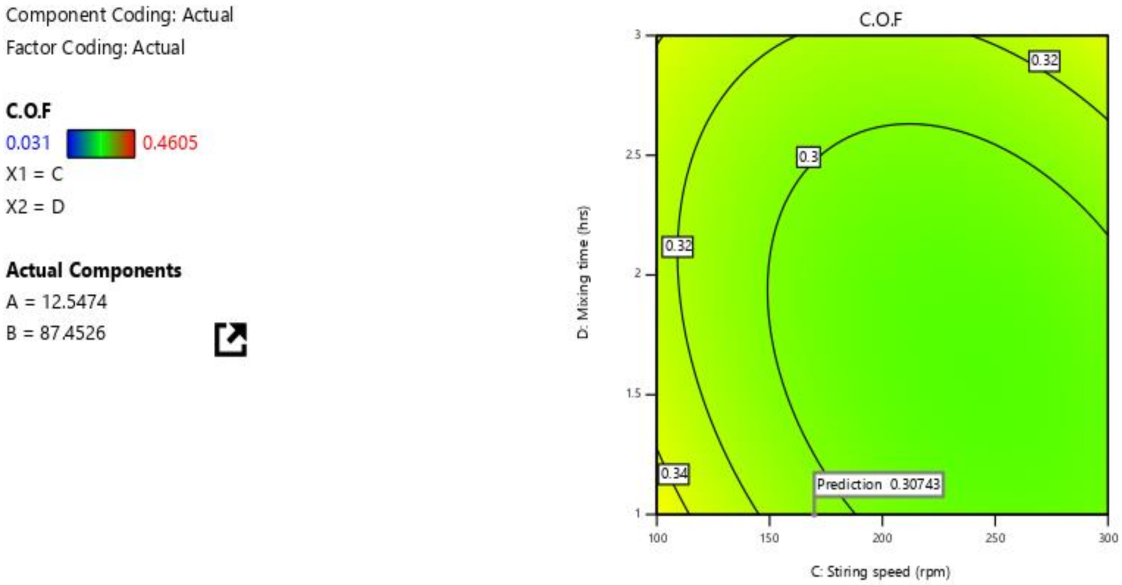

Figure 9: Composition plot of coefficient of friction

CONCLUSIONS

1. An optimized composite material of pure aluminium (Al) reinforced with rice husk ash (RHA) was developed using the powder metallurgy route.
2. The mixing parameters (speed and time) were optimized using a mixture design experiment in Design Expert 13 software.
3. The mechanical and tribological properties, specifically hardness, wear rate, coefficient of friction (COF) were evaluated.
4. Before the reinforcement (RHA) was added, the pure aluminium exhibited a hardness of 61.36666667HV, a wear rate of 0.000013889mm/Nm, and a coefficient of friction of 0.3215.
5. The optimization process resulted in an optimal composition of 12.552% RHA and 87.448% aluminium, with a stirring speed of 169.812rpm and a mixing time of 1 hour.
6. The composite achieved a hardness of 108.326HV, a wear rate of 0.000mm^3/Nm, and a coefficient of friction of 0.307.
7. The optimization led to a percentage improvement of 76% in hardness, a 100% reduction in wear rate, and a 4.1% reduction in coefficient of friction. The reduced coefficient of friction remains within the optimal range for effective braking application.

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