



Dry Sliding Behaviour Study of Novel Low-metallic Friction Materials by using DoE-Taguchi Method

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ABSTRACT

With the plethora of automobiles introduced in the last 2 decades, brake emissions have been a notorious contributor to overall emissions. In the present work, the low-metallic friction material is developed (for 3 samples accounting for 10 different ingredients) to reduce non-exhaust emission. The friction materials are manufactured by the compression molding method and various samples are required for physical, mechanical and tribological characterization are prepared as per ASTM standards. The tribological performance is tested by a 'pin on disc' apparatus. The tribological parameters such as speed, load and sliding distance is selected by considering for the scooter application. Taguchi Design of Experiment (DoE) is used to find optimal operating parameters. Additionally, ANOVA and regression analysis are also done. Results reveal that the wear rate is minimum at the optimal operating parameters. The average wear rate obtained from sample 3 is less than samples 1 and 2. The higher and lower wear rate and coefficient of friction for sample 3 are 0.002 and 0.00033 mg/m and 0.462 and 0.301, respectively. The morphological behaviors are studied with the help of SEM. Moreover, Thermo gravimetric analysis (TGA) is carried out to explore the thermal behavior of friction material samples. Results illustrate that sample 3 proves to be a potential substitute as a novel brake friction material.

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1. INTRODUCTION

Development of low metallic eco-friendly friction material is a challenging task in front of researchers with concern environment and health. The brake pad composition contains several metallic and other ingredients in the form of binder, filler, reinforcement, abrasives, and lubricants (1-4). Some of the toxic elements in brake friction material pollutes the environment are Asbestos, Cu, Cd, Ni, Pb, Sb, Zn, etc. and responsible for human diseases like lung cancer, irritation to the respiratory system, kidney failure, liver cells, and the tissues damage. Environmental agencies of different countries in the world are found the bad effect of brake pads emitted pollutants on plants, animals, and human beings (5-7).

In non or low metallic and organic brake friction materials natural organic fibers like cotton, kapok, jute,

sisal, abaca, pineapple, sun hemp, oil palm, kenaf, coir, banana, flax, wheat straw, bamboo, and inorganic fibers such as metals, steel wool, man-made vitreous fibers (MMVF) glass, carbon, ceramic fibers, etc. are reinforced with modified resins and rubber. The other constituents contain filler, abrasives and elastomer are added to enhance hardness, wear resistance, coefficient of friction, and shock-absorbing properties of friction material. In low metallic friction materials some of the binders used are phenolic, epoxy modified, COPNA, cyanate ester, silicon modified resins, etc (8). The researchers also made eco-friendly brake friction material from seashell and agricultural waste to reduce non exhaust pollution (9, 10). The structural changes in brake friction material also effects on performance parameters (11).

Selection and deciding the percentage of ingredients play a crucial role in the formulation of friction material.

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The trial and error method is generally obeyed for the formulation of brake composition that depends on the experience of the researcher. But a drawback of this method is very time-consuming. A lot of iteration has to be done for the formulation of the material. The important tool employed to overcome this difficulty is the design of experiment (DoE). Taguchi method is a design of experiment tool that tests a pair of combinations such as manufacturing and operating parameters instead of testing all possible combinations like factorial design (12). With DoE concern, design like Taguchi, factorial and response surface are the tools used to obtain the rank of influenced parameters on response. The combinations of parameters that influence the response are also studied in Taguchi design. ANOVA and multiple linear regression analysis models for wear rate are obtained. An optimum value of wear rate and coefficient of friction is obtained from signal to noise ratio by considering smaller is better and larger is the best quality criteria respectively. Results showed that wear rate is a function of load but decreases with an increase in speed. The percentage of reinforcement is also a major factor in the coefficient of friction (13, 14). The dry sliding behavior of composite is studied by making specimen hand layup technique or compression molding method. Most of the time authors are selected sliding distance, load, speed, percentage of reinforcement, and friction modifier as operating parameters with their two or three levels. The outputs of all experiments are to study performance parameters like coefficient of friction, wear rate, and specific wear rate (15, 16).

The present work aims to develop novel friction material from low metallic and more natural ingredients that reduces toxic producer metallic ingredients at sources and helps in reducing non-exhaust pollution. The friction material is prepared by the compression molding method and dry sliding tribological behavior is studied by using a wear test. The optimal combinations of operating parameters that have improved the performance of friction material are found by using the design of the experiment (Taguchi design). The S/N ratio quality characteristics are considered for wear rate. Regression analysis is used to prepare the model for a low wear rate. Morphological and thermal behavior of friction material is studied by scanning electron (SEM) and Thermo gravimetric analysis (TGA) method.

2. MATERIAL AND METHODS:

Friction material composition is made by varying four ingredients by volume percentage viz. Epoxy resin, boron carbide, cashew dust, and human nail nevertheless the volume percentage for vermiculate, sodium silicate, rubber crumb powder, zirconium dioxide powder, steel wool, and glass fiber ingredients are kept constant as

displayed in Table 1. The flow chart of manufacturing method is shown in Figure 1.

The pin specimen of 12 mm diameter is fabricated by preparing mold followed by crushing, sieving, mixing, and compaction procedure. The compressive molding technique is used to fabricate pin specimens. The homogeneous mixture of all the ingredients is mixed and poured into the mold at room temperature. The mold is kept under a compression molding machine by compressing the mixture with pressure 17 MPa. For the post-curing, the mold is further kept in the furnace at 150°C for 6 hours. The pin is withdrawn from the mold after 24 hours. To ready the pin for the test, the finishing of the pin is done to remove bur or any foreign matter by grit paper.

3. DESIGN OF EXPERIMENT

The Taguchi design of experiment (DoE) method is used to optimize various parameters or factors by considering

TABLE 1. Volume % of three different samples

Name of Ingredients	Sample 1	Sample 2	Sample 3
Epoxy resin	32	33	34
Cashew shell powder	31	26	21
Boron carbide	3	8	13
Human nail	3	2	1
Steel wool	3	3	3
Glass fiber	1	1	1
Vermiculate	12	12	12
Zirconium dioxide	5	5	5
Rubber crumb	3	3	3
Sodium silicate	7	7	7
Total	100	100	100

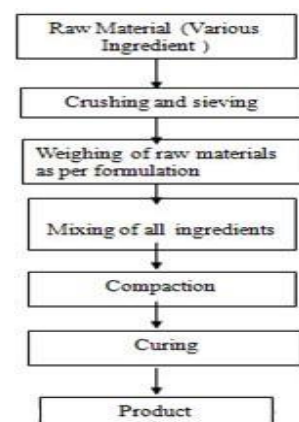


Figure 1. Flow chart of friction material manufacturing method

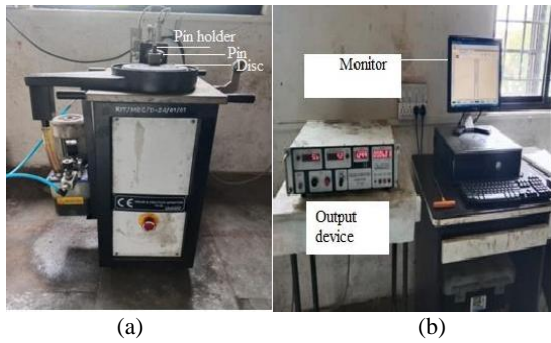


Figure 2. a) Pin on disc wear test rig, b) Output monitoring device

signal to noise ratio (S/N ratio). For the brake pad application coefficient of friction should be high and low wear rate. Signal-to-noise ratio considered for the coefficient of friction is larger is best while smaller is better is considered for wear rate. Equation 1 is used to calculate the S-N ratio smaller is better for the wear rate (17, 18).

$$\frac{S}{N} \text{ ratio} = -10 \log_{10} \frac{1}{n} \sum (y^2) \tag{1}$$

where, n is the number of experiments and y denotes response for wear rate The control factors and their levels are shown in Table 2.

4. DRY SLIDING WEAR TEST

Pin-on disc wear and friction monitor apparatus are shown in Figure 2 (a) and (b). (Ducom Instruments, Bangalore, India) is used to study dry sliding behavior of a sample pin according to ASTM G99 standard. The machine has a split hard jaw for clamping 3, 4, 6, 8, 10, and 12 mm cylindrical pins. The disc rotation range is between 200 rpm to 2000 rpm and the load range is 1 N to 200 N. The wear measurement capacity of tribo-tester is 2000 micrometers. In this study, a 12 mm diameter friction material pin is rubbed against a cast-iron disc having a diameter of 165 mm and 8 mm thickness. To uniform contact of the pin with the disc, the pin was ground with a SiC paper of 500 grits before starting the experiment. The time duration of each test is different according to sliding speed and sliding distance. The range of 900 rpm to 1300 rpm speed is selected by

TABLE 2. Control factors and levels

Levels	Factors	Load (N)	Speed (rpm)	Sliding Distance (m)
1		9.81	900	1000
2		19.62	1100	2000
3		29.43	1300	3000

considering the friction material used for moped or scooter application having average speed of 30 to 40 Km/h. Before starting and after completing the experiments weight of each specimen is recorded by electronic balance up to 4 decimals. Weight loss is noted to obtain wear rate by the Equation 2 (19, 20). Also, specific wear rate of material is calculated by calculating wear volume.

$$\text{Wear rate} = \frac{\Delta w}{S} \tag{2}$$

where,

Wear rate in mg/m

Δw - Difference in weight of pin sample before and after test in mg

S - Sliding distance in meter

5. RESULT AND DISCUSSION

5.1. Density Test Samples experimental density is found by ASTM D792 standard by Archimedes principle. The digital balance is used to measure the dry weight of the friction specimen and volume is measured by the water displacement method. The results showed that among the 3 samples, the high density obtained from sample 2 is 2.246 g/cm³ are shown in Table 3.

5.2. Friction and Wear Test The results obtained from the pin on disc wear and friction monitor revealed that for sample 1, at low speed 900 rpm and high level of load 29.43 N the wear observed is 53 microns while at

TABLE 3. Comparative properties of three different pin samples (21)

Properties	Sample 1	Sample 2	Sample 3	Commercial friction material
Density (g/cm ³)	1.566	2.246	1.735	1.89
Water absorption test (%)	5.7	4.07	4.99	NA
Oil absorption test (%)	6.4	5.12	5.74	NA
Rockwell hardness test (HRB)	61	80	89	101
Wear rate (mg/m)	0.0003-0.006	0.0003 - 0.003	0.0003-0.002	3.8
CoF	0.29-0.45	0.30 - 0.45	0.30 - 0.46	0.3-0.4
Specific wear rate * 10 ⁻¹²	0.014 - 0.130	0.007-0.045	0.006-0.057	NA
TGA at 200°C weight loss (%)	9	8.76	9.05	NA

low level of load and speed 9.81 and 900 rpm, respectively, it is 46.03 microns. At a load of 9.81 N and speed of 900 rpm, 1100 rpm, 1300 rpm, and sliding distances of 1000 m, 2000 m and 3000 m the average coefficient of friction value is 0.91 whereas at 19.62 N and 29.43 N for the same speeds and sliding distances the average value of coefficient of friction is 0.45 and 0.30, respectively. This reveals that as speed and load increases, the coefficient of friction decreases. This is because at higher speed only higher asperities are contact between a pin and disc that reduces contact area. The same behavior was observed in the case of sample 2, the average wear value is less as compared to sample 1 due to increase in boron and epoxy volume percentage. Sample 3 showed the average coefficient of friction 0.9 at 9.81 N load, 900, 1100, 1300 rpm speed and 1000, 2000, and 3000 m sliding distances whereas at 19.62 N and 29.43 N load for the same speeds and sliding distances the average coefficient of friction is 0.455 and 0.31, respectively. The average coefficient of friction values in sample 1 at the load of 19.62 and 29.43 is found 0.459 to 0.298, respectively while samples 2 and 3 show a slight increase in coefficient of friction 0.304 and 0.459 and 0.301 to 0.462, respectively are shown in Table 3.

5. 3. Taguchi Experiment The DoE is carried out using the Taguchi method of optimization the levels and quality characteristics are shown in Table 2 and Equation 1. The design of the experiment is conducted using Minitab 17, the orthogonal array L27 is selected. The results showed that sliding distance is the dominant factor are shown in Table 4, having significant p-value of 0.001 whereas the speed and load are not significant illustrating

in Table 5. For the optimal operating parameters, 29.43 N load, 1100 rpm speed, and 1000 m sliding distance, the low wear rate obtained in 1, 2, and 3 samples are 0.006, 0.003, and 0.002 mg/m, respectively are shown in Table 6. The main effect plot for means and the main effect plot for the S-N ratio are shown in Figure 3. The regression equation for the wear rate is shown in Equation 3.

5. 4. Taguchi Confirmation Test

Taguchi confirmation test is conducted for sample 3 with optimal operating parameters 29.43 N load, 1100 rpm speed and 1000 m sliding distance. From the regression equation predicted wear rate of 0.021 is calculated. After the experiment, the wear rate for sample 3 is 0.002 mg/m. The percentage error between predicted and experimentation 4.76 is obtained. The results are shown in Table 7.

5. 5. Regression Equation

$$\text{Wear rate} = 0.00534 + 0.000001 \text{ Load} - 0.000002 \text{ Speed} - 0.000001 \text{ Sliding Distance} \quad (3)$$

TABLE 4. S/N ratio response table for wear rate

Level	Load	Speed	Sliding Distance
1	58.15	57.02	51.76
2	60.89	61.54	61.42
3	58.95	59.43	64.81
Delta	2.74	4.52	13.05
Rank	3	2	1

TABLE 5. ANOVA result table showing significance levels of different parameters

Source	DF	Adj SS	Adj MS	F-Value	P-Value	level Significant
-Regression	3	-0.00002	-0.000007	5.47	0.005	Significant
Load (N)	1	0	0	0	0.98	Not Significant
Speed (rpm)	1	-0.000002	-0.000002	2	0.17	Not Significant
Sliding distance (m)	1	017-0.000	017-0.000	14.4	0.001	Significant
Error	23	-0.000028	-0.000001			
Total	26	-0.00004				

TABLE 6. Optimal operating parameters for wear rate

Parameters	Load (N)	Speed (rpm)	Sliding Distance (m)	Wear rate (mg/m)
Sample 1	29.43	1100	1000	0.006
Sample 2	29.43	1100	1000	0.003
Sample 3	29.43	1100	1000	0.002

5. 6. Optimal Combination Results Graph

The combined behavior pattern of coefficient of friction versus time and wear in micron versus time are shown in Figure 4. (a) and (b) at 29.43 N load, 1100 rpm speed and 1000 m sliding distance respectively. In Figure 4 (a), the slightly fluctuation in the coefficient of friction is observed due to stick and slip phenomena between tribo-

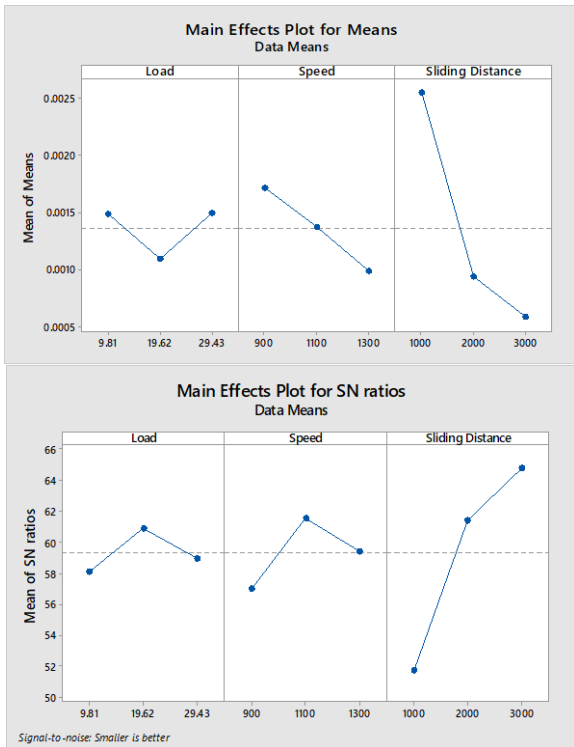


Figure 3. Main effect plot of means and Main effect plot of S-N ratio

TABLE 7. Taguchi experiment confirmation test result

Sample No.	Test	Taguchi confirmation test results		
	Optimal operating parameter test	Wear rate (experimentally)	Wear rate (predicted)	Error %
Sample 1	29.43 N,	0.0022	0.0021	-4.76
Sample 2	1100 rpm and 1000 m	0.0019	0.0021	9.52
Sample 3	m	0.002	0.0021	4.76

pair. The wear in sample 3 is lower as compared to sample 1 and 2 is shown in Figure 4(b). This is due to increase in percentage of boron carbide (13%) and epoxy resin (34%) as compared to sample 1 and 2. Also hard particles of boron carbide increases primary plateaus that reduces rate of wear. Also at high speed 1300 rpm, the less asperities of tribo-pair are in contact that is responsible to reduce wear rate and coefficient of friction simultaneously.

5. 7. TGA Analysis

Thermo gravimetric analysis (TGA) of samples 1, 2 and 3 is shown in Figure 5. All samples show an average weight loss of 9 % at 200°C that is linear weight loss with temperature. From 200°C to 370°C, progressive weight loss is found. The weight loss

rate increased in all three samples that are noted 30% in progressive weight loss region. After 370°C, the sample 1, 2 and 3 lines differ and show a complete combustion region at temperatures 750°C, 800°C and 980°C respectively (22).

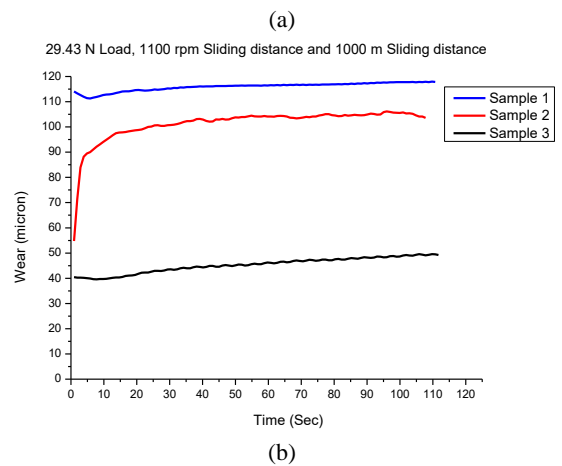
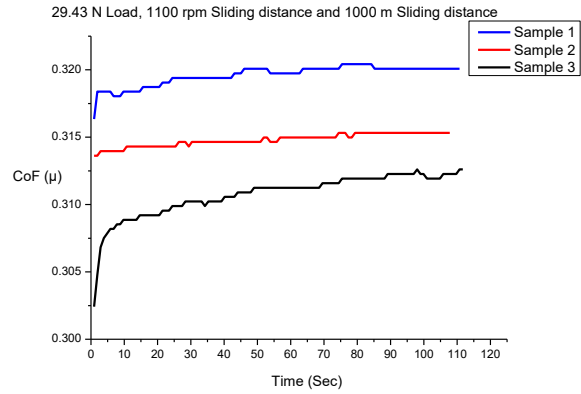


Figure 4. Optimal combination graph of sample 1, 2 and 3 a) Plot of coefficient of friction against time, b) plot of wear against time

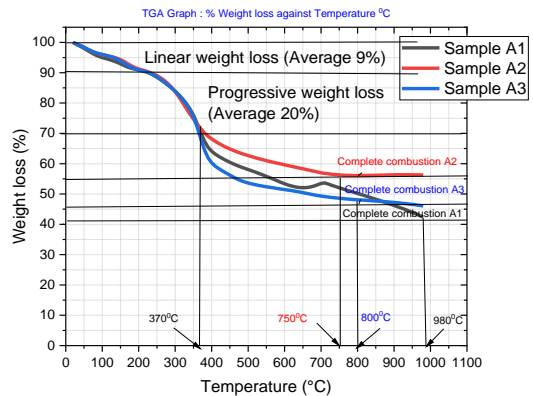


Figure 5. Thermo Gravimetric Analysis (TGA) of sample 1, 2 and 3

5. 8. SEM Analysis SEM are done for the wear test having operating parameters load, speed and sliding are shown in Figures 6-8(a) and (b) respectively. Scanning electron microscope (SEM) is used to study the wear of samples pin surface morphology of the friction material before and after the wear test. The micrograph reveals a wear debris pattern and the nature of surface appearance. The SEM is operated under high vacuum mode by providing grounding for earthling with carbon tape. On SEM image 4 nm resolution can be obtained. Two types of layers are formed, one is a loose granular type and another is a dense layer type. The primary plateaus are produced and served to adhere to fibers and flakes of ingredients that arrest fine wear debris in the tribe-pair interface. The primary Plateaus have a high load-bearing capacity and support the secondary plateaus. In the operation pin-on-disc test as the load increases, the load is first taken up by primary plateaus and then it is transmitted to secondary plateaus. Mostly loosely trapped hard particles of ingredients between pin and disc form the third body and are responsible for the generation of micro cracks. In the present work, hard particles of abrasive material such as boron carbide create a third body between the disc and pin surfaces and plough the material from the pin surface and form wear debris and pits on the contact surface of the pin. Also, there is a generation of active and passive debris, active debris is responsible for the supplementary wear mechanism that entrapped between tribe-pair surfaces and causes micro-

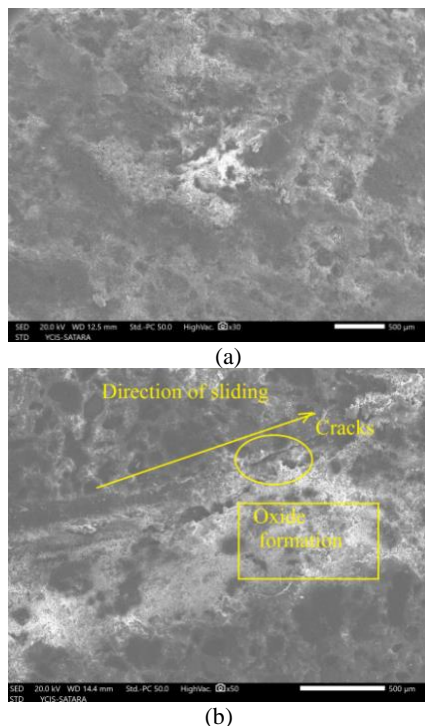


Figure 6. (a) SEM image before test (sample 1), (b) SEM image after test (sample 1)

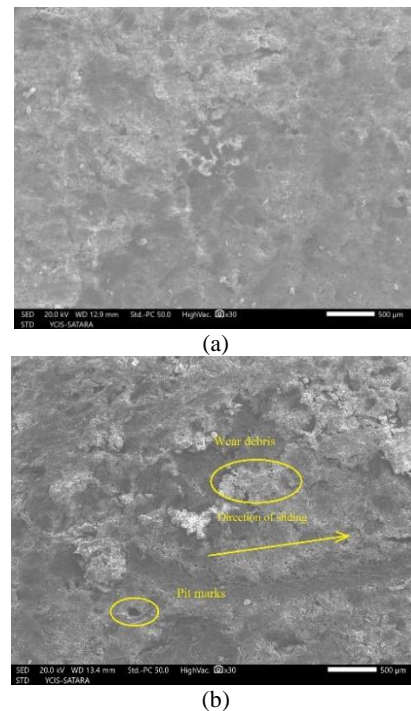


Figure 7. (a) SEM image before test (sample 2), (b) SEM image after test (sample 2)

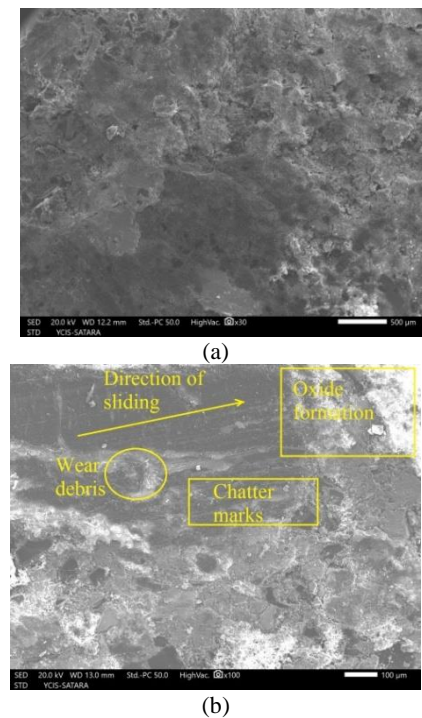


Figure 8. (a) SEM image before test (sample 3), (b) SEM image after test (sample 3)

cutting. At high speed, only a few asperities are contacted between the tribe-pair that generates small debris. In all three samples 1, 2 and 3, the wear of the pattern is clearly shown by yellow marks which are in the sliding direction.

6. CONCLUSION

The dry sliding behavior of three samples of friction materials is studied on wear and friction monitor pin on disc apparatus. By conducting experiments with the help of the Taguchi method, the findings reveals that 29.43 N load, 1100 rpm sliding speed, and 1000 m sliding distance, are the optimal operating parameters possesses the least wear rate (0.002 mg/m). The S/N ratio response table reveals sliding distance is dominant parameters among three operating parameters. From ANOVA p-value is 0.005 and 0.001 which are significant by considering confidence levels of 95 %. Sample 1 possesses highest average-wear-rate whereas sample 3 has lowest average-wear-rate (0.00033 - 0.002 mg/m). The average coefficient of friction obtained in sample 3 is 0.301 to 0.462. The properties of parent friction materials are compared with commercial friction material and find better results. With morphological behavior concerns, all samples shows wear debris accumulated over the surface after wear test. Some chatter marks also observed in the sliding direction. The thermo gravimetric analysis has recorded 9 % average weight loss at 200°C of all three samples. The complete decomposition temperatures for all 3 samples were 750°C, 800°C and 980°C, respectively. With low metallic concern it is possible to develop brake friction material for low duty applications that helps to reduce non toxic pollution.

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**Persian Abstract****چکیده**

با انبوهی از خودروهایی معرفی شده در 2 دهه گذشته، آلاینده‌گی ترمز یکی از عوامل بدنام در انتشار کلی بوده است. در کار حاضر، مواد اصطکاک فلزی کم (برای 3 نمونه که 10 ماده مختلف را تشکیل می‌دهند) برای کاهش انتشار غیر آگروز توسعه داده شده است. مواد اصطکاک به روش قالب‌گیری فشاری تولید می‌شوند و نمونه‌های مختلفی برای توصیف فیزیکی، مکانیکی و تریبولوژیکی طبق استانداردهای ASTM مورد نیاز است. عملکرد تریبولوژیکی توسط دستگاه "پین روی دیسک" آزمایش می‌شود. پارامترهای تریبولوژیکی مانند سرعت، بار و فاصله لغزشی با در نظر گرفتن برای کاربرد اسکوتر انتخاب می‌شوند. طراحی آزمایش تاگوچی (DoE) برای یافتن پارامترهای عملیاتی بهینه استفاده می‌شود. علاوه بر این، ANOVA و تجزیه و تحلیل رگرسیون نیز انجام می‌شود. نتایج نشان می‌دهد که نرخ سایش در پارامترهای عملیاتی بهینه حداقل است. میانگین نرخ سایش به‌دست‌آمده از نمونه 3 کمتر از نمونه‌های 1 و 2 است. نرخ سایش و ضریب اصطکاک بالاتر و پایین‌تر برای نمونه 3 به ترتیب 0.00033 mg/m و 0.002 و 0.462 و 0.301 است. رفتارهای مورفولوژیکی با کمک SEM مورد مطالعه قرار می‌گیرند. علاوه بر این، تجزیه و تحلیل گرما وزنی (TGA) برای بررسی رفتار حرارتی نمونه‌های مواد اصطکاک انجام می‌شود. نتایج نشان می‌دهد که نمونه 3 به عنوان یک ماده اصطکاک ترمز جدید، جایگزینی بالقوه است.