Predicting and analyzing of tribo-performance of NAO friction composites using Taguchi method

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Research Article

Keywords: Brake pad, Non-asbestos, Copper, Graphite, Taguchi, Pin-on-Disc, Friction, Wear, Heat

Posted Date: April 19th, 2021

DOI: <https://doi.org/10.21203/rs.3.rs-368545/v1>

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Abstract

The present study is aimed to investigate the dry sliding behavior of phenolic friction brake pad materials for industrial applications. Low metallic phenolic friction composites with addition copper-graphite (Cu-C) particles produced by traditional powder metallurgy methods. The friction test is carried out by pin-on-disc configuration on universal tribometer MMW-1 with hardened steel as a counterface material. The plan of experiments conducted by Taguchi's L_{27} orthogonal array on MINITAB 19.1.1 software using 3-level design model. Analysis of variance (ANOVA) was performed for predicting and analyzing the effect of design parameters like contact pressure (1.9, 5.75 and 9.6 MPa), sliding velocity (0.64, 1.57 and 2.5 m/s) and filler content (5, 10 and 15%) to tribological properties. Results of modeling and optimization of composites has showed that contact pressure has the greatest impact on the friction process following sliding distance and filler content. On the other hand, the most influential factor for the wear process was the sliding velocity, following contact pressure and finally filler content. It has also been determined that 5–10 Wt.% Cu-C filler content has an effective impact on tribological properties. The friction surface examination of the composites using a scanning electron microscope (SEM) revealed that Cu-C content has a significant effect on improving heat resistance properties.

1. Introduction

One of the most important parts of brake mechanisms is the brake pad or lining materials. The brake pad function is to either slow down or completely stop the movement by creating a frictional resistance against the rotating element of the device during the braking process [1]. The reliable operation and productivity of automobiles and industrial applications are determining by work of the brake systems [2]. The main requirement for friction materials in tribotechnical system is to ensure the rapid conversion of energy into heat and to meet complex requirements such as friction and keeping of tribological properties stability during multiple braking processes [3]. Stable friction characteristics, durability, noise and vibration minimization are considered to be the main norms when designing materials included in brake systems [4]. One of the main demands for brake pad materials is high stability of the friction coefficient in wide ranges of working temperature [5]. The high wear resistance during friction contact and keep its initial physical and mechanical properties, as much as possible, at rising temperatures allow these materials to work in heavily loaded tribotechnical systems [6]. The wear of materials used in friction units became inevitable. For this reason and to meet new industrial requirements there was always a need to study new materials.

Composite materials have become a common material for various industries fields due to its unique properties [7]. The brake friction materials are the most complicated structured among composite materials, consisting of multiphase constituent elements such as binders, fillers, modifiers and fibers [8]. Friction composites are mainly consisting of metallic and non-metallic components. Choosing the right composition is an important factor in the design of modern friction materials for improving reliability and durability. To meet the above discussions is highly depends on the selection of suitable ingredients. The

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other traditional materials, as in the case of melting, the mixing and integration of the ingredients must be provided with defined parameters [9]. Although polymers have not proved themselves in the industry enough as friction material, recent studies by researchers have shown that it is possibility to obtain completely new properties using nano-scale particles and various functional fillers [10, 11].

One of the most promising materials in terms of economically and environmentally that can meet the requirements of the tribotechnical system is polymer-based friction composite materials [12]. Due to these properties, polymer-based powder composite materials containing metal elements can be considered an ideal candidate to work in extreme operating conditions. Metal elements not only increase the required mechanical properties, but also have the ability to improve the friction properties [13].

The role of copper and graphite as solid lubricants in brake friction materials already studied by different researchers. An increase in graphite content reduces the coefficient of friction but depending on the shape and size, it also increases the wear resistance [14]. Copper increases thermal conductivity, improves sliding properties for different braking regimes [10]. As the nanocrystals on the surface recrystallize at high temperatures, a new brittle friction layer is forming, which acts as a solid lubricant, like graphite. Although the new friction layer reduces the coefficient of friction, it eventually mixes with other material particles released from the contact area of the disc and brake pad, stabilizing the tribological properties [15]. However, the copper-graphite (Cu-C) effect is not studied as well and it has a particular interest in terms of studying surface characteristics.

The characteristics of the contact surfaces are very important for friction materials. The surface roughness of the contacting materials is not only related to the nature of the mechanical properties of the material, but also to the friction and wear process itself. Along with depending on the type of rubbing pair materials, as a result of interaction effects such as adhesion, surface fatigue, abrasive wear and tribochemical reactions in the contact surface also influence friction and wear mechanisms of tribological materials [16]. It is known that the process of friction of metallic materials is often characterized by plastic deformation and oxidation wear [17]. Besides, at elevated temperatures friction layer formation, degradation and decomposition of non-asbestos organic (NAO) also influences tribological properties [18, 19]. However, the simultaneous occurrence of several wear mechanisms makes it difficult to assess the friction process and requires a systematic approach.

In order to study these conflicting properties design of experiments (DOE) can be helpful. Taguchi approach is a widely used powerful statistical technique for multifactoral experiments (Taguchi and Konishi, 1987). Unlike traditional DOE methods, Taguchi technique uses “smaller-is-better”, “larger-the-better” or “nominal-the-better” criteria for achieving the best quality performance and allows evaluate the robustness of the process with numerous approaches [20]. The technique may be more valuable when selected control parameters are discrete and substantiated [21].

In the present study, produced friction composites with Cu-C particles subjected to the analyzing impact of the control parameter and the interactions on friction coefficient and wear rate behaviours. The design

parameters selected for experiments were determined based on the commercial materials used in the industry.

2. Materials And Methods

2.1 Fabrication of composites

The friction brake pad composites were fabricated using traditional powder metallurgy methods. Three specimens containing eleven ingredients were produced by ball milling, mechanical mixing in dry conditions (for 16 hours with a speed of 60 cycles/min on a horizontal drum mixer), pre-forming at 10MPa and hot pressing for 20 min. at 160°C under 25.5MPa pressure. The glycerin used as a plasticizer for improving powder processing. The manufacturing process is finished with the post-curing procedure in a muffle furnace for 5 hours in 140°C. The information about produced specimens formulation is given in Table 1.

Table 1
The composition of brake pad samples (Wt.%)

Sample codes	CG-5	CG-10	CG-15
Copper-graphite	5	10	15
Barite (balance)	28	23	18
Base composition	67	67	67

Base composition consists of following weight proportions: wollastonite (synthetic), 5%, aluminum dioxide 7%, lead 10%, tin 10%, silicon dioxide 7%, modified phenolic resin 25% and additionally, 1% molybdenum disulfide, magnesium oxide and brass particles. The copper-graphite particles obtained by the electrochemical method with grade GK-3 graphite (GOST 17022–76) with a grain size of 100–160 µm. The electrolyte consisted of: 89.2% $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, 0.06% Fe, 0.015% As, and 25% sulfuric acid H_2SO_4 (15–45 g/L of concentrated solution) [22]. The base composition (67% Wt.%) was kept constant and Cu-C content was used in three proportions (5, 10 and 15 Wt.%). The remaining composition was compensated with barite filler. Specimens were fabricated in cylindrical pin form in size of Ø 4.7 x 12.8 mm (Fig. 1a). Brake pad composites codes called CG-5, 10 and 15 represent specimens containing 5, 10 and 15 Wt.% Cu-C particles respectively.

2.2 Plan of experiments

In this study, MINITAB 19.1.1 software used to analysis and correlate responses between different variances. $L_{27} (3^3)$ orthogonal array was chosen to design the planning matrix according to the Taguchi technique. Analysis of variance (ANOVA) was performed with 95% confidence level. Three control factors were used to study the friction and wear performance of the composite materials: contact pressure (A),

sliding velocity (B), and filler content (C). Each control level combination was repeated three times and the data evaluating was based on obtained average test results. Information on factors and selected levels is presented in Table 2.

Table 2
Design factors and their levels

Design factors	Code	Unit	Level I	Level II	Level III
Contact pressure	A	MPa	1.9	5.75	9.6
Sliding velocity	B	m/s	0.64	1.57	2.5
Filler content	C	Wt.%	5	10	15

The levels of contact pressure and sliding velocity are determined by testing of analog material Retinax B (FK-24A) (GOST 10851-94) under laboratory conditions. Retinax B (FK-24A) is commonly used asbestos brake pad material for industrial application, especially for medium and heavily loaded tribotechnical systems.

DOE was conducted mainly in five stages for estimating quality characteristics:

- Signal-to-noise (S/N) calculation
- ANOVA statistical analysis
- Obtaining linear regression model & cheking adequacy of model
- Optimization process parameters
- Confirmation test

Friction coeffcient S/N ratio was calculated with setting goal of the experiment to “larger-the-better” and wear rate S/N ratio was calculated using “smaller-is-better” criteria. S/N ratios friction and wear was determined using logarithmic loss function (1) and (2) respectively.

$$\frac{S}{N} = -10 \log \left[\frac{1}{n} \sum_{i=1}^n \frac{1}{y_i^2} \right] \quad (1)$$

$$\frac{S}{N} = -10 \log \left[\frac{1}{n} \sum_{i=1}^n y_i^2 \right] \quad (2)$$

where y is the response for the given factor level, n is the number of experiments.

2.3 Friction and wear test

The dry sliding friction tests were performed on a vertical MMW-1 tribometer with “pin-on-disc”

configuration at room temperature (25°C). The principle of friction tests is based on the simultaneous

clockwise rotating of three cylindrical pin samples on a stationary steel disc (Fig. 1). The quenched steel (hardness: 44-46HRC, surface roughness: $\sim 0.2\mu\text{m}$) with an outer diameter of 31.7 mm, an inner diameter of 16 mm and a thickness of 10 mm was used as the counter-face material. Load applied to the disc by the movement of the rotor which is installed at the bottom part of the machine (Fig. 2.).

Prior to each test, the surface of the disc and specimens was cleaned with ethyl alcohol to prevent lubrication. The surface of the specimens was grounded on a polishing machine with 100, 1000 and 2000-grid SiC papers. Worn samples were weighed on an electronic balance (Mettler Toledo) with an accuracy of ± 0.1 mg. The sliding distance was 1.5996 km for all tests.

The following formula was used to calculate the wear rates using weight loss measurements:

$$J = \frac{m_1 - m_2}{\tau} \quad (3)$$

where m_1 and m_2 are the mass before and after wear respectively (gram), τ is the duration of the experiment (hours), and J is the wear rate (g/hour).

3. Results And Discussion

3.1 Signal-to-noise (S/N) calculation

Taguchi generated design with three factors namely contact pressure (A), sliding velocity (B), and filler content (C) at three levels is used for conducting experiments. Table 3 exhibits the experimental results of tribotechnical tests based on the selected planning matrix and calculated S/N ratio values in accordance with the obtained data. Based on the S/N result, it is possible to determine which is the most influential factor in increasing the coefficient of friction.

Table 3
Experimental test output for L₂₇ orthogonal array design

№	Contact pressure (MPa)	Sliding velocity (m/s)	Filler content (Wt. %)	Friction		Wear	
				Friction coefficient (μ)	S/N ratio (dB)	Wear rate (g/hour)	S/N ratio (dB)
1.	1.9	0.64	5	0.403 $\pm$ 0.002	-7.89390	0.003 $\pm$ 0.001	50.4576
2.	1.9	0.64	10	0.409 $\pm$ 0.001	-7.76553	0.008 $\pm$ 0.002	41.9382
3.	1.9	0.64	15	0.395 $\pm$ 0.003	-8.06806	0.014 $\pm$ 0.002	37.0774
4.	1.9	1.57	5	0.391 $\pm$ 0.001	-8.15646	0.019 $\pm$ 0.001	34.4249
5.	1.9	1.57	10	0.396 $\pm$ 0.004	-8.04610	0.026 $\pm$ 0.001	31.7005
6.	1.9	1.57	15	0.385 $\pm$ 0.002	-8.29079	0.032 $\pm$ 0.002	29.8970
7.	1.9	2.5	5	0.375 $\pm$ 0.003	-8.51937	0.039 $\pm$ 0.003	28.1787
8.	1.9	2.5	10	0.380 $\pm$ 0.003	-8.40433	0.046 $\pm$ 0.002	26.7448
9.	1.9	2.5	15	0.367 $\pm$ 0.002	-8.70668	0.053 $\pm$ 0.001	25.5145
10.	5.75	0.64	5	0.425 $\pm$ 0.003	-7.43222	0.012 $\pm$ 0.002	38.4164
11.	5.75	0.64	10	0.431 $\pm$ 0.001	-7.31045	0.019 $\pm$ 0.001	34.4249
12.	5.75	0.64	15	0.417 $\pm$ 0.002	-7.59728	0.027 $\pm$ 0.001	31.3727
13.	5.75	1.57	5	0.412 $\pm$ 0.002	-7.70206	0.024 $\pm$ 0.003	32.3958
14.	5.75	1.57	10	0.417 $\pm$ 0.003	-7.59728	0.032 $\pm$ 0.002	29.8970
15.	5.75	1.57	15	0.393 $\pm$ 0.001	-8.11215	0.041 $\pm$ 0.002	27.7443
16.	5.75	2.5	5	0.396 $\pm$ 0.002	-8.04610	0.034 $\pm$ 0.001	29.3704

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Nº	Contact pressure (MPa)	Sliding velocity (m/s)	Filler content (Wt. %)	Friction		Wear	
				Friction coefficient (μ)	S/N ratio (dB)	Wear rate (g/hour)	S/N ratio (dB)
17.	5.75	2.5	10	0.403 $\pm$ 0.005	-7.89390	0.043 $\pm$ 0.003	27.3306
18.	5.75	2.5	15	0.388 $\pm$ 0.006	-8.22337	0.052 $\pm$ 0.001	25.6799
19.	9.6	0.64	5	0.441 $\pm$ 0.003	-7.11123	0.029 $\pm$ 0.001	30.7520
20.	9.6	0.64	10	0.445 $\pm$ 0.004	-7.03280	0.035 $\pm$ 0.004	29.1186
21.	9.6	0.64	15	0.439 $\pm$ 0.003	-7.15071	0.042 $\pm$ 0.002	27.5350
22.	9.6	1.57	5	0.436 $\pm$ 0.003	-7.21027	0.035 $\pm$ 0.003	29.1186
23.	9.6	1.57	10	0.440 $\pm$ 0.002	-7.13095	0.044 $\pm$ 0.002	27.1309
24.	9.6	1.57	15	0.431 $\pm$ 0.002	-7.31045	0.054 $\pm$ 0.001	25.3521
25.	9.6	2.5	5	0.412 $\pm$ 0.002	-7.70206	0.046 $\pm$ 0.004	26.7448
26.	9.6	2.5	10	0.416 $\pm$ 0.005	-7.61813	0.058 $\pm$ 0.005	24.7314
27.	9.6	2.5	15	0.395 $\pm$ 0.003	-8.06806	0.069 $\pm$ 0.001	23.2230

The average S/N values for each factor level are given in Tables 4 and 5. As can be seen from the results, the contact pressure is the most significant factor influencing the change in the coefficient of friction followed by sliding velocity and filler content, respectively. In contrast to the coefficient of friction, the most significant factor influencing the wear rate was the sliding velocity followed by contact pressure and filler content, respectively (Table 5).

Table 4
Response table for S/N ratios for friction coefficient

Level	Contact pressure (MPa)	Sliding velocity (m/s)	Filler content (Wt. %)
1	-8.206	-7.485	-7.753
2	-7.768	-7.729	-7.644
3	-7.371	-8.131	-7.948
Delta (Δ)	0.835	0.647	0.303
Rank	1	2	3

Table 5
Response table for S/N ratios for wear rate

Level	Contact pressure (MPa)	Sliding velocity (m/s)	Filler content (Wt. %)
1	33.99	35.68	33.32
2	30.74	29.74	30.34
3	27.08	26.39	28.16
Delta (Δ)	6.91	9.29	5.16
Rank	2	1	3

Figure 3a and b show the control factor dependence graphs of S/N ratios values for the friction coefficient and wear rate, respectively. Delta (Δ) is the average difference between the maximum and minimum response values for each factor. Taguchi suggests that response with a large S/N ratio indicates its importance on the process. As can be seen from Figure 3, graphs whose starting and ending points are farther from the horizontal line show that they have a greater effect. The graphs demonstrate that the most important factor influencing the increase in the coefficient of friction is the contact pressure. The most important factor influencing the wear rate is the sliding velocity. The obtained curves reflect the given values in Tables 4 and 5 and demonstrate the most significant factor effects. Defined impact ranges for both response values helped to determine the optimal regime parameters.

3.2 Statistical analysis (ANOVA)

Processing of experimental test results by ANOVA helped to identify the factors affecting the coefficient of friction (Table 6). As can be seen from Table 6, the P-values of the friction coefficient is zero for the

main factors, which shows their statistical significance, but the α -value of each of the interaction factor conditions was higher than 0.05.

Table 6
Analysis of variance (ANOVA) table for friction coefficient

Source	DF	Seq SS	Adj SS	Adj MS	F-value	P-value	P, %
Contact pressure (A)	1	0.006962	0.001660	0.001660	91.24		55.75%
Sliding velocity (B)	1	0.004140	0.000001	0.000001	0.06	0.809	33.16%
Filler content (C)	1	0.000364	0.000439	0.000439	24.14		2.92%
B*B	1	0.000078	0.000078	0.000078	4.30	0.052	0.63%
C*C	1	0.000554	0.000554	0.000554	30.46		4.44%
A*B	1	0.000024	0.000024	0.000024	1.32	0.264	0.19%
B*C	1	0.000019	0.000019	0.000019	1.03	0.323	0.15%
Error	19	0.000346	0.000346	0.000346			2.77%
Total	26	0.012488	0.001660	0.001660			100.00%

Table 7
Analysis of variance (ANOVA) table for wear rate

Source	DF	Seq SS	Adj SS	Adj MS	F-value	P-value	P, %
Contact pressure (A)	1	0.001644	0.000278	0.000278	27.99		24.53%
Sliding velocity (B)	1	0.003500	0.000503	0.000503	50.66		52.24%
Filler content (C)	1	0.001136	0.000027	0.000027	2.69	0.117	16.96%
A*B	1	0.000176	0.000176	0.000176	17.76		2.63%
A*C	1	0.000024	0.000024	0.000024	2.43	0.135	0.36%
B*C	1	0.000021	0.000021	0.000021	2.15	0.158	0.32%
Error	20	0.000199	0.000199	0.000010			2.96%
Total	26	0.006700					100.00%
<i>DF-degrees of freedom, Seq SS-sequential sum of squares, Adj SS-adjusted sum of squares, P-percentage of contribution</i>							

Significance levels below $\alpha = 0.05$ indicate their significance is low. Thus, the higher the value of F, the higher the effect on the process, and according to this principle, the impact percentage is determined in

the following order. Contact pressure is the most significant parameter with the highest percentage contribution (55.75%), following sliding velocity (28.48%), and finally filler content (2.92%). The most important of the interaction factors was the combination of filler content - filler content (C*C), which was 4.44%.

The percentage of factors influencing the wear rate is determined in the following order (Table 7). The most dominant factor is sliding velocity (52.24%), following contact pressure (24.53%) and filler content (16.96%). The most important of the interaction factor conditions was the combination of contact pressure - sliding velocity (A*B) at 2.63%.

The Pareto chart can be useful to better understand the effect of interactions. Figure 4. shows the Pareto chart for the main factors and their combinations. In both graphs reference line is 2.11. This shows that the factors crossing the reference line are statistically significant at the 0.05 level for the given model. The graphs show that the contact pressure and sliding velocity are the most influential factors for both output parameters. However, the Pareto chart does not show which factors increase or decrease the response.

Based on the results obtained, a 3D interpretation of the dependence of the friction coefficient and wear rate on the regime parameters is given in Figs. 5 and 6, respectively. As can be seen from Pareto charts, factor C (filler content) is the least important among the main factors. Therefore, in order to understand better the effect of process parameters on changes of response values, material factor (C) is not taken into account in 3D plots. As can be seen from Fig. 5, the increase in sliding velocity reduces the frictional characteristics of the material. This is due to the increased intensity of wear rate at high sliding velocity (Fig. 6). Changes in the contact surface as a result of wear lead to a decrease in the coefficient of friction. This dependence can be better seen in Figs. 9 and 10.

During sliding, material removal or gain occurs due to interactions between two solid surfaces. In the initial phase of sliding distance meeting of contact surfaces with each other, mainly resulting with plastic deformation. The occurrence of plastic deformation leading to the transformation of mechanical energy into frictional heat. Under frictional force in the surface of heterogeneous structured brake pads materials primary and secondary contact plateaus is forming [23]. In closer a look, if we approach it on a nano-scale, it is known that friction surface asperities are very rough. During the first stage of friction under big contact stress, harder asperities of the disc are breaks off softer brake pad asperities and form new real contact areas. Although the increase in the real contact areas has a positive effect on the friction characteristics, the structure of the contact surfaces changes again as a result of wear [24, 31]. For this reason, the highest coefficient of friction was at a contact pressure of 9.6 MPa and a sliding speed of 0.64 m/s (0.445). The coefficient of friction began to decrease more intensively after ~ 1.5 m/s. Kragelski, I. V. (1962) attributed the decrease in the coefficient of friction with the increase in the sliding velocity to the weakening of the formation of a strong bond between the two friction surface due to generated oxide layer. This is a phenomenon related to the mechanical properties of materials and may have different effects to friction depending on the properties of the individual components. Simultaneously, increasing

the sliding velocity also increases the frictional temperature at pairing surfaces. Generated heat during friction process increases interface temperature, which is resulting in decrease in friction coefficient [25]. As the temperature rises, changes in the tribological behaviour of contact materials occur. A great amount of dissipated energy flow is absorbing by the brake pad and affecting material properties. The high temperature generated on the contact surface is distributed from the interface layer into the material. The distribution of temperature within a material depends on the thermo-physical characteristics of that material [26]. At the end of the friction tests, two models of thermal effects were identified in the samples, especially in high contact and sliding velocity conditions. A graphical description of observed heat effects based on the cross-section of the specimens is given in Fig. 7a.

The first model (Fig. 7a) describes the completely burned contact surface of the sample with low filler content (CG-5). This can be understood as the low thermal conductivity of the CG-5 sample, due low content of copper-graphite. In samples CG-10 and 15, no significant difference was found in the contact surface and the other parts. This effect shows that heat is rapidly distributed and transferred within these materials, which also indicates that possibly increase of thermal conductivity by Cu-C particles [27]. The difference between CG-10 and 15 was only noticeable in images taken with scanning electron microscope (SEM) (Fig. 8). Figure 8 shows the SEM observations of worn surfaces of CG-5, 10 and 15. These observations cover 9.6MPa pressure and 2.5 m/s sliding velocity regimes (except Fig. 8c, it shows worn surface in 9.6MPa pressure and 0.64 m/s sliding velocity combination). Unlike the CG-15 sample, the number of cracks on the surface of the CG-5 was larger and deeper (Fig. 8b). Due to the low mechanical properties of CG-15, the wear was high and its complex wear mechanism reflected in the surface morphology. However, in the CG-15 sample, an increase in the sliding velocity was observed with microcracks (Fig. 8d). It is also possible to see the deepening of micro-voids, which indicates the intensification of the participation of metal particles such as aluminum oxide and silicon dioxide in the abrasive wear process with the degradation of organic components. Signs of plastic deformation are also visible in various areas of the friction surface which indicates that high Cu-C content affected the surface structure (Fig. 8c and d). Oxidation of Cu as a result of local temperature increase can influence wear mechanism by forming tribo-layer [28]. Oxidation influences were not observed in CG-15 as much as CG-5 and CG-10 specimens due to low copper content. Generated heat during friction affects the oxidation, thermal strength and plasticity of the material. This leads to changes in surface following complicated physical-chemical reactions (diffusion and adsorption of atoms or molecules on the friction surface) [29]. The change in the interaction between the surfaces as a result of structural changes is reflected in the dry frictional behaviour of brake pad composites. Graphs show that, the coefficient of friction does not decrease in high contact pressure regimes, on the contrary, an increase is observed (Figs. 5 and 9). This difference is especially noticeable in setting with low sliding velocity and high contact pressure settings. The reduction of friction coefficient can be explained decrease in the contact time at the asperities due increase of speed [30]. Due reduce of the contact time possibility of forming enough junctions for friction by adhesion is reducing. However, this process may be different depending on the properties of the brake sample surface and friction layer characteristics. When the pressure is high, the heating of the contact surface can cause to formation of a friction layer of solid lubricants, which

leads to the stabilization of the coefficient of friction [31]. Depending on thermo-mechanical properties of material sliding velocity is leading generation of non-uniform pressures with high local contact stresses in relative motion [32]. These effects are impacting surface morphology and cause non-stability of friction coefficient.

Mentioned effects on friction surface can be seen clearly in Fig. 8a and c. Since the wear of the friction layer is not high at low sliding velocity, this prevents the friction coefficient from decreasing over a period of time.

The lowest friction coefficient was observed at a contact pressure of 1.9 MPa and a sliding velocity of 2.50 m/s (0.367) (Fig. 9). Wt.% 15 of filler content in this combination is one of the reasons that reduces friction. This can be attributed to the formation of a copper-graphite lubricating film at the sliding surface. The graphite layer significantly reduces the contact of the specimen with the rotating disc surface. The formed film thickens due to the worn particles of the sample, further reducing the coefficient of friction.

An increase in wear rates was observed in a typical sequence - the increase in contact pressure and sliding velocity resulted in an increase in wear rate. The highest wear rate responses were observed at 9.6 MPa contact pressure and 2.50 m/s sliding velocity conditions (0.068 g/hour). The lowest was observed at a contact pressure of 1.9 MPa and a sliding speed of 0.64 m/s (0.003 g/hour) (Figs. 6 and 10). It should also be noted that, contour plots implies that filler content up to 10Wt.% is improving the wear resistance of samples. But 15Wt.% filler is not appropriate in terms of tribological properties. In CG-15 specimens wear rate values were high in comparison. When the contact pressure is high the graphite particles are squeezed out from the contact area and this leads to an increase in the wear rate [33]. This phenomenon causes a change in the structure of the friction surface. Surface roughness parameters are one main factor which is determining the contact behavior of rubbing pair materials. As discussed above frictional heat formed a surface roughness by changing the surface morphology in the CG-15 brake pad sample (Fig. 8c). During dynamic asperity - asperity contacting, depending on initial surface roughness elastic or a plastic deformation may occur [34]. These tribo-mechanical influences are impacting friction stability and wear of mating brake materials. Thus, surface roughness can also be considered as one of the main reasons for high wear rates in CG-15 specimens.

3.3 Obtaining linear regression equation & checking adequacy of model

The linear regression equation was applied to establish a correlation between the important factor conditions obtained with based to the ANOVA results, and the following equations (4) and (5) were obtained for the coefficient of friction and wear rate, respectively:

$$\text{Friction coefficient } (\mu) = 0.36633 + 0.005729 A + 0.00177 B + 0.00721 C - 0.00418 B*B - 0.000384 C*C - 0.000396 A*B - 0.000269 B*C \quad (4)$$

Wear rate (g/hour) = $-0.01997 + 0.003427 A + 0.01828 B + 0.000716 C - 0.001071 A*B + 0.000074 A*C + 0.000287 B*C$ (5)

The positive regression coefficients indicate that response values increase with selected control factors and negative regression coefficients suggest that response values decrease with selected control factors. Information about the coefficients related to the models can be found in Tables 8 and 9.

Table 8
Regression coefficients for friction coefficient

Term	Coef	SE Coef	T-value	P-value
Constant	0.36633	0.00922	39.75	
Contact pressure (A)	0.005729	0.000600	9.55	
Sliding velocity (B)	0.00177	0.00722	0.24	0.809
Filler content (C)	0.00721	0.00147	4.91	
B*B	-0.00418	0.00201	- 2.07	0.052
C*C	-0.000384	- 0.000070	- 5.52	
A*B	-0.000396	- 0.000344	- 1.15	0.264
B*C	0.000269	- 0.000265	- 1.02	0.323
<i>Model summary: S = 0.0042659, R² = 97.23%, R² (adj) = 96.21%</i>				

Table 9
Regression coefficients for wear rate

Term	Coef	SE Coef	T-value	P-value
Constant	-0.01997	0.00524	- 3.81	0.001
Contact pressure (A)	0.003427	0.000648	5.29	
Sliding velocity (B)	0.01828	0.00257	7.12	
Filler content (C)	0.000716	0.000436	1.64	0.117
A*B	-0.001071	- 0.000254	- 4.21	
A*C	0.000074	0.000047	1.56	0.135
B*C	0.000287	0.000196	1.47	0.158
<i>Model summary: S = 0.0031144, R² = 97.04%, R² (adj) = 96.15%</i>				

From Eq. (3) it is observed that contact pressure plays a major role on the friction coefficient. A number of factor combinations (B*B, C*C, A*B and B*C) were negatively related to the coefficient of friction. This is due to the processes occurring on the friction surface which is discussed above. Eq. (4) showed that wear rate highly influenced by sliding velocity and contact pressure. And thus, these factors increase the wear rate of brake pad specimens. Almost all of the selected factors and their combinations contribute to the increase of wear rates. Obtained regression equations can be used to predict tribological behaviours of the brake pad composites.

Residuals vs. Fitted Value graphs showing the adequacy of the model obtained by linear regression equations (4) and (5) are given in Fig. 11. The graph is reflecting the distribution of negative residual values below the zero line, and the positive results above that zero line in the vertical direction along the ordinate axis. Residual values are calculated by the following formula [35]:

$$e = y - \hat{y}$$

6

where e is residual, y is experimental response value and $\hat{y}$ is predicted response value.

According to the accepted distribution patterns, obtained results prove the adequacy of the model. Thus, in adequate models, residual values are usually characterized by random distribution in different directions above and below the zero line (presented in Minitab). For the friction coefficient, the largest deviations in the graph are the results for rows 15, 18 and 27 in the planning matrix given in Table 3 (Figure 11a). Each of the above-stated rows is containing a sample where the filler content is 15 Wt.%. In specimens from 5 to 10 Wt.% filler content, an error of row 15 occurred due to increased friction and wear.

The plots showing the distribution of the residual between the experimental results and the predicted values for the wear rate also show that this model is adequate for the process (Fig. 11b). Despite this, the unequal distribution of residuals can be seen in Fig. 11b. Red arrows show that residuals are increases with fitted response values magnitude. All large residuals are referring to the results for the first starting point of the third level of sliding velocity (2.5 m/s). The residual was higher in these rows because the effects at level 3 of the sliding velocity were different depending on the filler content.

As can be seen from basic information on both models in Tables 8 and 9, 97.23% of the predicted values of the model (3) coincide with the experimental results, which can be considered as a satisfactory result. The results of wear rate also were satisfactory. The coefficient of determination (R^2) for the wear rate (model 4) was 97.04%.

3.4 Optimization process parameters

The results of the optimization for the friction coefficient are given in Fig. 12. In the presented optimization graphs blue lines show optimal settings and black lines show the predicted response values at different regimes. As described in Fig. 9, the optimal friction condition is a combination with a contact

pressure of 9.6 MPa, a sliding velocity of 0.64 m/s and a content filler of 10 Wt.%. The predicted value of the friction coefficient in this regime is 0.451898.

The results of the optimization for the wear rate are given in Fig. 13. As shown in the figure, the optimal condition for the wear is at the lowest level of each factor: the contact pressure is 1.9 MPa, the sliding velocity is 0.64 m/s and the content filler is 10 Wt.%. The predicted value of wear rate in this condition is 0.0020093 g/hour.

3.5 Confirmation test

Checking the accuracy of the results is the last step in the planning methods of experiments. Table 10 exhibits the conditons selected for both response parameters. According to these parameters, the obtained results are given in Tables 11 and 12.

Table 10
Parameters used for confirmation test

Experiments, №	Contact pressure	Sliding velocity	Filler content
1	9.6	2.5	10
2	1.9	0.64	15

Table 11
Confrimention results for friction coefficient

№	Experimental	Predicted	Error (%)
1	0.416	0.418519	0.61
2	0.395	0.395407	0.10

Table 12
Confrimention results for wear rate

№	Experimental	Predicted	Error (%)
1	0.058	0.0558889	3.64
2	0.014	0.0140833	0.60

A comparison of the predicted values with the experimental values according to the regression model showed that the error was less than ~ 3% for both configuration, which is an acceptable result.

4. Conclusion

Using the Taguchi technique following conclusions are drawn based on statistical processing of obtained data from experimental friction and wear tests:

- P-values of selected design factors were higher than 0.05, which proves their importance to tribological properties.
- ANOVA results showed that contact pressure (55.75%) and sliding velocity (33.16%) is a most significant factor on the friction coefficient. Filler content impact was only 2.92%.
- Most significant variable on wear rate is sliding distance (52.24%), then contact pressure (24.53%) and filler content 16.96%.
- The highest friction coefficient (0.445) and lowest wear rate was obtained in brake pad composition containing 10 Wt.% copper-graphite particles.
- The lowest friction coefficient (0.367) and highest wear rate was found in brake pad composition containing 15 Wt.% copper-graphite particles.
- Friction coefficient increasing with the increase of contact pressure with lowest sliding velocity. but decreases with the increase of sliding velocity.
- Surface characterization with SEM and graphical scheme for frictional heat effect indicates that copper-graphite particles are increasing of heat conductivity of the composite materials. Brake pad specimen containing 10 Wt.% copper-graphite particles have better friction surface.
- From generated regression equations it is observed that contact pressure is positively related to process and plays a major role on friction coefficient. Also, the contact pressure and sliding velocity are positively related to the output parameter and serve to increase the wear rate.
- Optimal combination for friction coefficient was determined to be contact pressure 9.6 MPa, sliding velocity 0.64 m/s and for wear rate typically at the lowest level of parameters.

Declarations

Competing interests:

The author declares no competing interests.

References

1. Asif M, Chandra K, Misra PS (2012) Characterization of iron based hot powder brake pads for heavy duty applications. *Int J Mech Mater Eng* 8:94–104
2. Liew KW, Nirmal U (2013) Frictional performance evaluation of newly designed brake pad materials. *Mater Des* 48:25–33
3. Sugözü I, Mutlu IH, Sugoza KB (2015) The effect of ulexite to the tribological properties of brake lining materials. *Polym Compos* 39:1–8
4. Friedrich K (2018) Polymer composites for tribological applications. *Adv Ind Eng Polym Res* 1:3–39

5. Gård A, Hallbäck N, Krakhmalev P (2010) Temperature effects on adhesive wear in dry sliding contacts. *Wear* 268:968–975
6. Xingming X, Yan Y, Jiusheng B et al (2016) Review on the friction and wear of brake materials. *Adv Mech Eng* 8:1–10
7. Zishan HK (ed). *Recent trends in nanomaterials: synthesis and properties*, 2017, p. 305
8. Pradnya EK, Pradeep AP, Muthukumar M et al (2019) Experimental investigation of physical and mechanical properties of steel powder filled disc brake friction materials. *J Phys Sci* 30:81–97
9. Nilov A, Kulik V, Garshin A (2015) Analysis of friction materials and technologies developed to make brake shoes for heavily loaded brake systems with disks made of a ceramic composite. *Refract Ind Ceram* 56:57–68
10. Sanjeev S, Jayashree B, Mukesh K (2013) Comparison between nano-and micro-sized copper particles as fillers in NAO friction materials. *Nanomater Nanotechnol* 3:1–9
11. Kamal KK (ed). *Composite materials processing, applications, characterizations*, 2017, p.686
12. Ghosh P, Banerjee S, Khastgir D (2020) Elastomer modified phenolic resin-based composites with reduced scale friction: Influence of calcined petroleum coke on tribological and thermo-mechanical behavior. *Polym Eng Sci* 60:10: 1446–1458
13. Shagil A, Mohammad S, Mohd RM et al., Recent advancements in powder metallurgy: a review. *Materials Today: Proceedings (Materials Processing and characterization)*, 5 (9), March 16–18, 2018, Madhapur, India, p.18649–18655
14. Jiazhi Y, Jing Z, Zhenxin D Effect of large size graphite on dry friction and wear characteristics of copper-based brake friction lining materials. *IOP Conference Series: Mater Sci Eng* 2018; 397: 1–7
15. Kumar M, Bijwe J (2011) Non-asbestos organic (NAO) friction composites: role of copper; its shape and amount. *Wear* 270:269–280
16. Grigoriev AY, Dubravina AM, Kovalev AV et al (2003) Measurement of contact adhesion and attraction between the engineering surfaces. *J Frict Wear* 24:51–58
17. Ding J, Leen SB, Williams EJ et al (2009) A multi-scale model for fretting wear with oxidation-debris effects. *Proc ImechE Part J: J Eng Tribol* 223:1019–1031
18. Singh T, Patnaik A, Gangil B et al (2016) Thermal stability analysis of nano-particulate-filled phenolic-based friction composite materials. *J Indus Textiles* 45:1335–1349
19. Guanchen L, Xiaoliang S, Yuchun H et al (2018) Effects of friction layer characteristics on the tribological properties of Ni3Al solid-lubricating composites at different load conditions. *Mater Res Express* 5:1–10
20. Ranjit KR. *Design of experiments using the taguchi approach: 16 steps to product and process improvement*. 2001, p.560
21. Gujrathi MS, Dhamande LS, Patare PM (2013) Wear studies on polytetrafluoroethylene (PTFE) Composites: Taguchi approach. *Bonfring IJEMS* 3:47–51

22. Мустафаев СМ, Шарифова АВ, Гулиев АА (2013) Исследования по разработке медь - графитовой композиции с повышенным содержанием графита. Прогресивні Технології і Системи Машинобудування 1:2: 187–191. (In Russian)
23. Barros LY, Neis PD, Ferreira NF et al (2016) Morphological analysis of pad-disc system during braking operations. *Wear* 352–353:112–121
24. Kim SJ, Min CH, Cho KH et al (2007) Complementary effects of solid lubricants in the automotive brake lining. *Tribol Int* 40:15–20
25. Sugozy B, Daghan A, Akdemir N, Ataberk (2016) Friction and wear properties of friction materials containing nano/micro-sized SiO₂ particles. *Indus Lubrication Tribol* 68(2):259–266
26. Bao J, Zhu Z, Tong M et al (2011) Dynamic friction heat model for disc brake during emergency braking. *Adv Sci Lett* 4:3716–3720
27. Yusubov FF (2020) Influence of surface structure of sintered compositions with copper–graphite particles on their tribotechnical properties. *J Frict Wear* 41:125–128
28. Akbarpour MZ, Alipour S, Farvizi M et al (2019) Mechanical, Tribological and electrical properties of Cu-CNT composites fabricated by flake powder metallurgy method. *Arch Civ Mech Eng* 19:694–706
29. Jiusheng B, Yin Y, Zhu Z et al (2012) Wear Modalities and Mechanisms of the Mining Non-asbestos Composite Brake Material. *Appl Compos Mater* 20:331–339
30. Straffelini G *Friction and wear methodologies for design and control*. Springer, 2015, p.283
31. Piyush CV, Rodica C, Andrea B et al (2016) Role of the friction layer in the high-temperature pin-on-disc study of a brake material. *Wear* 346–347:56–65
32. Petinrin MO, Oji JO (2012) Numerical simulation of thermoelastic contact problem of disc brake with frictional heat generation. *New York Sci J* 5:39–43
33. Baradeswarana A, Perumal AE (2015) Effect of graphite on tribological and mechanical properties of AA7075 composites. *Tribo Trans* 58:1–6
34. Bikramjit B, Mitjan K. *Tribology of ceramics and composites: a materials science perspective*, Wiley, 2011, p.550
35. Teruo M. *Taguchi methods benefits, impacts, mathematics, statistics, and application*. ASME Press, 2011, p.800

Figures

Figure 1

a:left) Cylindrical specimens and b:right) disc used in friction tests

Figure 2

Scheme of pin-on-disc contact: a:left) top and b:right) front view

Figure 3

Main effects plot for S/N ratios of a:top) friction coefficient and b:bottom) wear rate

Figure 4
 Pareto chart of the standardized effects for a:left) friction coefficient and b:right) wear rate

Figure 5
 Surface plot for dependence between friction coefficient and regime parameters

Figure 6

Surface plot for dependence between wear rate and regime parameters

Figure 7

Frictional heat effect on specimen a:left) CG-5 b:right) CG-10 and 15

Figure 8

SEM images of worn surfaces of composites a) CG-5, b) CG-10 c) CG-15 ($B=0.64$ m/s) and d) CG-15

Figure 9

Contour plot for dependence between friction coefficient and regime parameters

Figure 10

Contour plot for dependence between wear rate and regime parameters

Figure 11

Residual plots of linear regression model for a:left) friction coefficient and b:right) wear rate

Figure 12

Setting and optimal solution for friction coefficient

Figure 13

Setting and optimal solution for wear rate