

Tribological Properties of Friction Materials Developed from Non- Asbestos Materials using Response Surface Methodology

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ABSTRACT

Over many years, asbestos has been used as reinforcement material in the production of brake pads production but it has lost favour due to its carcinogenic nature, as a result, there is need to investigate other possible substitute which can offer similar tribological properties as the carcinogenic material (asbestos). Several works has been carried out using different reinforcement material with the aim of finding a possible replacement for asbestos. In this work, Rule of mixture (ROM) was used for sample formulation and the tribological properties of natural based material (coconut shell and seashell) were investigated using experimental design (response surface methodology) and multi-response optimisation technique (Grey relational analysis). The multi-response performance of the formulated brake pads samples was compared with a commercial brake pad sample. The research findings revealed that sample can be produced using 52% reinforcement, 35% binder, 8% abrasive and 5% friction modifier while the Grey relational analysis (GRA) showed that optimum multi-response performance of the developed coconut shell based sample can be achieved using MP, MT and CT and HTT of 12MPa, 100 °C, 6mins and 2hrs respectively while that of the developed seashell based brake pad can be achieved using MP, MT and CT and HTT of 10MPa, 160 °C, 12mins and 2hrs respectively. Also, the Analysis of variance (ANOVA) results show a percentage error of less than 5% indicating minima noise effect. In addition, the optimized coconut shell-based brake pads falls within the category of class H ($\mu > 0.55$) type of brake pads while seashell based sample falls within the class G ($\mu: 0.45-0.55$) type of brake pads. It therefore concluded that the use of coconut shell can serve as a better substitute for asbestos-based brake pads.

Keywords: Brake pad, Response surface methodology, tribological properties, Grey relational analysis.

1 INTRODUCTION

Friction materials are materials used for the development of automobile parts such as clutch and brake pads which are utilised in the transmission and braking of various machineries like cars, aircraft, motorcycles and other automobile systems. The constituents kept varying with the aim of meeting up with environmental technology and emerging. Blau (2001) reported that friction materials can be classified as semi-metallic, organic and carbon-based, depending on the elemental composition. Mechanics working on automobile are in most cases exposed to asbestos dust in several ways. This include, grinding of friction brake or clutch, repair work on brakes and clutch, where accumulated dusts are always wiped off before the old ones are replaced using brush (Abutu et al., 2018). All these methods are capable of causing asbestos particles to become airborne which is very hazardous to the environment. Dagwa and Ibhaddode (2008) reported that if old brake pads are still hard enough to be applied, automobile mechanics working on them often utilize a bench grinder to normalize the surface, or dissolve the dirt of the lining which often lead to the release of the particles of asbestos, thereby putting human at risk of contacting diseases such as pleural, peritoneal or pericardial mesothelioma, asbestos related cancer and asbestosis (Norton, 2001). Also, Mutlu et al. (2009) reported that tribological properties are very important properties in the performance of brake pads and a relatively high friction coefficient in the range of 0.3-0.7 and lower wear rate is normally desirable when using brake lining materials. Several works have been carried out with the aim of replacing asbestos as inclusion in brake pads production.

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Gabriel (2016) utilized periwinkle/palm shell as reinforcement material using Taguchi experimental design and reported that optimal performance of the developed friction material can be obtained using moulding pressure (140 KPa), moulding temperature (150 °C) and curing time (10 minutes) as process parameter. Also, Abutu et al. (2018) with the aim of finding a substitute for asbestos developed a friction material using seashell as reinforcement material and utilizing response surface-central composite design (RSM-CCD) technique and grey relational analysis (GRA). The authors reported that seashell can serve as good substitute for asbestos in friction material production and that multi-response performance of the developed material can be obtained using 1 hour heat treatment time, 12 minutes curing time, 160 °C moulding temperature and 14MPa moulding pressure. Also Ruzaidi et al. (2011) formulated a non-asbestos containing brake pad with varying composition of Polychlorinated Biphenyls (PCB) and palm ash waste along with thermoset resin as a binder and metal filler as abrasive. Five samples were produced using moulding pressure, moulding temperature and curing time of 122 MPa, 150 °C and 5 minutes respectively and were tested to examine its compression strength, water absorption rate, wear rate, and morphological properties. Experimental findings indicated that the optimum performance (mechanical and wear properties) of the brake pads was obtained using higher percentage of palm ash and also, wear properties of the developed brake pads compared satisfactorily with conventional brake pad. Also, Mutlu et al. (2009) with the aim of finding a possible replacement for asbestos whose dust is hazardous developed friction materials using Rice Husk Dust (RHD) and Rice Straw Dust (RSD) to study the tribological behaviour of brake pads. The materials in each brake pad were composed of RHD, RSD, copper particles, barite, brass, cashew, steel fibres, graphite and alumina. The newly formulated brake pads were tested in order to study their tribological performance and the results revealed a mean friction coefficient of 0.315 – 0.381 which is very low to be applied in heavy duty automobiles brake pads as specified in the work of Dagwa and Ibhado (2008) while the wear rate varies from 0.000853 – 0.001041 g/mm². In addition, Fono–Tamo and Koya, (2011), also developed friction materials using palm kernel shell combined with other materials and observed that the optimal mechanical properties of the developed material showed a hardness of 32.34 and shear strength of 40.95 MPa while the optimum coefficient of friction of was found to be 0.43. Also, Ikpambese et al. (2014) developed an asbestos-free friction material using palm kernel fibres as reinforcement material and reported that sample with 10% palm kernel fiber, 6% Al₂O₃, 40% epoxy-resin, 29% graphite gave and 15% calcium carbonate gave the optimum performance. Similarly, Bashar et al. (2012) utilized coconut shell powder as reinforcement material to develop friction material and found that high inclusion of coconut powder may result in brittleness of the composite and also samples with 60 % matrix and 10% reinforcement as well as 50% matrix and 10% reinforcement can be utilized in the production of friction materials. Yawas et al. (2016) also developed an asbestos-free friction material using periwinkle shell as reinforcement material and revealed that optimal periwinkle shell reinforced sample produced using sieve size of 125µm possessed specific gravity (1.01 g/cm³), coefficient of friction (0.41), hardness (116.7 HRB), compressive strength (147 N/mm²), and thickness swell in water (0.39 %) and thickness swell in SEA oil (0.37 %). Also, Ibhado and Dagwa, (2008) used palm kernel shell (PKS) as reinforcement material along with other constituents in the production of friction materials and found that optimal sample produced using 56% reinforcement, 24% binder, 14% abrasives and 6% friction modifier compared favourably with commercial samples as a result can serve as replacement for asbestos in friction lining production. Therefore, in this study, locally sourced coconut shell and seashell were separately used as reinforcement material along with other constituents to develop an environmentally friendly brake pad samples using Central composite design (CCD)-Response surface methodology (RSM) experimental design technique. The multi-response performance of the two brake pad samples was compared with commercially available brake pad by examining their tribological properties (wear rate and friction coefficient).

2. MATERIALS AND METHODS

2.1 Materials

Coconut shells (Figure 1a) were sourced locally from a local coconut trader in Sabon Tasha market, Kaduna – Nigeria, seashells (Figure 1b) were collected from a local seafood vendor in Lagos bar beach, Lagos-Nigeria. Also, alumina and commercial brake sample (Figure 1c) designed for Mazda 323 and produced by Ibeto Group of Companies were sourced from a commercial shop situated in Kaduna-Nigeria while epoxy resin used together with hardener (Sikadur 42T) were purchased from a chemical store located in Onitsha-Nigeria while graphite (Figure 1d) sourced from dry cell batteries (1.5 volt, TIGER).

2.2 Method

Development of brake pads constitute the preparation of sourced materials, experimental design using Minitab 17 software, compression moulding process, testing of developed samples and analysis of experimental results using signal to noise (S/N), analysis of variance (ANOVA) and Grey relational analysis (GRA).

2.2.1 Materials preparation

Materials preparation involved the preparation of the coconut shells, seashells and graphite powder. These involved washing, cleaning using tissue paper, drying under the sun for 24 hours followed by crushing with metallic mortar and pestle and thereafter, grinding using grinding machine situated at Samaru, Zaria and then sieving using a sieve size of 10µm.

2.2.2 Design of Experiment using RSM Design Technique

In this study, CCD via RSM experimental design consisting of moulding pressure (MP), moulding temperature (MT), curing time (CT) and heat treatment (HTT) was built in accordance to standard $L_{27}(2)^4$ using Minitab 17 statistical software. The factor levels of process parameters and experimental design matrix are shown in Table 1 and 2 respectively.

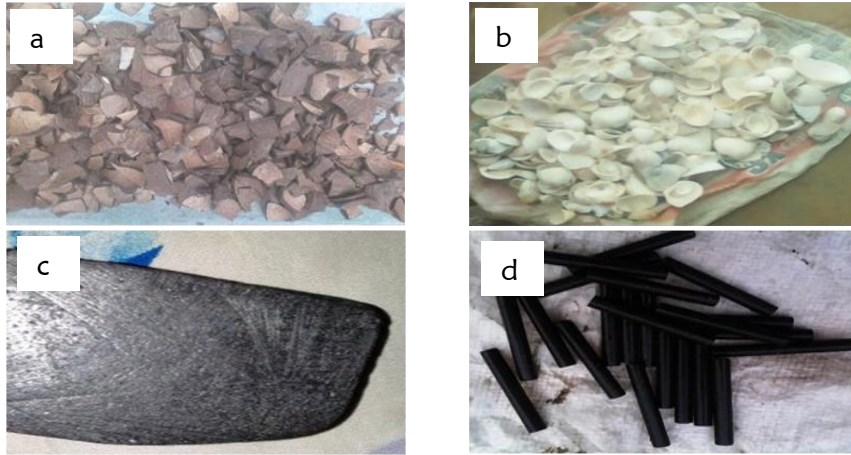


Figure 1: Reinforcement and friction modifier used (a) crushed coconut shells (b) seashells (c) commercial brake pad sample (d) extracted graphite rods

Table 1: Factor level of process parameters

Factor	MP (MPa)	MT (°C)	CT (min)	HTT (hr)
High	16	160	10	4
Low	12	120	6	2

Table 2: RSM-CCD experimental design matrix

Run	MT (°C)	MP (MPa)	CT (minute)	HTT (hour)
1	120	12	6	2
2	120	16	6	2
3	160	12	6	2
4	160	16	6	2
5	120	12	10	2
6	120	16	10	2
7	160	12	10	2
8	160	16	10	2
9	120	12	6	4
10	120	16	6	4
11	160	12	6	4
12	160	16	6	4
13	120	12	10	4
14	120	16	10	4
15	160	12	10	4
16	160	16	10	4
17	140	10	8	3
18	140	18	8	3
19	100	14	8	3
20	180	14	8	3
21	140	14	4	3
22	140	14	12	3
23	140	14	8	1
24	140	14	8	5
25	140	14	8	3
26	140	14	8	3
27	140	14	8	3

2.2.3 Samples Formulation and Production

Sample formulation was carried out using Rule of mixture (ROM) technique outlined in the work of Askeland (1985) utilising density (ρ) as a criteria while production was carried out on a compression moulding machine situated at Federal College of Chemical and Leather Technology (FCCLT), Samaru, Zaria (Polymer workshop) and was conducted using the procedure adopted by Abutu et al. (2019) which involved utilising varying process parameters shown in Table 2 with constant percentage composition as obtained from ROM. This procedure further involved pouring and mixing epoxy resin and hardener (catalyst) in the ratio of 2:1 in a separate container, followed by filling of mould cavity with total mixture of mixed binder and filler materials, then, the withdrawal of cured samples from the moulding machine, cooling of samples and removal of the cooled samples from the mould.

2.2.4 Sample Characterization

Tribological properties (wear rate and friction coefficient) of the developed and commercial samples were evaluated using a Tribometer (ANTON PAAR GmbH, CSM Instrument, Strasse 20, 8054 Graz-Austria) and experiment was conducted in accordance with ASTM G99 testing procedure using the test conditions presented in Table 3.

3. RESULTS AND DISCUSSION

3.1 Samples formulation and Production

The results of sample formulation indicate that samples should be produced using 52% reinforcement material (seashell or coconut shell), 35% binder (epoxy resin and hardener), 8% abrasive (Alumina) and 5% friction modifier (graphite).

3.1 Experimental Results

The average values of experimental result along with the individual signal-to noise (S/N) ratios for wear rate (Wr) and friction coefficient (μ) of the developed brake pad samples are shown in Table 4. S/N ratios of friction coefficient were calculated using larger-the better (Eqn. 1) while that of wear rate was calculated using smaller-the better (Eqn. 2).

$$S / N = -10 \log \frac{1}{N} \left(\sum_{N=1}^N \frac{1}{r^2} \right) \quad (1)$$

$$S / N = -10 \log \frac{1}{N} \left(\sum_{N=1}^N r^2 \right) \quad (2)$$

r = response value of given factor level combination,
N = number of factor level combination

From the experimental results presented in Table 4, it can be observed that the values of the friction coefficient varied from 0.477 to 0.788 for coconut shell-based samples and 0.43 to 0.61 for seashell-based samples. These values falls within the class F (0.35 – 0.45), G (0.45 – 0.55) and H (>0.55) type of brake pads recommended for use in automobile by Society of Automobile Engineers (SAE) and reported by Blau (2001) as well as Dagwa and Ibadode (2006). These results are in good agreement with the earlier work of Roubicek et al. (2008) who reported that friction coefficient that falls within the range of 0.30–0.70 is desirable in brake pads. Therefore, seashell and coconut shell-based brake pads are suitable for use in automobiles.

3.2 Multi-response optimisation

Multi-response optimisation of experimental results was carried using Grey relational analysis (GRA) technique. This technique was adopted to investigate the optimal process parameters that will produce the multi-response performance of the coconut and seashell-based brake pads. The procedure for GRA include using the values of S/N ratios shown in Table 3 to calculate the Grey relational grade (GRG) for friction coefficient and wear rate using larger the better (Eqn. 3) and smaller the better (Eqn. 4) attribute respectively. This is followed by the calculation of Grey relational coefficient (GRC) using Eqn. 5 and finally, the calculation using Grey relational grade using Eqn. 6.

$$\text{Larger-the-better attributes } (w_{ij}) = \frac{a_{ij} - \underline{a}_j}{a_i - \underline{a}_j} \quad (3)$$

$$\text{Smaller-the-better attributes } (w_{ij}) = \frac{\overline{a}_{ij} - a_{ij}}{a_j - \underline{a}_j} \quad (4)$$

Where, a_i = the performance value of alternative i attribute j and $\overline{a}_j = \max\{a_{ij}, i = 1, 2, \dots, x\}$. response and $\underline{a}_j = \min\{a_{ij}, i = 1, 2, \dots, x\}$.

$$\text{GRC, } (x_{0j}, x_{ij}) = \frac{D_{\min} + \lambda D_{\max}}{D_{ij} + \lambda D_{\max}} \quad (5)$$

$j = 1, 2, \dots, 27$ and $i = 1, 2, \dots, 27$, $D = x_{0j} - x_{ij}$, $\lambda = \min(D_{ij}, i = 1, 2, \dots, x; j = 1, 2, \dots, y)$, D_{max} and D_{min} = maximum and minimum GRG value for response and λ is the distinguishing coefficient, $\lambda \in (0, 1)$. The aim of distinguishing coefficient is to reduce or increase the range of the grey relational coefficient and 0.5 is the widely accepted value (Chin, 2003).

$$\text{Grade} = \frac{\sum \text{GRC}}{\text{No. of responses}} \tag{6}$$

The results of GRG, GRC and grades obtained from GRA are presented in Table 5 and the resulting factor effects of process parameters are shown in Table 6 while the main effect plots for coconut shell and seashell based brake pads are shown in Figure 2 and 3 respectively.

Table 3: Test parameters/conditions for tribology test

Ball Diameter	10 mm
Speed	10 cm/s
Load	7 N
Ball Material	Stainless Steel
Duration of Test	223 seconds
Humidity of environment	55 %
Temperature of environment	25 °C

Table 4: Experimental results and S/N ratios of developed samples

Run	MP (MPa)	MT (°C)	CT (min)	HT T (hr)	Coconut shell based				Seashell based			
					μ	μ (dB)	Wr (mg/m)	Wr (dB)	μ	μ (dB)	Wr (mg/m)	Wr (dB)
1	12	120	6	2	0.788	-2.07	0.2620	11.64	0.492	-6.16	0.3340	9.525
2	16	120	6	2	0.652	-3.72	1.0936	-0.78	0.447	-6.99	0.2685	11.42
3	12	160	6	2	0.686	-3.27	0.1310	17.66	0.548	-5.22	0.2358	12.55
4	16	160	6	2	0.688	-3.25	0.1703	15.38	0.529	-5.53	0.1834	14.73
5	12	120	10	2	0.685	-3.29	0.2030	13.85	0.556	-5.10	0.3995	7.970
6	16	120	10	2	0.601	-4.42	0.1244	18.10	0.525	-5.60	0.3536	9.029
7	12	160	10	2	0.601	-4.42	0.3995	7.970	0.58	-4.73	2.6130	-8.34
8	16	160	10	2	0.651	-3.73	0.0393	28.11	0.593	-4.54	0.2881	10.81
9	12	120	6	4	0.566	-4.94	0.6614	3.590	0.565	-4.96	0.2358	12.55
10	16	120	6	4	0.533	-5.47	0.2161	13.31	0.572	-4.85	0.1637	15.72
11	12	160	6	4	0.688	-3.25	0.0720	22.85	0.573	-4.84	0.2554	11.86
12	16	160	6	4	0.496	-6.09	0.0655	23.68	0.568	-4.91	0.1113	19.07
13	12	120	10	4	0.558	-5.07	0.6549	3.677	0.430	-7.33	0.2096	13.57
14	16	120	10	4	0.649	-3.76	0.7138	2.928	0.610	-4.29	0.2816	11.01
15	12	160	10	4	0.568	-4.91	0.6680	3.505	0.534	-5.45	0.1899	14.43
16	16	160	10	4	0.477	-6.43	0.7204	2.849	0.496	-6.09	0.7924	2.021
17	10	140	8	3	0.668	-3.50	0.1441	16.83	0.567	-4.93	0.1834	14.73
18	18	140	8	3	0.571	-4.87	0.0720	22.85	0.546	-5.26	0.4977	6.061
19	14	100	8	3	0.617	-4.19	0.6483	3.764	0.521	-5.66	0.1244	18.10
20	14	180	8	3	0.648	-3.77	0.0589	24.59	0.491	-6.18	0.2816	11.01
21	14	140	4	3	0.650	-3.74	0.0197	34.13	0.571	-4.87	0.2161	13.31
22	14	140	12	3	0.550	-5.19	0.0786	22.09	0.544	-5.29	2.1545	-6.67
23	14	140	8	1	0.720	-2.85	0.0066	43.68	0.509	-5.87	0.1048	19.59
24	14	140	8	5	0.614	-4.24	0.0720	22.85	0.519	-5.70	0.6549	3.677
25	14	140	8	3	0.503	-5.97	0.7138	2.928	0.498	-6.06	0.2227	13.05
26	14	140	8	3	0.528	-5.55	0.8382	1.533	0.511	-5.83	0.2096	13.57
27	14	140	8	3	0.521	-5.66	0.7924	2.021	0.497	-6.07	0.2030	13.85

Table 5: Results of Grey relational analysis (GRA)

Senario	GRG				GRC				Grade	
	Coconut shell based		Seashell based		Coconut shell based		Seashell based		Coconut shell based	Seashell based
	μ	W_r	μ	W_r	μ	W_r	μ	W_r		
X _o	1.00	1.00	1.00	1.00	-	-	-	-	-	-
1	1.00	0.72	0.39	0.36	1.00	0.64	0.45	0.44	0.82	0.45
2	0.62	1.00	0.11	0.29	0.57	1.00	0.36	0.41	0.79	0.39
3	0.72	0.59	0.69	0.25	0.64	0.55	0.62	0.40	0.60	0.51
4	0.73	0.64	0.59	0.17	0.65	0.58	0.55	0.38	0.62	0.47
5	0.72	0.67	0.73	0.42	0.64	0.60	0.65	0.46	0.62	0.56
6	0.46	0.58	0.57	0.38	0.48	0.54	0.54	0.45	0.51	0.50
7	0.46	0.80	0.86	1.00	0.48	0.72	0.78	1.00	0.60	0.89
8	0.62	0.35	0.92	0.31	0.57	0.43	0.86	0.42	0.50	0.64
9	0.34	0.90	0.78	0.25	0.43	0.84	0.70	0.40	0.64	0.55
10	0.22	0.68	0.82	0.14	0.39	0.61	0.73	0.37	0.50	0.55
11	0.73	0.47	0.82	0.28	0.65	0.48	0.74	0.41	0.57	0.58
12	0.08	0.45	0.80	0.02	0.35	0.48	0.71	0.34	0.42	0.53
13	0.31	0.90	0.00	0.22	0.42	0.83	0.33	0.39	0.63	0.36
14	0.61	0.92	1.00	0.31	0.56	0.86	1.00	0.42	0.71	0.71
15	0.35	0.90	0.62	0.18	0.43	0.84	0.57	0.38	0.64	0.48
16	0.00	0.92	0.41	0.63	0.33	0.86	0.46	0.57	0.60	0.52
17	0.67	0.60	0.79	0.17	0.60	0.56	0.71	0.38	0.58	0.55
18	0.36	0.47	0.68	0.48	0.44	0.48	0.61	0.49	0.46	0.55
19	0.51	0.90	0.55	0.05	0.51	0.83	0.53	0.35	0.67	0.44
20	0.61	0.43	0.38	0.31	0.56	0.47	0.45	0.42	0.52	0.44
21	0.62	0.21	0.81	0.23	0.57	0.39	0.73	0.39	0.48	0.56
22	0.28	0.49	0.67	0.94	0.41	0.49	0.60	0.89	0.45	0.75
23	0.82	0.00	0.48	0.00	0.74	0.33	0.49	0.33	0.54	0.41
24	0.50	0.47	0.54	0.57	0.50	0.48	0.52	0.54	0.49	0.53
25	0.11	0.92	0.42	0.23	0.36	0.86	0.46	0.40	0.61	0.43
26	0.20	0.95	0.49	0.22	0.39	0.91	0.50	0.39	0.65	0.45
27	0.18	0.94	0.41	0.21	0.38	0.89	0.46	0.39	0.64	0.43

Table 6: Resulting factor effects of process parameters

Level	Coconut shell based				Seashell based			
	MP (MPa)	MT (°C)	CT (min)	HTT (hr)	MP (MPa)	MT (°C)	CT (min)	HTT (hr)
1	0.5800	0.6700	0.4800	0.5400	0.5500	0.4400	0.5600	0.4100
2	0.6400	0.6565	0.6200	0.6325	0.5475	0.5088	0.5038	0.5513
3	0.5405	0.5190	0.5562	0.5419	0.5095	0.5410	0.4795	0.5324
4	0.5812	0.5687	0.6012	0.5887	0.5388	0.5775	0.5825	0.5350
5	0.4600	0.5200	0.4500	0.4900	0.5500	0.4400	0.7500	0.5300

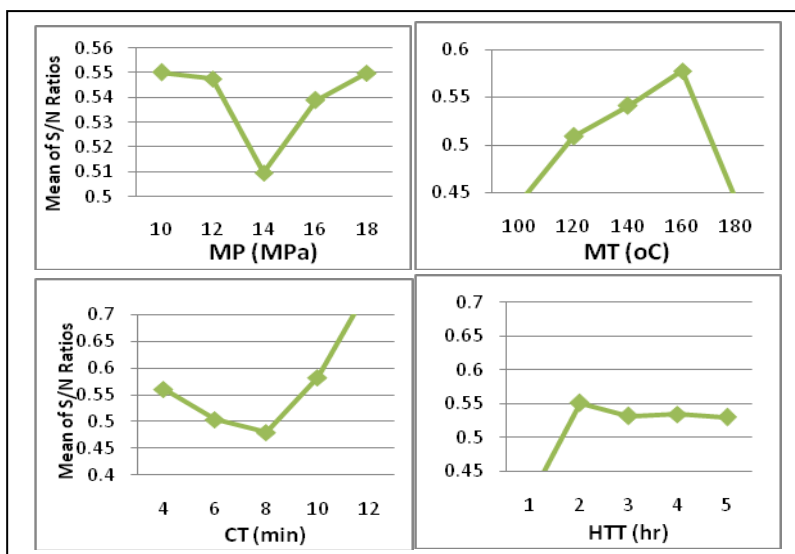


Figure 2: Main effect plots for coconut shell based sample

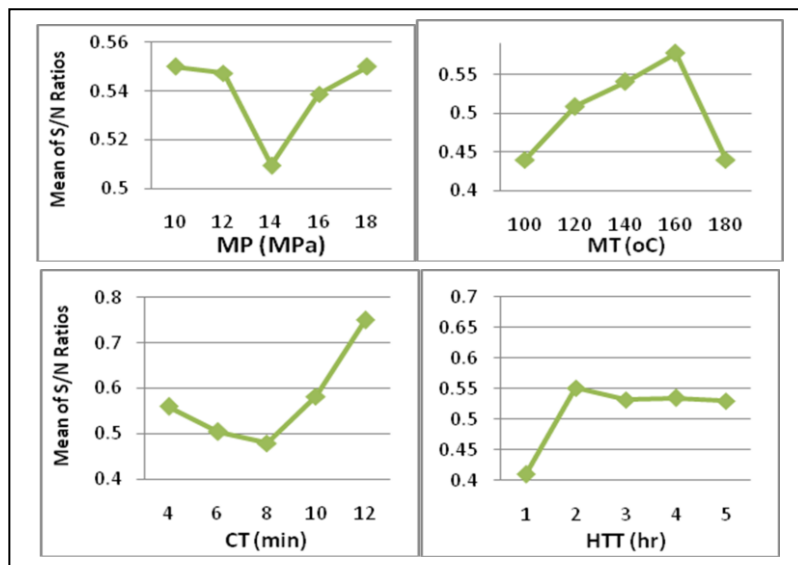


Figure 3: Main effect plots for seashell based sample

The main effect plots shown in Figure 2 indicates that optimum multi-response performance of the developed coconut shell based brake pad samples can be achieved using MP, MT and CT and HTT of 12MPa, 100°C, 6mins and 2hrs respectively while Figure 3 revealed that optimum multi-response performance of the developed seashell based brake pads can be achieved using MP, MT and CT and HTT of 10MPa, 160 °C, 12mins and 2hrs respectively. Any change in these optimal parameters may lead to poor bonding between the resin and its constituent fillers (Abutu et al., 2018).

3.3 Production of optimized samples

The optimised brake pad samples were produced using standard compression moulding process described in the earlier section. Production of the coconut shell-based sample was done using MP, MT and CT and HTT of 12MPa, 100 °C, 6mins and 2hrs respectively while the seashell-based sample was produced using MP, MT, CT and HTT of 10MPa, 160 °C, 12mins and 2hrs respectively as obtained from GRA. The percentage composition of the brake pad samples remains constant throughout the moulding process.

3.4 Characterisation of optimised and commercial samples

In order to study and compare the tribological properties of the optimised and commercial (control) brake pads, samples were characterised using the testing methods discussed in the previous section. The results of experimental findings compared with the control are summarised in Table 7. The results presented in Table 7 revealed that friction coefficient and wear rate of the commercial brake pads are 0.634 and 0.04184 mg/m respectively while that of coconut shell and seashell reinforced samples are 0.614 and 0.03156 mg/m as well as 0.542 and 0.07252 mg/m respectively. These results indicate that the coconut shell-based brake pads possesses lower wear rate compared to commercial and seashell-based samples and also falls within the category of class H ($\mu > 0.55$) type of brake pads. Thus, is recommended for use in heavy duty automobile by the Society of Automobile Engineers (SAE) as reported in the work of Blau (2001) and Dagwa and Ibadode (2006). Also, the samples reinforced with seashell falls within the class G (μ : 0.45–0.55) type of brake pads and thus suitable for use in light weight automobile. The morphology of the wear track section of the optimized and commercial sample is shown in Figure 4(a-c). The part labeled 'A' represent the wear track section of the test specimen.

As shown in Figure 4a, it can be observed that the coconut shell-based sample has the least track section with an area of 154597.2 μm^2 . This is followed by commercial sample (Figure 4b) with track sectional area of 204896.1 μm^2 and finally the seashell-based sample (Figure 4c) which showed the widest track section (355165.0 μm^2). These differences in the wear track area may be attributed to the variation in the hardness and flexural strength of the samples. This is in agreement with the earlier work of Zum-Gahr (1987) who reported that wear rate of materials are strongly dependent on the size, hardness, shape and flexibility of the abrasive particles as a results harder materials tend to have lower wear track sectional area.

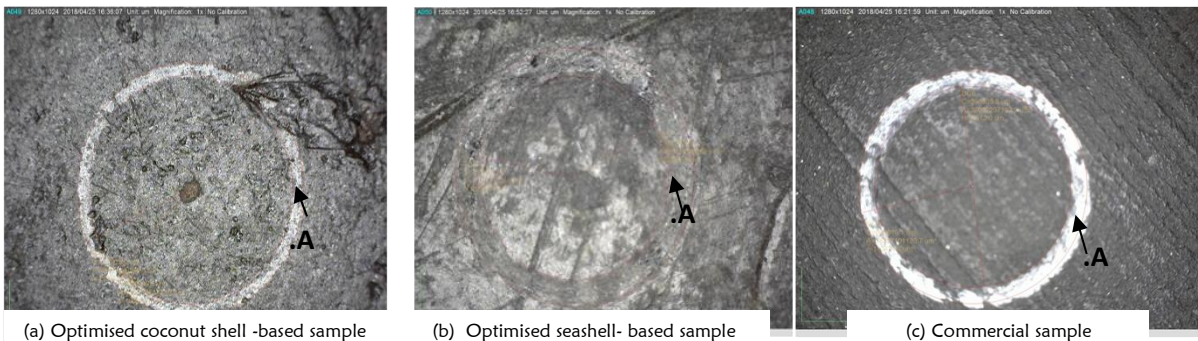


Figure 4: Morphology of wear track section on tribometer

Table 7: Tribological properties of optimised and control sample

S/N	Properties	Commercial product (X)	Coconut shell based (C)	Seashell based (S)
1.	Wear rate (mg/m)	0.04184	0.0315	0.0725
2.	Coefficient of friction	0.634	0.614	0.525

Table 8: ANOVA for Friction Coefficient

Factor	Coconut shell based					Seashell based				
	DOF	SS	MS	F	P (%)	DOF	SS	MS	F	P (%)
MP (MPa)	4	0.0297	0.0074	9.715	19.388	4	0.0116	0.0029	16.422	24.440
MT (°C)	4	0.0177	0.0044	5.787	11.549	4	0.0142	0.0035	20.037	29.819
CT (min)	4	0.0252	0.0063	8.249	16.464	4	0.0117	0.0029	16.586	24.684
HTT (hr)	4	0.0729	0.0182	23.855	47.609	4	0.0082	0.0021	11.648	17.335
Error	10	0.0076	0.0008		4.989	10	0.0018	0.0002		3.721
Total	26	0.1532	0.0059		100.0	26	0.0476	0.0018		100.0

Table 9: ANOVA for wear rate

Factor	Coconut shell based					Seashell based				
	DOF	SS	MS	F	P (%)	DOF	SS	MS	F	P (%)
MP (MPa)	4	0.492	0.123	12.365	17.65	4	1.332	0.333	15.608	14.93
MT (°C)	4	0.916	0.229	23.046	32.9	4	1.518	0.38	17.788	17.01
CT (min)	4	0.603	0.151	15.169	21.65	4	4.93	1.233	57.755	55.23
HTT (hr)	4	0.675	0.169	16.975	24.23	4	0.932	0.233	10.917	10.44
Error	10	0.099	0.01		3.569	10	0.213	0.021		2.391
Total	26	2.786	0.107		100	26	8.926	0.343		100

3.5 Analysis of Variance (ANOVA)

ANOVA was conducted on the experimental responses in order to study the significant effects and percentage contribution of individual process parameters. This analysis was carried out using confidence level of 99 % and significance level of 1 %. ANOVA table shown in Table 8-9 consist of degree of freedom (DOF), sum of square (SS), mean square (MS), f-value and percentage contribution (P). The ANOVA for the coefficient of friction of coconut shell-based composite shown in Table 9 indicates that HTT with percentage contribution of 47.609% provides the greatest impact on the friction coefficient of the friction materials. This is followed by MP (19.388%) and CT (16.464%), finally, the least significance, MT with percentage contribution of 11.549%. Also, the ANOVA for the coefficient of friction of seashell-based composite shown in Table 9 indicates that MT with percentage contribution of 29.819% provides the greatest influence on the friction coefficient of the friction materials, followed by CT (24.684%) and MP (24.440%) and HTT (17.335%). In addition, the ANOVA for wear rate of coconut shell-based composite shown in Table 10 revealed that MT with percentage contribution of 32.896% provides the greatest impact on the wear rate of the friction materials, followed by HTT (24.231%), CT (21.653%) and finally, MP (17.651%). Also, the ANOVA for the wear rate of seashell-based composite shown in Table 10 showed that CT with percentage contribution of 55.232% provides the greatest impact on the wear rate of the friction materials. This is followed by MT (17.011%), MP (14.93%) and finally, MP (10.442%). Also, the percentage error obtained for this analysis were less than 5% which indicate that the experimental processes were conducted with minimum influence of noise (Lawal et al., 2016; Zaharudin et al., 2011).

3.2.4 Empirical regression analysis

Empirical regression equation was obtained using the experimental data with the aim of predicting the value of the investigated responses. The regression equations along with correlation coefficients (Rsq) for coconut shell and seashell based sample are shown in Equ. 7-11.

For coconut shell reinforced composite,

$$\mu = 1.128 - 0.01223 \text{ MP} - 0.000240 \text{ MT} - 0.01056 \text{ CT} - 0.0429 \text{ HTT} \quad (7)$$

$$\text{R-sq} = 85.52\% \text{ and R-sq (adj)} = 75.62\%$$

$$\text{Wear rate} = 0.4095 - 0.0011 \text{ MP} - 0.00592 \text{ MT} + 0.0202 \text{ CT} + 0.0607 \text{ HTT} \quad (8)$$

$$\text{R-sq} = 66.77\% \text{ and R-sq (adj)} = 51.63\%$$

For seashell reinforced composite,

$$\mu = 0.471 + 0.00042 \text{ MP} + 0.000342 \text{ MT} - 0.00050 \text{ CT} + 0.00408 \text{ HTT} \quad (9)$$

$$\text{R-sq} = 53.28\% \text{ and R-sq (adj)} = 50.11\%$$

$$\text{Wear rate} = 0.6536 - 0.0292 \text{ MP} + 0.00570 \text{ MT} + 0.0303 \text{ CT} - 0.082 \text{ HTT} \quad (10)$$

$$\text{R-sq} = 76.47\% \text{ and R-sq (adj)} = 63.10\%$$

As shown in Eqn. 7-10, it can be observed that the value of R-sq_{adj} (correlation coefficient) falls below the recommended of 80 % as a result of noise which could occur from experimental uncertainty (Asuero et al., 2006).

4. CONCLUSIONS

This study was carried out with the aim of investigating the tribological properties of natural based (coconut shell and seashell) friction materials as a substitute for asbestos in the production of brake pads using response surface methodology and multi-response optimisation technique (GRA). From the results obtained, the following conclusion can be drawn;

- i. Changes in experimental factors affects the tribological properties of the developed friction materials as all the samples produced with varying parameters gave different performance characteristics.
- ii. Multi-response optimization results indicate that optimum multi-response performance of the developed coconut shell based brake pad can be achieved using MP, MT and CT and HTT of 12MPa, 100 °C, 6mins and 2hrs respectively while optimum multi-response performance of the developed seashell based brake pad can be achieved using MP, MT and CT and HTT of 10MPa, 160 °C, 12mins and 2hrs respectively,
- iii. The optimized coconut shell based brake pads falls within the category of class H ($\mu > 0.55$) type of brake pads while seashell based sample falls within the class G (: 0.45–0.55) type of brake pads as a result can be recommended for use in heavy and light duty automobile as specified by the Society of Automobile Engineers (SAE) standards.
- iv. The wear on seashell based and commercial sample showed a wider track section compared with that coconut shell which has a lower wear track section indicating a better wear resistance and friction coefficient.
- v. Finally, the percentage errors obtained for ANOVA were less than 5% which indicate that the experimental processes were conducted with minimum influence of noise.

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LIST OF ABBREVIATIONS

- DOE=Design of experiment
 MP=Moulding pressure
 MT=Moulding temperature
 CT=Curing time
 HTT=Heat treatment time
 μ =Coefficient of friction
 W_r =Wear rate
 GRA=Grey relational analysis
 GRG=Grey relational generation
 GRC=Grey relational coefficient
 S/N=Signal to-noise ratio
 D_{max} = maximum GRG
 D_{min} =maximum GRG
 $\hat{\lambda}$ =Distinguishing coefficient
 ρ =density
 Rsq=Correlation coefficient
 ANOVA=Analysis of variance
 ROM=Rule of mixture.