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On the Morphological and Tribological Characterization of Green Automotive Brake Pads Developed from Waste Thais Coronata Seashells

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Abstract

In this study, morphological and tribological analyses (optical microscopy, scanning electron microscopy as well as friction and wear tests) were used to investigate the properties of newly developed, non-toxic organic brakepads. The green automotive brake pads were developed from wastes Thais Coronata seashell reinforcement materials. The energy-dispersive X-ray spectrometer (EDX) was used to determine the chemical composition of the developed samples. Similar analyses were conducted on two commercial (control) samples for comparative purposes. EDX analysis showed the presence of toxic heavy metals (7 - 40%Sb, 6.44 -12.88%Ti) in the commercial (control) samples, unlike the green brake pad samples. The friction coefficient of the developed sample was obtained to be 0.584 while the commercial samples were 0.106 and 0.196. The tribological results show that the developed samples can maintain a superior frictional performance compared to the control samples and can be installed on heavy-duty automobiles. Optical microscopy results for samples of different particle sizes show that the wear depth increases with increase in the particle size of the reinforcement materials. Also, the presence of wear debris, friction films, and plateaus was more evident on the deformed surfaces of the commercial samples than on the deformed surfaces of the developed brake pad samples.

Keywords: brake pads, eco-friendly, Thais Coronata shells, particle size, microstructure, SEM, mechanical and tribological properties.

1. Introduction

Modern braking systems have been developed to compensate for increased engine power, safety, and shorter stopping distances. This accounts for the high rate level of vehicular emissions and environmental pollution [1]. Brake wear is the highest contributor of non-exhaust vehicular emissions, particularly in urban areas with heavy traffic density and braking frequency [2, 3]. These emissions have adverse health and environmental effects, especially when the brake pads are produced from carcinogenic reinforcements and non-biodegradable metals.

To address the problem of pollution from automobile brakes, there is a growing need to employ the use of improved eco-friendly organic materials in the development of the pads. A detailed review of green ecological constituents for the formulation of friction materials and the influence of these constituents on the tribological and mechanical properties of the final brake pad composites was given by Ekpruke et al. [4]and Confiengo and Faga [5]. The studies show that many organic, eco-friendly materials have been proposed to replace hazardous reinforcement materials used in the manufacture of conventional friction components.

Different studies on the development and morphological characterization of organic automotive brake pads exist. Bachchhav and Hendre [6] investigated the wear behavior of asbestos-free automotive brake pads developed from metallic-based reinforcement materials. Scanning electron microscopy (SEM) results showed that heat can significantly affect the wear dynamics of the products as increased temperature led to an exponential increase in the wear rate of the samples. However, EDX analysis showed the presence of antimony (SB) in the product. Also, the wear behavior of aluminum-reinforced matrix composites was investigated by Dixit and Khan [7]. The study showed that varying graphite particle sizes in an oil lubricant can be used to control the wear behavior of the reinforcement aluminum composites. Erickson and Jacobson [8], investigated the mechanical properties and tribological compositions of organic brake pad surfaces using high-resolution SEM, and energy-dispersive X-ray fluorescence spectroscopy. In another study, [9]they found out that the contact mechanism of a brake pad sliding against a rotor (grey cast iron) is quite different from other tribological systems. When a brake pad is worn against the rotor, the larger contact plateaus rises a few micrometers above the rest of the surface. Babu and Solomon [10] presented several case studies on the characterization of five different brake pad formulations using the scanning electron

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microscope (SEM). They used the features in SEM to explain the change in friction and wear mechanism of the composites. The presence of loose particles, friction films, primary and secondary plateaus was identified on the worn surfaces. Other sources that employed the SEM in investigating surface deformation and wear mechanisms of newly developed organic fiber-reinforced composites can be found in refs. [11-17].

Thais Coronata shells are organic materials primarily composed of calcium carbonates with significant mechanical properties in terms of weight, stiffness, toughness, and strength [18, 19]. In a study by Ekpruke et al. [20], the process parameters involved in the development of eco-friendly automotive brake pads from wastes Thais Coronata seashell reinforcement materials were investigated using the central composite face-centered (CCF) design. The brake pad composites were developed by compression molding based on 3-levels of 3- factors: curing time, curing temperature, and molding pressure. The determined optimal process parameters of the developed brake pads were a molding pressure of 13MPa, a curing temperature of 165°C, and a curing time of3 hours. The mechanical and tribological properties of the developed samples were characterized and found to possess superior quality compared to the two commercial samples studied as control.

In this present study, the microstructural morphology of the green organic brake pads developed from Thais Coronata shells(see ref. [20]) was investigated using the Optical Microscope (OM) and the Scanning Electron Microscope (SEM). OM and SEM analyses were carried out before and after deformations of the brake pad surfaces. Also, the chemical composition of the developed samples was analyzed to understand their environmental and health friendliness and the results were compared with commercial brake pad samples. The effect of particle size on the morphology of the sample was also studied on a microscopic level.

2. Materials and Methods

2.1. Materials

The main materials used for the development of the brake pads are as follows: Thais Coronata shells, sourced locally and used as reinforcement; Epoxy resin *(99% purity; Dachy polymer, Taiwan)* was used as a binder. Calcium carbonate *(99.5% purity; Skyline chemical, USA)* was used as fillers. Iron and copper filings were used as abrasives and thermal conductivity enhancers respectively. Methyl ethyl ketone peroxide (MEKP) *(98% purity; Akzonobel, China)* was added as a catalyst, and carbon black *(99.9% purity; Loba Chemie PVT Ltd., India)* was used as a friction modifier. The percentage composition by weight of the various materials used in the formulation is shown in Table 1.

2.2. Method

The brakepad was developed followingthe method employed byOssia et al. [21] as shown in Figure 1. A similar method has also been employed in the following literature [22-25].

Table 1. Percentage composition by weight of various materials used in the formulation

S/N	Materials	Composition (%wt)
1	Thais Coronata Shells (reinforcement)	54.7
2	Epoxy resin & Hardener (binder)	30
3	Calcium carbonate (filler)	9.7
4	Methyl ethyl ketone peroxide (catalyst)	2.8
5	Fe filings (abrasive)	1.0
6	Copper filings (thermal conductivity enhancer)	1.3
7	Carbon black (friction modifier)	0.5

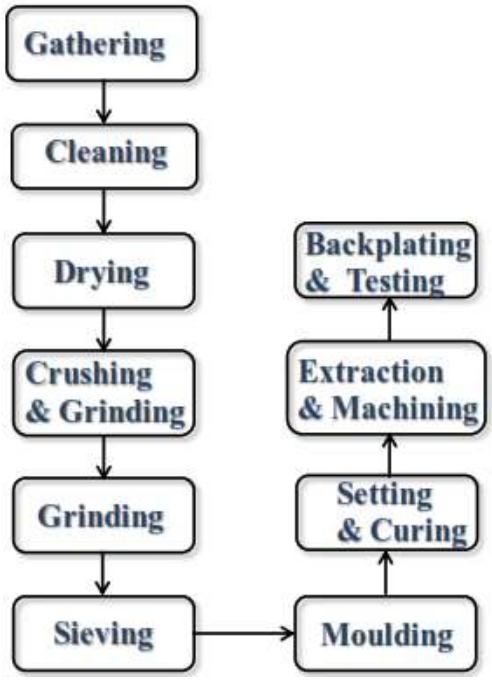


Figure 1. Flowchart of brake pad development process (Ossia et al. [21])

2.2.1. Gathering / Cleaning/ Drying / Crushing / Grinding/ Sieving

The Thais Coronata shells were bought from the local market. The shells were washed thoroughly to remove dirt and sundried for one week to remove moisture. The dried shells were crushed into smaller pieces using a pestle and mortar and finally pulverized using the Denver laboratory ball milling machine. The ground shells were sieved into different particle sizes of 60µm, 90µm, 125µm, 250µm, 500µm, and 850µm with the aid of the Sieve shaking machine and sieve stack.

2.2.1.1. Molding/Extraction/Setting/Curing

A weighted percentage of the Thais Coronata shells, $CaCO_3$, carbon black, Fe, and Cu filings as specified in Table 1 was taken and mixed thoroughly in a neat bowl (Mix A). A mixture of the resin and hardener (taken in ratio 2:1) together with the MEKP was also obtained in a separate bowl (Mix B). Mix B was poured into Mix A and stirred thoroughly to obtain a homogenous mixture (Figure 2c). The homogeneous slurry was poured into a circular mold for setting. The set samples were left in the mold under a load of 13MPa for 24 hours. The developed samples were then extracted and subjected to heat treatment in an electric oven for 3 hours of curing time at a temperature of 165ºC (Figure 2d).

2.2.2. Machining/Backplating

The developed brake pad sample was machined to shape using a carrot stone and a cutting disk with a spindle speed of 288 rpmand the milling machine *(Model: HURE SA-PU771, France)* and backplated (Figure 2e).

2.3. Mechanical Characterization of developed samples

2.3.1. Energy Dispersive X-Ray (EDX)Spectroscopy

The surfaces of the samples were thoroughly cleaned with ethanol *(99% purity, Sigma, china)* solvent to remove dirt and other adhering substances. The EDX spectroscopy test was carried out using Oxford Instrument *(X-Met 7000 XRF Spectrometer UK)*.

2.3.2. Friction and Wear Tests

Friction and wear tests were conducted on a pin-on-disk tribometer *(CSM Instrument, Austria)* using the ASTM D99-05 test method[26]. The sample weight before and after the friction test was taken and the wear rate was calculated as a ratio of weight loss over the sliding distance.

2.3.3. Optical Microscopy

Images of the developed samples were taken by an optical microscope (*BXFM-S, Olympus Corporation, Japan*). This was done before and after the friction and wear test to obtain a magnified image of the worn and unworn surfaces of the samples.

2.3.4. Scanning Electron Microscopy

The Zeiss SEM (*Zeiss Ultra Plus 55 FECSEM, Zeiss, Oberkochen, Germany*) was used to carry out a morphological study on the samples. Before the examination, the samples were prepared by cutting them with a bench vice and hacksaw followed by gluing them on the sample holder and coating with carbon using Carbon Coater.

3. Results and Discussion

3.1. Energy Dispersive X-Ray Spectroscopy

The EDX spectroscopy tests result for the developed brake pad and two commercial brake pad samples used as control are shown in Figure 3. Prolonged exposure to wear dust from the two commercial brake pads can cause serious health and environmental problems [27-29]. This is because of the presence of toxic heavy metals like antimony Sb (7%-40%), Vanadium V (1.69%), Tin Sn (86), Selenium Se (0.11%), and Molybdenum Mo (0.5%), detected in the commercial brake pads (Figures 3a, b).Studies have also reported these metals to be non-essential nutrients since they have no biological significance to the human body [30, 31]. However, heavy metals like Iron Fe (53.73%), Copper Cu (20.33%), and Cobalt Co (1.58%) detected in the developed brake pad (Figure 3c) are classified as essential nutrients since they are beneficial and relatively harmless to humans [31]. The Cu and Fe found in Figure 3c were deliberately added in the development process to improve the thermal conductivity of the product while Palladium Pd (8.80%), Gold Au (7.43%), Zinc Zn (4.78%) and Zirconium Zr (4.36%) have low levels of toxicity, and are poorly assimilated by the body when consumed.

3.2. Friction coefficient

The coefficient of friction histories of the commercial and developed samples are shown in Figure 4. The friction history has two stages; transient and steady-state. The transient phase develops from the minimum friction value to the beginning of the steady-state phase. The steady-state phase is characterized by an almost constant value of friction till the end of the sliding contact. The mean coefficient of friction for the two commercial samples are 0.106 (Figure 4a) and 0.196 (Figure 4b) respectively. The developed sample attained a stable state faster than the commercial sampleswith an average friction coefficient of 0.584 (Figure 4c). Based on the standards of identifying and classifying friction coefficients of brake linings by the Automotive Society of Engineers, friction coefficients of brake pads can be within the range of0.15 to over 0.55 (SAE J866a [32] and Blau [33]) but to achieve a good braking efficiency, a high and stable coefficient of friction is desired (Calderón et al.[34]). This shows that the developed brake pads can maintain a superior frictional grip than the commercial samples.

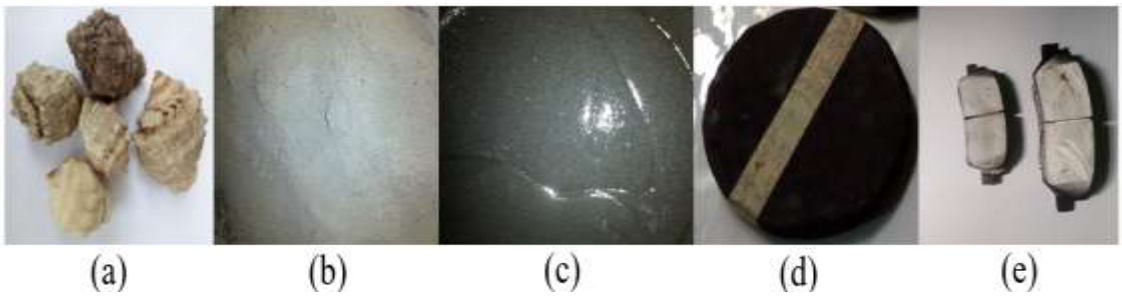
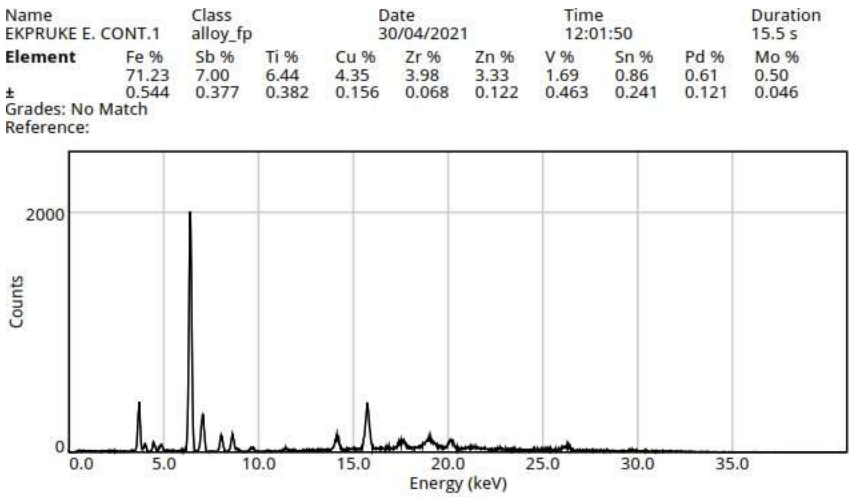
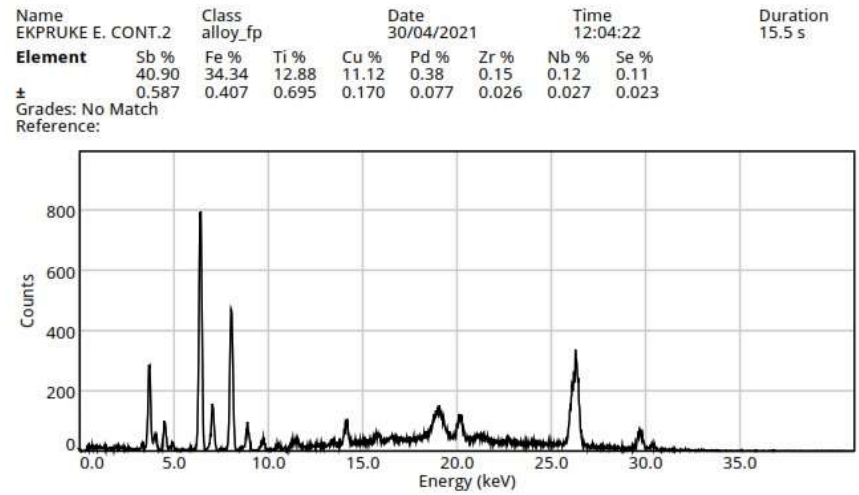


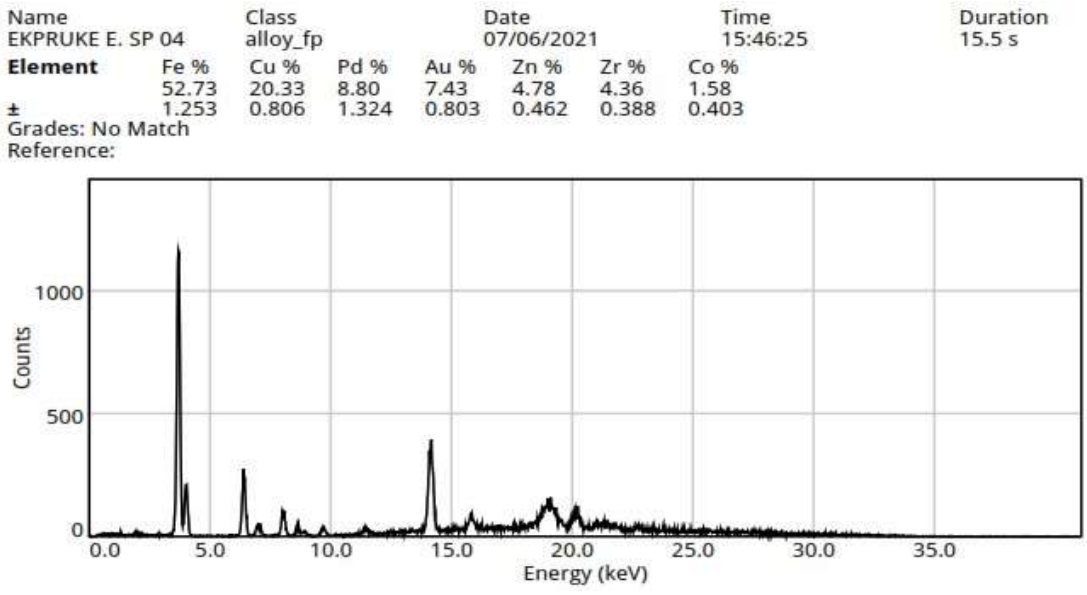
Figure 2. Brakepad development process: (a) uncrushed Thais Coronata shells, (b) crushed Thais Coronata shells, (c) homogenous mixture, (d) heat-treated sample, (e) machined finished product



(a)

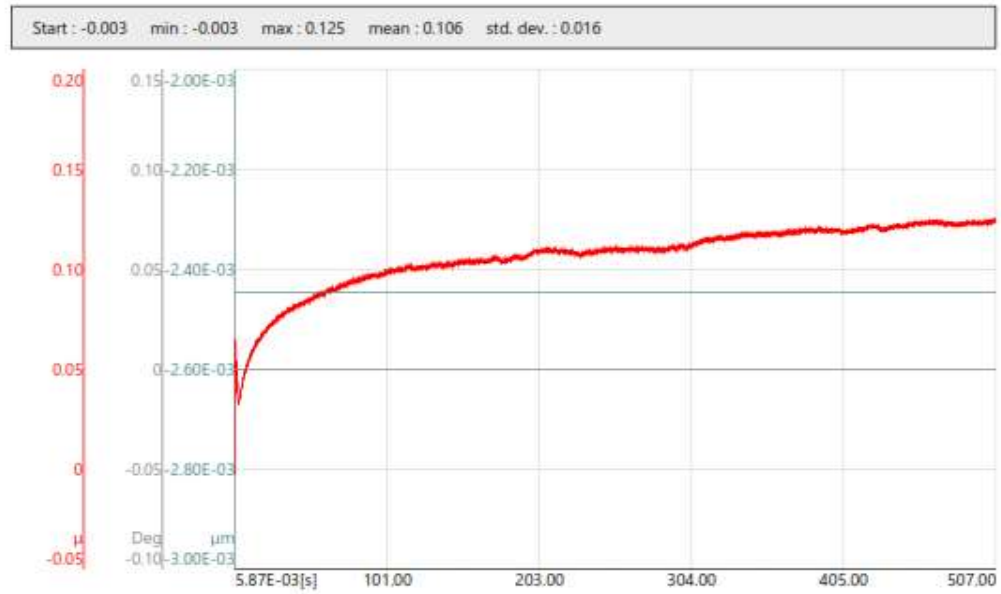


(b)

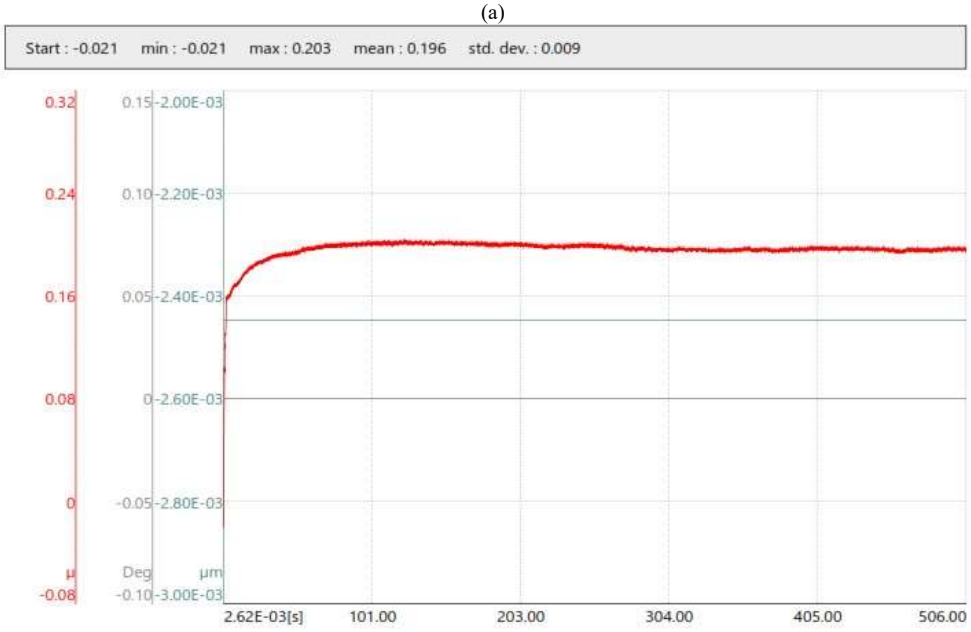


(c)

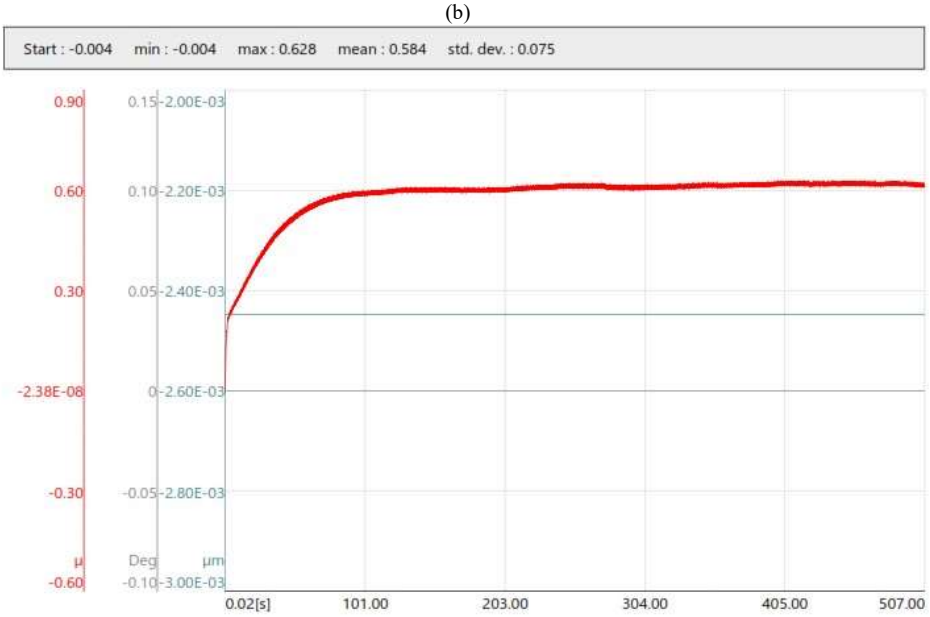
Figure 3. EDX spectroscopy results for (a) - (b) commercial brake pad samples and (c) developed brake pad sample



(a)



(b)



(c)

Figure 4. Coefficient of friction history of (a) – (b) commercial brake pad samples and (c) developed brake pad sample

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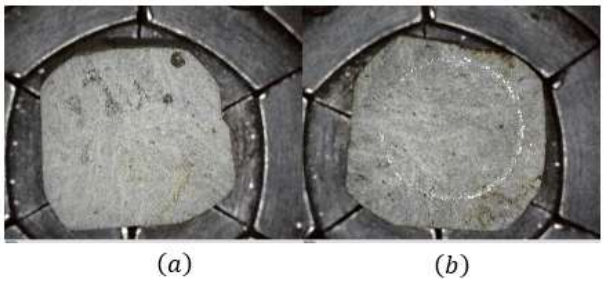
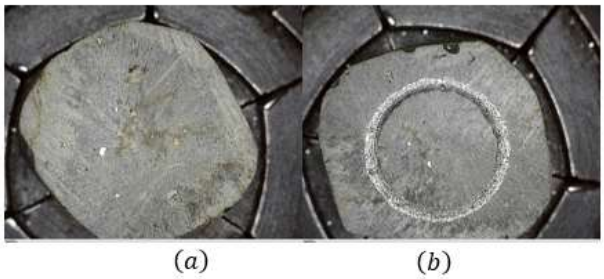
3.3. Effect of particle size distribution on the morphology of developed brake pads

3.3.1. Optical Microscopy

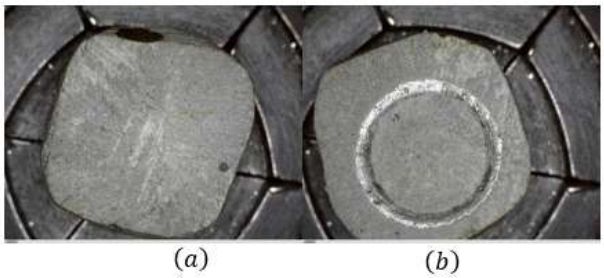
Optical micrographs of tribological interfaces of the developed brake pad samples (a) before wear and (b) after wear, are shown in Figures 5(i) – (vii) for the different particle sizes. The crack propulsion left on the surface of the samples after the friction test was observed to be more pronounced on the surfaces of the more coarse samples. The wear rate of the 53µm, 63µm, and 90µm particle size brake pads are 0.008mg/m, 0.024mg/m, and 0.038mg/m respectively while the wear rate of the 125µm, 250µm, 500µm, and 850µm particle size brake pads are 0.044mg/m, 0.188mg/m, 0.232mg/m and 0.4mg/m respectively. This shows that the wear rate increases as the particle size increases. This was clearly shown by the optical micrographs as the wear depth can be seen to increase with respect to the increase in particle size (Figures 5(i) – (vii)). This phenomenon is due to the uneven distribution of material ingredients on the surfaces of the larger particles-sized samples due to the coarseness of the debonded particles creating more voids on the surfaces.



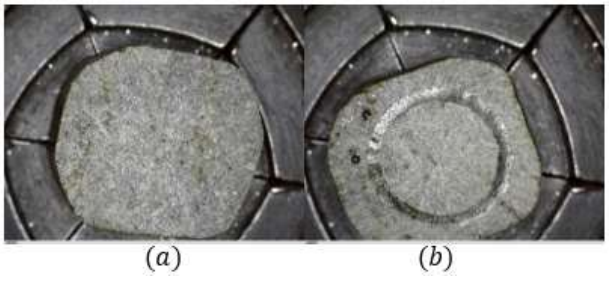
(i)Developed brake pad sample with 53µm particle size



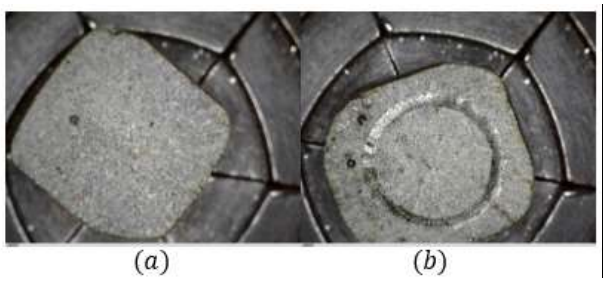
(ii)Developed brake pad sample with 63µm particle size



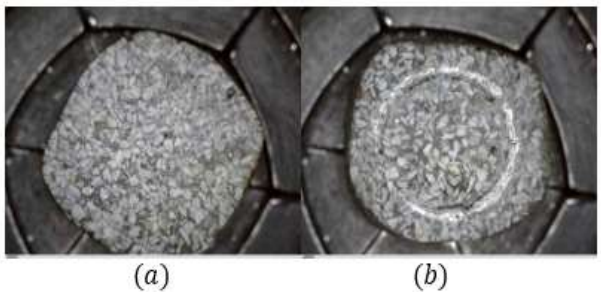
(iii)Developed brake pad sample with 90µm particle size



(iv)Developed brake pad sample with 125µm particle size



(v)Developed brake pad sample with 250µm particle size



(vi)Developed brake pad sample with 500µm particle size

(vii) Developed brake pad sample with 850µm particle size

Figure 5. Optical micrographs (a) before wear and (b) after wear of developed brakepad samples

3.3.2. Scanning Electron Microscopy

SEM micrographs at different magnifications show the enlarged shapes, surface asperities and microstructural voids on the surface of the developed brake pad samples. SEM Microstructures of 53µm and 850µm particle size brake pad samples are shown at magnifications of 500x, 1000x and 1500x (Figures 6 and 7). Figure 6 shows that in the 53µm-sample, the Thais Coronata particles and other ingredients employed in the development are well distributed with no obvious agglomeration of materials. Figure 7on the other hand, shows an even distribution of material ingredients with obvious separation of particle materials. The debonded particles form aggregates with obvious pores and voids in between. The 53µm-composite has a finer materials distribution than the 850µm-composite. Hence, particle size has a significant effect on the mechanical and tribological properties of the brake pads.

This finding corroborates the results obtained in the work of Friedrich [35] and Xing and Li [36] which shows that finer particles contribute to improved properties of the composite under sliding wear conditions than the more coarse particles. However, [37-39] reported the opposite.

The commercial sample (control 1) investigated shows high concentrations of small voids and intergranular pores (Figure 8a) which is more evident at a high magnification of 1500x (Figure 8b). Also, patches of inhomogeneous polymeric ingredients were seen unlike what was observed in the 53µm-sample (Figure 6). A larger percentage of uneven materials distribution, voids, and crack propagation was observed in the control sample 2(figure 9). This show that the wear properties of the developed brake pads are more efficient than that of the commercial brake pad investigated.

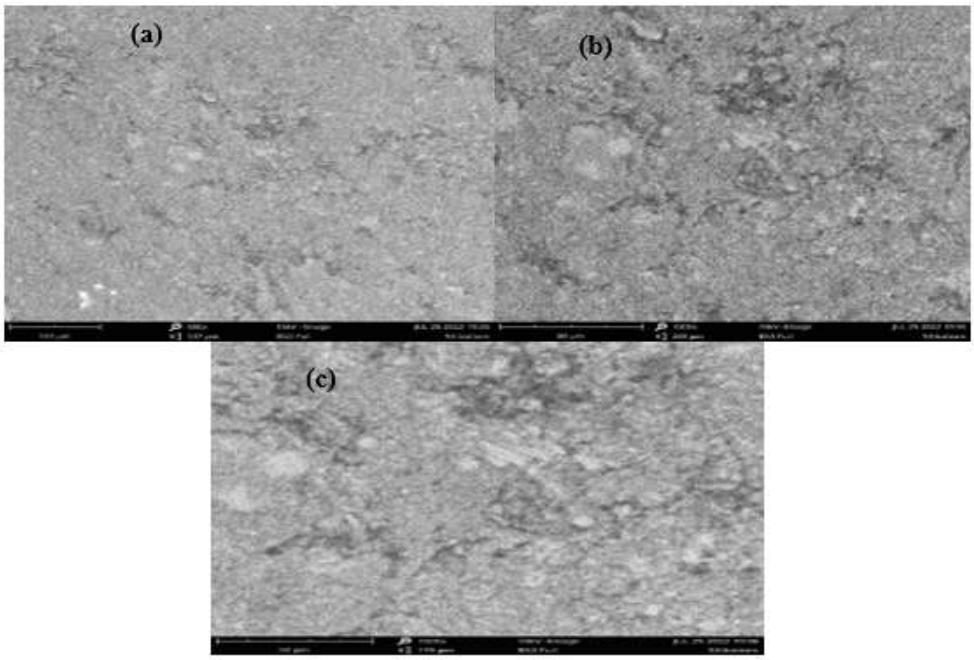


Figure 6. SEM micrographs of the developed brake pad sample with 53µm particle size at magnifications of (a) 500x, (b) 100x, and (c) 1500x

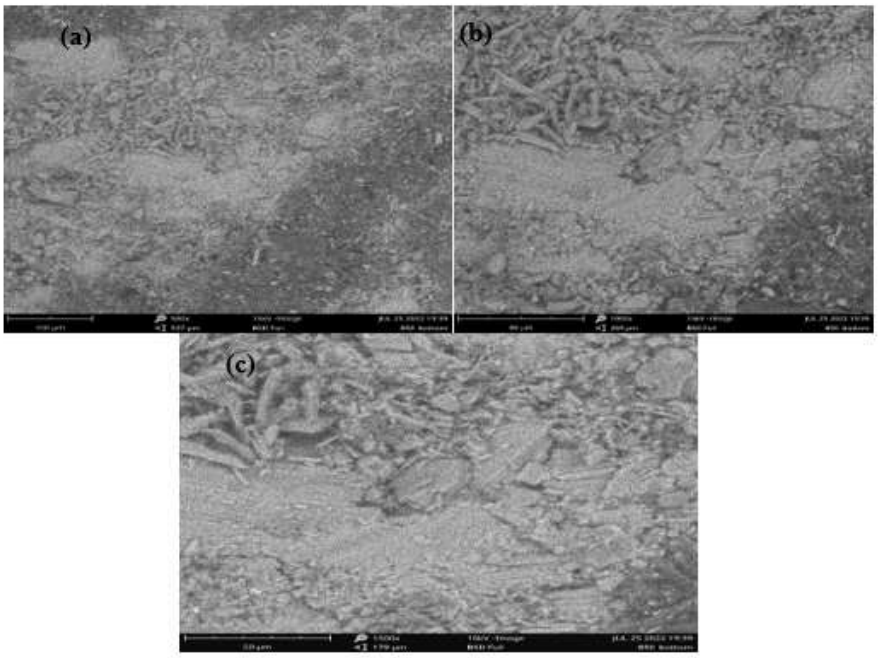
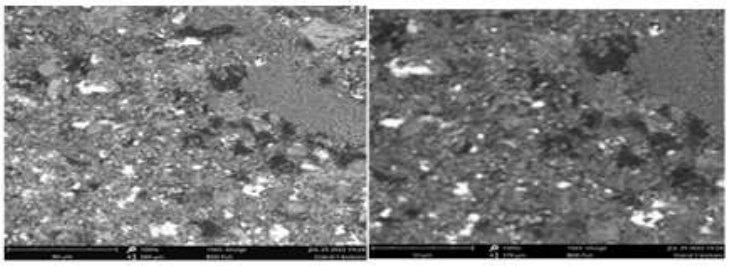


Figure 7. SEM micrographs of the developed brakepad sample with 850µm particle size at magnifications of (a) 500x, (b) 100x, and (c) 1500x

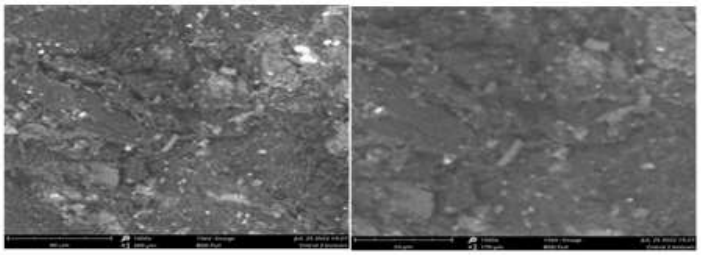
3.3.3. Scanning Electron Micrographs of deformed and Undeformed Surfaces

Images ofdeformed and undeformed surfaces of the developed brake pad samples were taken. The SEM micrograph was taken on the surfaces of the brakepad samples before (undeformed surface) and after (deformed surface) the coefficient of friction test. Figures10 and 11 were obtained from the worn and unworn topography of the developed brake pad composites with the smallest particle size (53µm) and largest particle size (850µm).The 53µm particle size brake pad sample (Figure 10)shows less deformations with the presence of minimal craters and cracks after wear compared to the 850µm particle size brake pad. Crack propulsion is more evident in the 850µm particle size sample(Figure 11) and this may be due to the loose distribution of the various material ingredients due to the coarseness of the debonded particles. The crack observed after friction and wear test further confirms that the wear rate of composites with larger particle sizes is higher than the wear rate of composites developed with finer particles. Hence, the finer the particle size the better the tribological performance of the developed composite. Figures 10 and 11give a better understanding of the trend observed in Figure 5. The wear rate increases with increase in particle size. This observation can be explained by the arrangement of the particles. Smaller particles fix themselves together into spaces between other matrix ingredients minimizing the presence of voids. As a result, the composite becomes very compact.



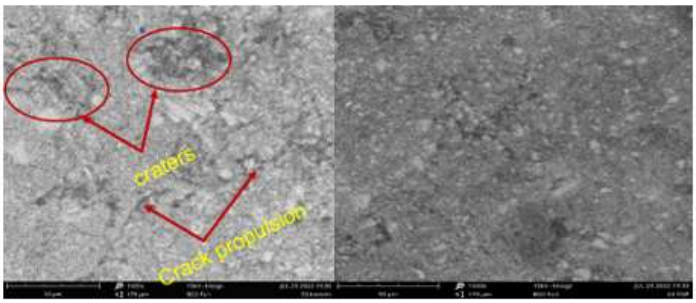
(a) (b)

Figure 8. SEM micrographs of commercial brake pad sample (Control 1) at magnifications of (a) 1000x, and (b) 1500x



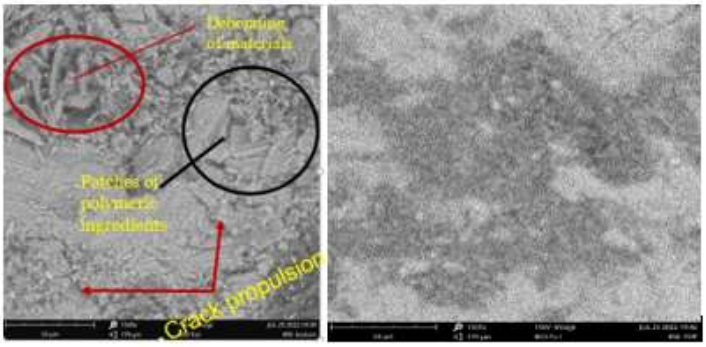
(a) (b)

Figure 9.SEM micrographs of commercial brake pad sample (Control 2) at magnifications of (a) 1000x and (b) 1500x



(a) Deformed surface (b) Undeformed surface

Figure 10. SEM images of the developed brake pad sample with 53µm particle size at a magnification of 1500x.



(a) Deformed surface (b) Undeformed surface

Figure 11. SEM images of the developed brake pad sample with 850µm particle size at a magnification of 1500x.

The presence of loose surfaces and debris craters patches of polymeric ingredients, and primary and secondary plateaus are more pronounced in the commercial samples. Abrasive wear mechanism was observed by the presence of a smear friction layer appearing as a different component on the surface (Figure 13). Also, the presence of small grooves was observed and this may be due to the shearing of small particles. However, the SEM analysis shows that the developed 53µm particle-sized brake pad has an improved overall morphological performance than the commercial brake pads.

4. Conclusion

The study analyzed the tribological properties and microstructureof organic automotive brake pads developed from Thais Coronata seashells. The energy-dispersive spectroscopy (EDX) test was used to evaluate the chemical compositions of the developed brake pads and a comparison of the findings was made with the results of two commercial samples used as control. EDX confirms the environmental healthiness of the newly developed brake pad and detected the presence of heavy toxic metals in the two commercial brake pads analyzed in this study. It was observed that the particle size can significantly affect the mechanical and tribological properties of the brake pad composite. Friction and wear test results show a decrease in wear rate with an increase in particle size and vice versa. The study shows that the developed organic brake pad has better overall morphological and tribological performance than the commercial (control) brake pads and can be used in heavy-duty vehicles.

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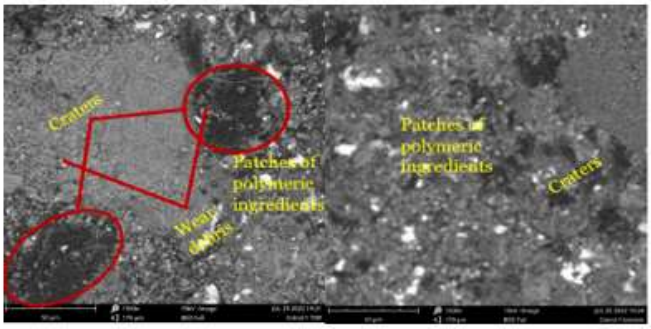
No grant or research fund was received for this work.

Disclosure of Conflict of Interest

The authors have no disclosures to declare.

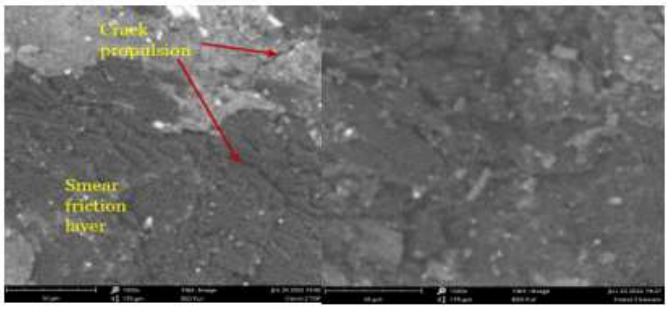
Compliance with Ethical Standards

The work is compliant with ethical standards.



(a) Deformed surface (b) Undeformed surface

Figure 12 SEM images of the commercial sample with (control 1) at 1500x.



(a) Deformed surface (b) Undeformed surface

Figure 13 SEM images of the commercial sample with (control 2) at 1500x.

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