



Oil Residue as an Eco-friendly Brake pad Friction Materials: An Insights on FT-IR and TGA Analysis

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ABSTRACT

The utilization of natural fibers in brake pad formulations offers several advantages, including reduced environmental impact, improved thermal stability, and enhanced friction performance. Thermo-Gravimetric Analysis (TGA) and Fourier-Transform Infrared (FT-IR) characterization techniques are employed to evaluate the performance of these materials. TGA provides insights into the thermal stability and decomposition behavior of natural fiber-based brake pad materials, while FT-IR analysis helps in understanding the chemical composition and bonding characteristics. The results obtained from TGA and FT-IR characterization are crucial in determining the optimal composition and processing conditions for developing high-performance brake pad materials. This research aims to contribute to developing eco-friendly materials including oil cake residues of coconut oil, sesame oil, and groundnut oil for the efficient brake pad materials that meet the rigid performance requirements of modern automotive applications. The TGA analysis represented that these oil residues can withstand thermally up to 630°C and the FT-IR analysis confirmed the presence of distinct chemical compositions and their respective functional groups.

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1. INTRODUCTION

The usage of eco-friendly materials aligned with the goals of sustainability and reducing the hazardous substances, these oil residue compounds support in sustainability initiatives and contributes to greener automotive industry. The brake pad materials are bounded with fillers, reinforcements, binders and resins to populated as structural elements together to provide

physical, chemical and thermal properties [1]. TGA plays a crucial role in the characterization of eco-friendly friction materials, particularly in brake pads [2]. The demand for sustainable and environmentally conscious friction materials is driving the focus on developing products that perform well and have minimal negative impacts on the environment [3]. This provides valuable insights into the thermal stability and decomposition behavior of these materials,

aiding in the evaluation and optimization of their performance. The analysis of friction materials using TGA involves subjecting the samples to controlled heating in an inert atmosphere while continuously monitoring weight changes [4, 5]. By studying weight loss and thermal behavior, researchers can gain a deeper understanding of the composition, stability, and thermal properties of eco-friendly friction materials [6, 7]. It enables the assessment of the thermal stability and performance of friction materials, providing valuable information for material selection, formulation optimization, and quality control [8, 9]. It allows to identify the temperature ranges at which significant weight loss or degradation occurs, which can be correlated with the operating conditions experienced by brake pads during braking [10, 11]. This knowledge helps in designing friction materials that can withstand high temperatures and maintain their performance over extended periods. It can also aid in the identification and evaluation of the thermal decomposition products of the friction materials. In conjunction with other characterization techniques such as FT-IR spectroscopy [12], TGA provides a comprehensive understanding of the thermal and chemical properties of eco-friendly friction materials [13].

FT-IR analysis allows for the identification of functional groups and chemical bonds present in the material, complementing the insights gained from TGA [14, 15]. By utilizing TGA and FT-IR characterization techniques, researchers can evaluate the thermal stability, decomposition behavior, and chemical composition of natural fiber-based brake pad friction materials. This knowledge is essential for developing high-performance, eco-friendly friction materials that meet the stringent requirements of modern braking systems while minimizing environmental impact. In addition, these spectrometer testing plays a vital role in the characterization of eco-friendly friction materials for brake pads. It enables the evaluation of thermal stability, decomposition behavior, and identification of potential harmful emissions. By utilizing TGA and FT-IR techniques, researchers can develop sustainable and high-performance brake pad materials that align with the growing demand for environmentally conscious solutions in the automotive industry. The objective of this research aims to contribute to the development

of eco-friendly alternatives by utilizing natural fibers in brake pad formulations, with a specific focus on oil cake residues. It seeks to investigate and quantify the thermal stability of brake pad materials through Thermogravimetric Analysis (TGA) and Fourier Transform Infra-red (FT-IR) analysis contribute to the improvement of braking efficiency in automotive applications. It also intends to gain insights into the thermal stability, decomposition behavior, chemical composition, and bonding characteristics of the brake pad materials. The transition to eco-friendly friction materials is primarily helped to reduce the particulate matter and dumping materials through wastes, that makes an environmental impact of brake pad wear debris.

2. LITERATURE REVIEW

The investigation on the effects of non-exhaust particle matter on the environment and human health, with a focus on ecologically friendly friction-lining composite materials (FLM) made from sufficient amounts of organic agricultural waste [16]. It has lessened the burden of disposing of natural waste and helped to reduce dangerous materials at their source. The utilization of two types of composites namely carbon fiber and basalt fiber combined with other materials such as vermiculite, epoxy resin, and graphite filler, and subjected to dry tribometer testing for frictional materials [17]. Recovering synthetic and natural fiber composites have been compared with two distinct commonly used asbestos and copper brake pads to demonstrate the brake pad's applicability. The entailed removing and using stem fiber from *Citrullus lanatus* that had been silane-treated and untreated to create environmentally friendly brake friction composites. Analyses have been done on the thermal, chemical, and physical properties [18]. In addition to the parental elements, the application appropriateness has been investigated by creating brake friction composites in the form of brake pads using the traditional approach and contrasting it with the glass fiber-based one.

The investigation of the effects of objective and subjective weighting methodologies in multi-attribute decision-making (MADM) and developed a systematic framework for identifying which natural fiber-reinforced friction composite is best for vehicle braking

applications [19]. Thirteen friction composites have been constructed and evaluated in terms of their tribological characteristics. The development of the natural fiber *Grewia Optiva* as a sustainable material for vehicle brakes [20]. Brake friction composite materials have been created in varying volumes. The composites' physicochemical and mechanical characterization, noise propensity, and tribological properties have all been evaluated. The investigation on how ramie fiber affected the mechanical, tribological, and physical characteristics of composite materials used in automotive brake friction. The composite samples' mechanical and physical characteristics have been assessed under industry standards, and their tribological characteristics have been assessed using a Chase machine and the IS 2472-4 test protocol [21, 22]. The investigation on the extraction and characterization of natural fibers from Guava plant stems that have been treated with silane and used as brake pads for automobiles. The crystallographic, physical, and chemical characteristics of the treated and untreated fibers were examined. These fibers were employed in the brake pad composite that was created utilizing traditional industrial methods, and they were examined for several performance attributes by industry norms [23]. The experiment of brake pad samples reinforced with lignocellulosic banana fibers reinforced with phenolic resin, which exhibits strong thermal stability and resistance to brake fade. Using a pin-on-disc tribometer, four distinct brake pad samples with varying ratios of phenolic resin and banana fiber have been created and examined. High loading and temperature have been employed to conduct the friction testing. Using TGA and DSC methods, the samples' thermal stability has been investigated [24]. The effective investigation on the effects of palm fiber on the properties of composite materials used in automotive brakes. Three distinct brake pads' tribological properties have been evaluated for performance parameters like temperature sensitivity and pressure-speed using a full-scale inertia dynamometer [25]. The investigation on the possibilities of high-performance Zylon fibers (ZF) in brake pads for the first time. Furthermore, Aramid pulp and fibers have been chosen as theme elements; these substances would compete within brake pads. By holding parent ingredients constant, six different types of brake pads have been created [26]. The bamboo

microcrystalline cellulose /poly (lactic acid)/ poly(butylene succinate) composites show potential as a biodegradable alternative to synthetic plastics due to their improved tensile and thermal properties [27]. The investigation on date palm fibers with NaOH solutions to enhance mechanical properties by testing through FTIR, TGA and DSC to be used as green reinforced composites [28]. The presence of bonding between the alkaline and aliphatic chains are compared with this study to enhance the functional groups without adding extra agents like NaOH solutions.

The pursuit of these understandings is not about the performance of chemical characteristics but to enhance the knowledge by contributing to society harmlessly. It is an essential and critical aspect of creating more sustainable world. Through continuous advancements in technologies the potential of eco-friendly materials can pave the way for a more environmentally conscious society.

3. METHODOLOGY

Coconut oil residue (COR), groundnut oil residue (GOR), sesame oil residue (SOR), and a combination of coconut, groundnut, and sesame (CGS) oil residues are the components that are taken into consideration for TGA and FT-IR testing. These ingredients are taken out of the corresponding oil extraction facilities and ground into a powder in a nearby flour mill. Using a technique called TGA, weight variations in temperature or time can be measured to learn more about a material's composition and thermal stability. Conversely, FT-IR examines a material's infrared absorption or emission to help determine which functional groups it has.



Fig. 1. Samples of oil residues.

2.1 Thermogravimetric Analysis

TGA is an effective method for determining a material's thermal stability. This method involves measuring a specimen's weight changes while its temperature is raised. TGA is a useful tool for measuring a sample's moisture and volatile levels. The device is made up of a programmable furnace to regulate the sample's temperature and an extremely sensitive scale to record weight variations. The balance is separated from the heat by a thermal barrier and is situated above the furnace. From the balance down into the furnace is suspended a high-precision hang-down wire. The sample pan is at the end of the hang-down wire, and its location needs to be consistent. To increase the sensitivity, accuracy, and precision of weighing, the balance must be insulated from thermal effects (for example, by using a thermostatic chamber). The identification and analysis of gases produced by the sample's deterioration is made possible by the inclusion of an infrared spectrometer to TGA. An easily cooled micro-furnace is a feature of the TGA device. Platinum is used to make the heating element, which is dependable up to 1000°C. The temperature can be raised to 1500°C using an external furnace equipped with a heating element composed of an alloy comprising 30% rhodium and 70% platinum. The weight-loss fraction or % is often calculated by a computer that comes with most contemporary equipment. A commercial TGA can operate in an environment of air or another gas at temperatures above 1000°C, with a 0.1µg balance sensitivity and a variably regulated heat-up rate. TGA has a range of heat-up rates from 0.1°C to 200°C/min. The x-axis of the TGA Thermal Curve displays time or temperature, while the y-axis shows weight (mg) or weight percent (%). The reference instrument of TG-DTA is shown in as fig. 2.

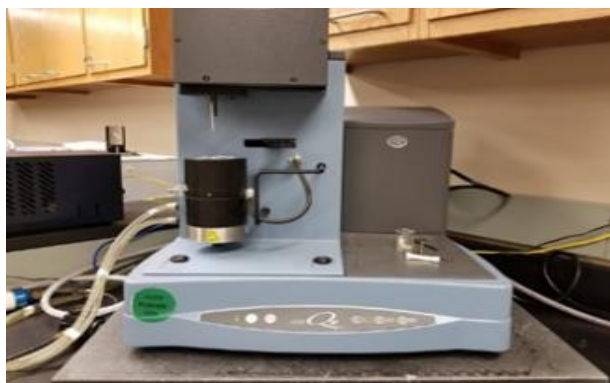


Fig. 2. Ref image of TGA instrument.

2.2 Fourier Transform Infrared Analysis

Owing to its capacity to examine and determine the chemical composition of a broad variety of materials, FT-IR spectroscopy is an important method for choosing a material composition. To scan test samples and observe functional characteristics, the FT-IR analysis method uses infrared light. Part of the infrared radiation that the FT-IR instrument passes through and part is absorbed by the sample when it passes through at a wavelength of between 10,000 and 100cm⁻¹. Utilizing rotational or vibrational energy conversion, the sample molecules transform the absorbed radiation. The signal that emerges at the detector is a spectrum that shows up as a molecular fingerprint of the material, usually ranging from 4000 cm⁻¹ to 400 cm⁻¹. Because every molecule or chemical bonding has a distinct spectral fingerprint, FT-IR analysis is an excellent method for identifying functional groups of powders, gaseous mixtures and liquid pollutant.



Fig. 3. Ref image of FTIR instrument.

The measurements were performed using the Shimadzu IR Affinity-1 FT-IR Spectrophotometer, which is a type of instrument manufactured by Shimadzu is shown in as reference in fig.2. The instrument has a wavenumber range of 400 – 4000 cm⁻¹ and a spectral resolution of 4 cm⁻¹. The measurement mode used was % Transmittance, and the infrared beam used for the measurements was internal. The speed of the interferometer mirrors, which affects the measurement process, was set at 2.8 cm/s.

4. RESULT AND DISCUSSION

4.1 Results from TGA

The high thermal stability of the polymer and composite, or high decomposition temperature, indicates an effective flame retardant function.

The thermal behavior, including weight loss and residual char level, as well as the material's decomposition at a particular temperature, were ascertained using the TGA and Optimum Degradation Temperature (DTA) curves, respectively. Tables 1 and 2 display the findings from the examination of the suggested materials, and fig.4 and fig.5 display the associated graphs for each table. The summary statistics of dependent, independent and control variables for pre-crisis period are presented in the Table 1.

Table 1. TGA result.

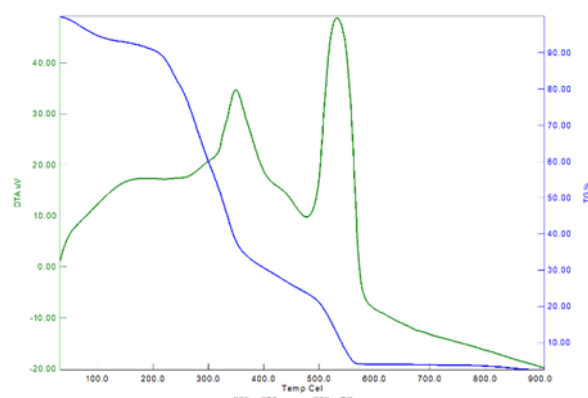
Material	First weight loss	Second weight loss	Combustion	Residue value
COR	4.3%	39%	13%	13%
GOR	4.2%	38.9%	12.2%	12.2%
SOR	3.5%	33%	14.6%	14.6%
CGS	2.5%	38.6%	12.1%	12.1%

Table 2. DT result .

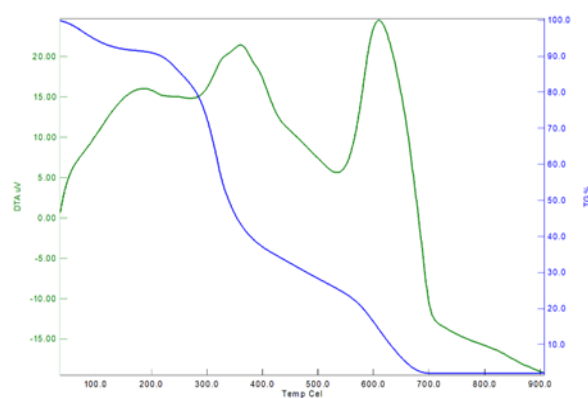
Materials Used	First derivative	Second derivative
COR	300°C	500°C
GOR	215°C	570°C
SOR	200°C	630°C
CGS	200°C	618°C

Table 1 displays thermal degradation data at different weight-loss stages for several materials, most likely polymers. During thermal analysis, the materials COR, GOR, SOR, and CGS go through three successive weight loss phases. For every material, there is a different degree of instability and plasticizer loss in the first phase. In the second stage, there is less volatility and more weight loss as a result of polymer degradation; CGS shows the least amount of initial weight loss. Comparable values among the components indicate weight loss by burning in the third phase. The residual values found in the final column represent the amount of ash that remains after burning, most likely as a result of inorganic filler components that were originally contained in the compounds. Overall, the evidence points to variations in the material's thermal stability and breakdown behavior, with CGS showing unique properties throughout the early phases of degradation. The results of TGA on four different materials—COR, GOR, SOR, and CGS—are summarized in Table II. The detection of DTA is

made easier by TGA, a technique that measures weight changes in materials as a function of temperature. Finding the temperature at which the first degrading step displays the highest rate of weight change allows one to determine DTA. The table offers insights into material-specific DTA values and summarizes the temperature range linked to considerable weight loss for each material by the curves shown in fig. 4. Remarkably, COR deteriorates primarily between 300 and 500 degrees Celsius, most likely peaking at 500 degrees. From 215°C to 570°C, GOR degrades, suggesting the possibility of a DTA close to 570°C. 200°C to 630°C is the key deterioration range for SOR, and 630°C is probably the DTA. Degradation is shown by CGS between 200°C and 618°C, with a predicted DTA of 618°C. Greater DTA values indicate improved thermal stability, indicating a higher level of resistance to deterioration at high temperatures. With the highest upper-temperature limit in this situation, GOR sticks out from the other materials and may have better thermal stability. Researchers and engineers can evaluate a material's appropriateness for a certain application, especially in high-temperature conditions, by using DTA as a quantitative measure.



3a) COR_TGA



3b) GOR_TGA

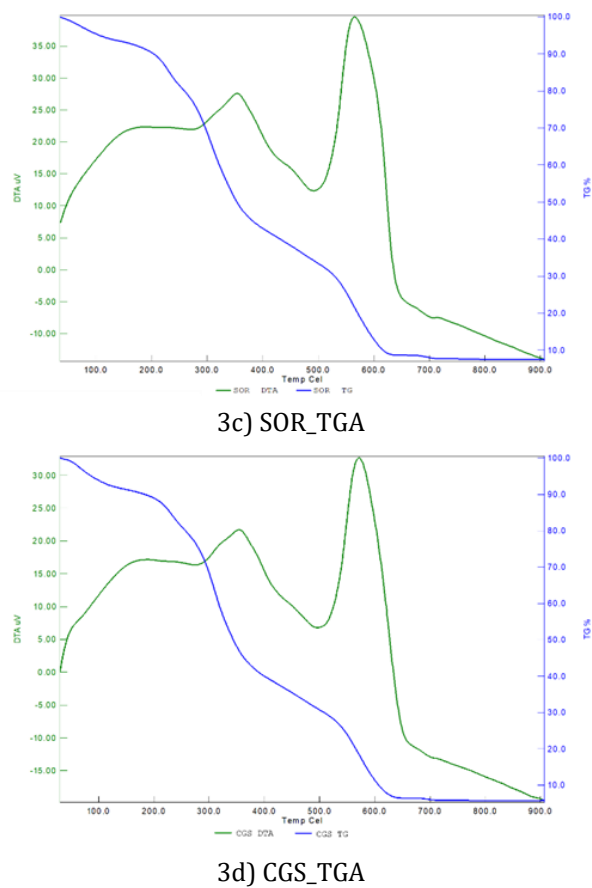


Fig. 4. Graphical representation of TG and DTA curves.

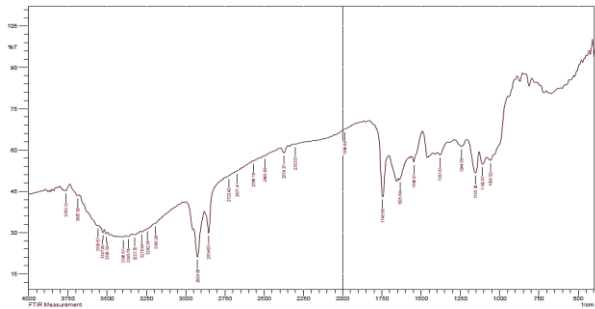
4.2 Result from FTIR

The FT-IR results for the brake pad friction materials based on natural fibers are shown in Table III, with an emphasis on distinguishing peaks at particular wavenumbers that reflect individual chemical compositions. The plot in fig. 5 shows indicative of alcohols or phenols, the O-H stretching vibrations at 3687.90 cm^{-1} in COR, 3624.25 cm^{-1} in GOR, 3775.32 cm^{-1} in SOR, and 3698.84 cm^{-1} in CGS point to the existence of hydroxyl groups. The C-H stretching at 2924.04 cm^{-1} in COR, 2924.09 cm^{-1} in GOR, 2926.01 cm^{-1} in SOR, and 2924.09 cm^{-1} in CGS indicates the presence of aliphatic hydrocarbons, which are constant in all samples. The presence of esters produced from fatty acids is indicated by the presence of carbonyl groups, which are underlined by C=O stretching at 1745.58 cm^{-1} in COR, 1662.64 cm^{-1} in GOR, 1650.21 cm^{-1} in SOR, and 1652.41 cm^{-1} in CGS. Moreover, the presence of carbon-carbon double bonds is indicated by the C=C stretching at 1635.64 cm^{-1} in COR, 1504.89 cm^{-1} in GOR, 1510.81 cm^{-1} in SOR, and 1525.31 cm^{-1} in CGS. By merging characteristics from

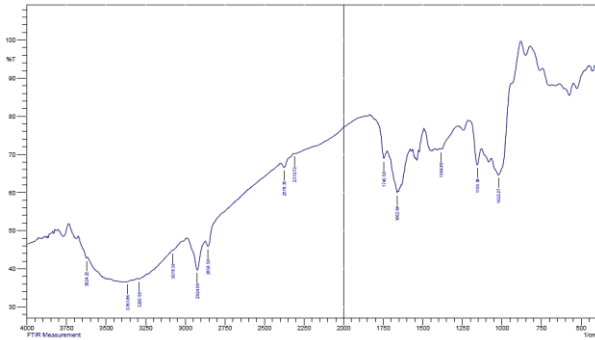
different residues, the CGS mixture displays a composite spectrum that offers a detailed comprehension of the chemical variety among these oils. In conclusion, the FT-IR analysis enables a comparative comprehension of the chemical compositions of each oil residue and offers precise insights into the functional groups present in each one.

Table 3. FTIR spectra band.

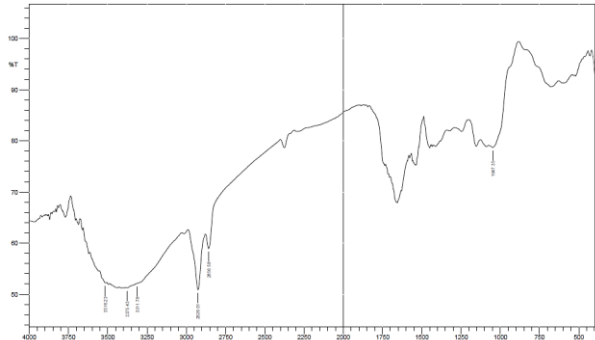
Materials	Wavenumber in cm^{-1}			
	O-H	C-H	C=O	C=C
COR	3687.90	2924.04	1745.58	1635.64
GOR	3624.25	2924.09	1662.64	1504.89
SOR	3775.32	2926.01	1650.21	1510.81
CGS	3698.84	2924.09	1652.41	1525.31



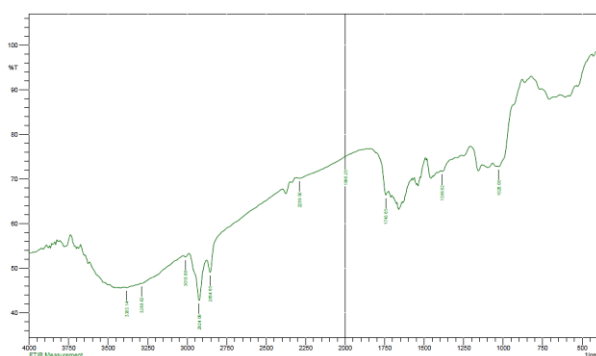
4a) COR_FTIR



4b) GOR_FTIR



4c) SOR_FTIR



4d) CGS_FTIR

Fig. 5. Graphical representation of FTIR curves.

4.3 Overall Result

The comprehensive analysis of natural fiber-based brake pad friction materials through combined TGA and FT-IR analyses yields a theoretical interpretation that deepens understanding of the material's thermal and chemical properties. Each material, including COR, SOR, GOR, and the composite CGS, serves distinct purposes, making it crucial to weigh their environmental impact, thermal stability, and friction performance. Regarding environmental impact, COR and SOR, derived from natural sources, may have a potentially lower adverse effect compared to polymer and material-based alternatives; however, a comprehensive life cycle evaluation is necessary for a thorough assessment. In terms of thermal stability, GOR and COR exhibits superior characteristics, making it preferable in situations where resistance to elevated temperatures is crucial. When considering enhanced friction performance, the diversified chemical composition of CGS, as indicated by FT-IR research, holds promise, though specific application requirements such as thermal conductivity and wear resistance play pivotal roles. The characteristics of functional groups can be compared with the existing literature studies [28].

5. CONCLUSION

The thermal stability, chemical composition, and possible performance characteristics of the natural fiber-based brake pad friction compounds COR, GOR, SOR, and the mixture CGS have all been better understood attributable to the TGA and FT-IR characterizations of the

forementioned materials. While FT-IR analysis discloses the diverse chemical compositions and functional groups of each material, TGA data show distinct patterns of thermal degradation, with GOR demonstrating higher thermal stability. The mixture known as CGS exhibits chemical variety. These discoveries establish the groundwork for future developments in brake pad technology by providing insight into the behavior of the materials under varied circumstances. To sum up, the knowledge gathered from TGA and FT-IR studies provides a strong basis for developing natural fiber-based brake pad materials. Subsequent investigations ought to give precedence to the enhancement of composition utilizing sharpening blend ratios or investigating substitute natural fibers. Thorough performance testing is necessary to get useful insights into wear resistance, friction performance, and overall braking efficiency in real-world scenarios. Further research into how surface treatments and additives affect thermal stability and friction characteristics might also improve the performance of materials. It will emphasize the utilization of industrial and agricultural wastes in brake pads minimizes environmental pollution and promotes solid waste management. This research may not only addressing waste disposal of brake pads, also recovers by product value from waste materials to implement and contributes in economy. These improvements can lead to reduce manufacturing costs that extracts from earth's crust and enhancing overall effectiveness.

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