Evaluation of Blue Cupressus Arizona Cone in Automotive Brake Pad Biocomposite

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Recently, new brake pads have been developed using sustainable materials that are not harmful to the environment. In this study, the effect of using blue-colored *Cupressus arizonica* cones was determined as a friction modifier in brake pad composites. Four different samples were prepared by grinding the cone material. The samples' physical and chemical properties and performances were compared, and finally, their microscopic analyses were visualized by scanning electron microscopy. Maximum friction coefficient and minimum wear rate are required for brake pads. The maximum friction coefficient of 0.39 and the minimum wear rate of the samples containing *Cupressus arizonica* were obtained in the 0.124X10^{-7} cm^3/Nm BCA3 sample. When the brake pads developed from lignocellulosic biomass were compared, it was determined that the BCA3 sample met the most reasonable performance values.

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INTRODUCTION

Due to environmental effects and strategies to achieve sustainability, alternative manufacturing chains are more essential in the materials sector. These demands prompt the value-added upcycling of agricultural waste. The waste is increasingly employed for biocomposite materials, particularly in the construction and transportation industries. These materials are selected for various socioeconomic and environmental reasons in addition to their physical and chemical qualities (Mathias *et al.* 2015).

Composite materials are materials obtained by combining two or more materials. On the other hand, biocomposites consist of plant fibres, matrices, *etc*., of the components used in the material. Composites by a biological source, such as properties that the formation of materials alone cannot obtain, are achieved by combining materials with different properties (Haris *et al.* 2022). Biocomposite, a mixed group, consists of materials from which at least one of the materials forming the structure, such as a matrix, filler, or reinforcing material, is of natural origin (Hassan *et al.* 2010).

Materials made from renewable resources have become more popular due to the promotion of sustainability in economic development, methods for ecology, and environmental conservation. Ecological and environmental regulations by nations have increased the utilization of natural resources as an innovative solution, in addition to the materials providing the desired economy, mechanical, and thermal properties in studies of polymer composites (Majewski and Cunha 2018).

Due to the need to develop environmentally friendly products worldwide, researchers are searching for new materials that will decrease the environmental impacts of the products they have developed. This need has led to the use of composites made from raw natural fibers and polymer matrices, recently making this subject a popular research topic. These materials help to control pollution problems as an alternative to replacing environmentally harmful substances. In addition, low cost, good mechanical properties, and low energy consumption are essential advantages. The use of various thermoset and thermoplastic polymers and their effects on their composites can also be discussed (Sanjay *et al*. 2018). Today, most researchers focus on modelling the properties of natural fiber-reinforced composites and developing biopolymer-based natural and ecological composites as matrix material. The central axis of these studies is that they are cost-effective and technologically advantageous compared to traditional materials. Composite materials developed from thermoplastic, thermoset, and biodegradable materials exhibit tribological, mechanical, thermal, and physical properties (Rangappa *et al*. 2022). As stated by the researchers, these features have an essential place in brake pads. Optimum performance properties for brake pads depend on the properties of the materials in the composite. Due to this, studies on plant-based materials have been conducted.

Researchers have ground pine cones (Sugözü and Sugözü 2020a; Mazur *et al.* 2022), pine cone powder (Aras and Tarakçıoğlu 2021), *Pinus brutia* cone (Jeganmohan and Sugozu 2019), hazelnut shell (Öktem and Uygur 2019), walnut shell (Akıncıoğlu *et al.* 2020), banana peel (Sugözü and Sugözü 2020b), miscanthus (Kchaou *et al.* 2021), palm kernel powder (Jeganmohan *et al.* 2020), rice straw powder (Mutlu and Keskin 2021), plant-based natural fibres (Yashwhanth *et al.* 2021), cashew (Singaravelu *et al.* 2019), pineapple fibre (Singh *et al.* 2020), palm kernel (Pujari and Srikiran 2019), corn stalk fiber (Liu *et al.* 2019), flax fibre (Sathish *et al.* 2019), and palm kernel husk (Fono-Tamo and Koya 2017) for use in brake pads. Researchers in different fields of biocomposite production have used epoxy composites reinforced with Banyan air root fibers (Ganapathy *et al.* 2021). Due to the specific parameters revealed by these advanced composites, there is research interest in subjects with low density and high thermal properties (Hemath *et al.* 2020). In the composite materials formed, the resistance to surface wear increased with the increase of the fiber matrix interface bond strength (Vinay *et al.* 2021). As the researchers have obtained from their studies, in brake pads, high wear resistance, high thermal properties, and a durable structure are desired in the desired friction coefficient performance. For all these reasons, this study will help the search for ecologically environmentally friendly green materials.

The desired performance of brake pads can be improved by well-designed recipes that use novel components (Öktem *et al.* 2021). Brake pads are the most critical parts of the braking system that slows down or stops vehicles. The fact that the pads are hard causes the disc or drum forming the opposite side of the friction surface to wear out quickly, and if it is too soft, the pad will wear out quickly. In addition to providing the desired performance characteristics, the developed pads should not release components that are harmful to the environment. The intended performance of plant-based goods brake lining composites has been employed in several scientific investigations. To the author's knowledge, no research has been done on the braking effectiveness of biocomposites made of blue colored *Cupressus arizonica* cones. This study examines biocomposites' manufacturing potential and braking capability made from blue colored *Cupressus arizonica* cones for automotive brake pads.

EXPERIMENTAL

Preparation of Blue *Cupressus arizonica* Cones

There are approximately 20 species of cypress trees or shrubs, as well as various subspecies. The *Cupressus arizonica* species can grow in moist pure sandy soils, light-heavy loam, or arid calcareous soils in suitable climatic conditions. It usually grows in arid and poor lands and calcareous soils where the Mediterranean climate dominates (OGM, 2009). Due to these features, it is a species seen in almost every region of Turkey. This species can also be produced in different regions of the world. This study characterized the material properties of blue *Cupressus arizonica* (BCA) cones harvested from Afyonkarahisar, Turkey. Cones were collected from the blue *Cupressus arizonica* trees when they were close to brown at maturity. The process of pulverizing BCA cones is shown in Fig. 1. The cones in Fig. 1a were collected from the BCA tree. Cones collected in the process of Fig. 1b were set up for one week under the sun. The seeds in the cones were removed by opening the dried cones. In Fig. 1c, the washing process purified the cones from foreign objects such as dust, soil, and dirt. The washed BCA cones were dried in a heat treatment oven at 40 °C for 48 h, as shown in Fig. 1d. Cones dried in Fig. 1e were ground by a mill and sieved with 815 µm for usability.

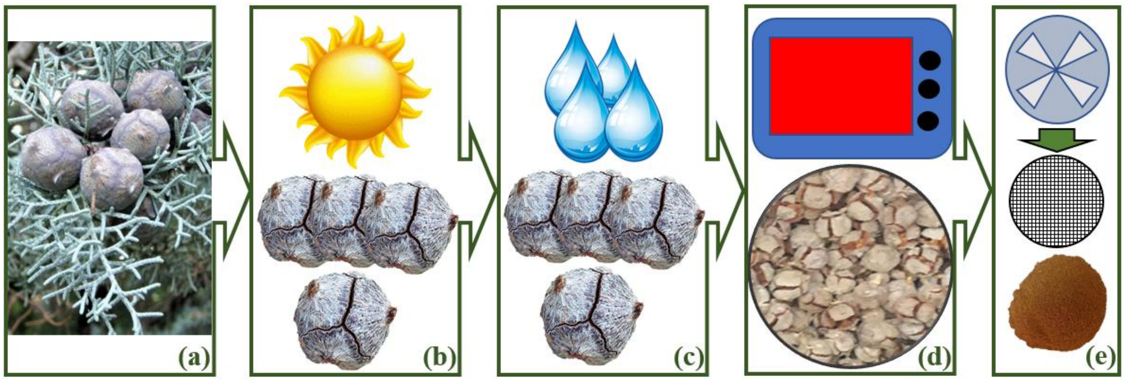

Fig. 1. Powdering process of BCA cones

Scanning electron microscopy (SEM) image and Energy Dispersive X-ray analysis (EDX) content of powdered BCA cones are shown in Fig. 2. The chemical content of powdered BCAs was approximately 99.36% carbon and 0.64% potassium content.

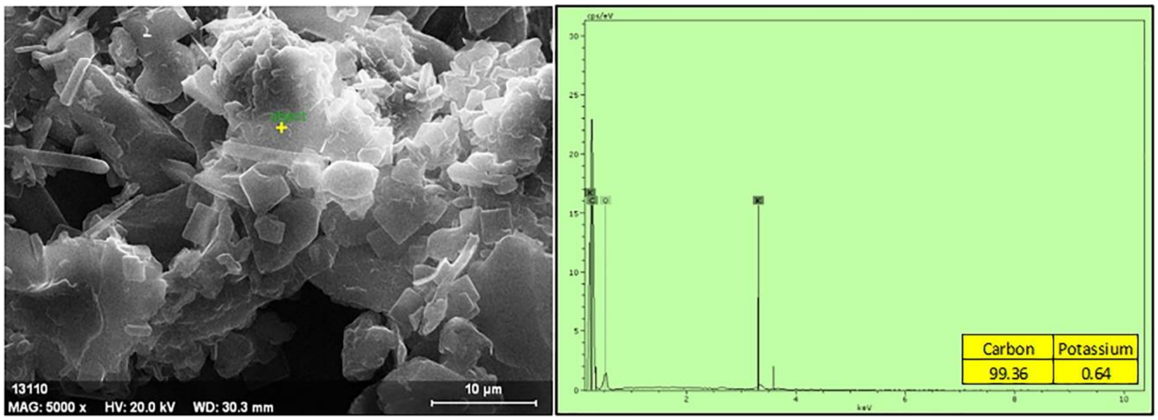

Fig. 2. SEM and EDX images of powdered cones that had been processed

Table 1 shows the materials used in brake pad biocomposites in 4 different forms and their percentages in the mixture. Brass powder, which is widely used as a friction modifier in brake pads, was used at a constant rate. Cashew powder and BCA cone powder were used as friction modifiers at varying addition levels. The number next to the sample codes indicates the amount of BCA cone powder contained in the biocomposite, measured in percent by weight.

Materials in Table 1 were used to create brake pad composites in the order depicted in Fig. 3. Production parameters were determined by previous studies (Yavuz and Bayrakçeken 2022; Yavuz 2023). The materials that make up the brake pad composites were weighed with a 0.001 mg precision balance in the first process. A kinetic mixer was used to ensure the weighed materials were mixed homogeneously. The materials were mixed at 60 rpm for 15 minutes in a kinetic mixer. The mixed materials were produced in a hot press for 15 minutes at 150 °C at a pressure of 15 MPa. The mold was produced with six chambers and simultaneously produced six samples.

Table 1. Weight Percent of the Substances that Make up the BCA Composite

Sample Code	**Binder**	**Fiber**	**Abrasive**	**Lubricant**	**Fillers**	**Friction Modifier**		
	Resin	Steel	Alumina	Graphite	Barite	Brass powder	Cashew	BCA powder
BCA0	20	16	10	5	35	5	9	0
BCA3							6	3
BCA6							3	6
BCA9							0	9

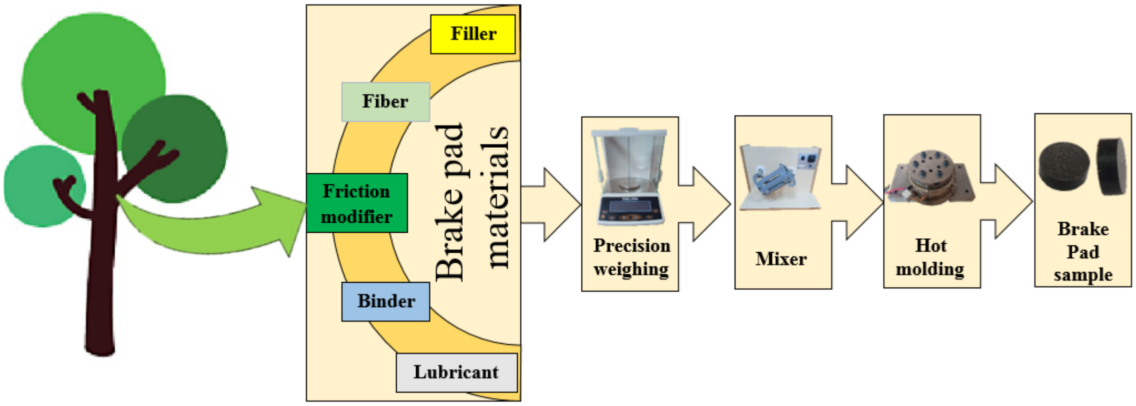

Fig. 3. Materials and production process of brake pad biocomposites

Mechanical Tests

Figure 4 displays the performance traits of the brake pads created during the experimentation phase. The densities of the brake pads were initially measured. The brake pad tester was then used to calculate the brake pad's friction coefficient and wear rates. On the friction surfaces of the samples, hardness evaluations, SEM (scanning electron microscopy), and EDX (energy dispersion X-ray) analyses were conducted.

It is crucial to determine the friction properties of the brake pads to ensure the safety and efficiency of brake systems for automobiles. Therefore, the brake pad tester was used to measure the brake pad's wear rates and friction coefficients. The endurance and mechanical characteristics of the material were revealed by hardness measurements taken on the friction surfaces of the samples. SEM analysis reveals the surface morphology and structural features of the samples, while EDX analysis determines the chemical compositions of the samples. These experiments were used to correctly evaluate the developed brake pads. The necessary information was obtained to develop and improve brake pads. Information about the measurements and experiments is given under the headings below.

Specific Weight Measurements

Specific weight measurements are required to determine the wear rates of the developed samples. The specific weights of brake pad biocomposites produced with BCA cone powder were determined with an accuracy of 0.001 mg with a density measurement kit on a precision balance (Murbay, Seles, JTA500, Turkey) according to the Archimedes principle.

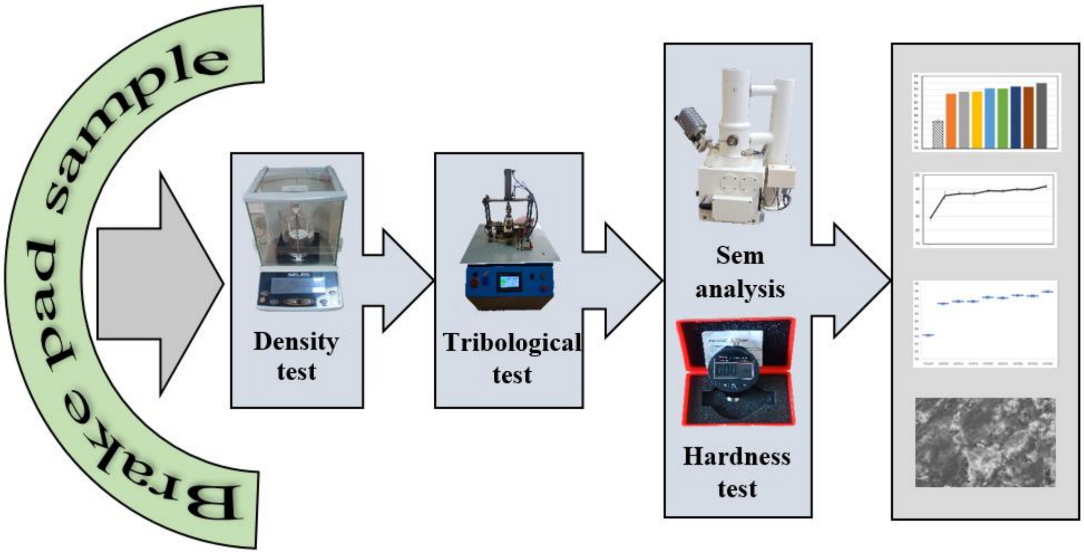

Fig. 4. Brake pad samples test stages

Coefficient of Friction and Wear Rate Tests

The friction coefficient and wear rate tests were performed in a brake pad test device produced in a laboratory environment under brake pad standards. The wear rate and coefficient of friction are crucial factors in determining how well the created biocomposites work. The brake pad tester instantly records the applied load, friction force, and temperature. The friction coefficient was obtained by calculating the ratio of the friction force measured in the brake pad tester to the applied load (*TS 555, Road Vehicles - Brake Linings and Pads for Friction Type Brakes*, 2019). The wear rate was calculated from Eq. 1 according to the mass wear rate formula.

$$W_a = \frac{\Delta G}{S.M.d} \tag{1}$$

where W_a is mass wear rate (cm^3/Nm), ΔG is weight loss (g), S is sliding distance (m), M is loading weight (N), and d is density of the wearing material (g/cm^3).

The experiments for friction coefficient and wear rate were run at disc speeds of 6 m/s, 1 MPa loads, and dry sliding distances of 5000 m.

Hardness Tests

Hardness tests of brake pad biocomposites were conducted with a Shore D measuring device (Tomastine, China) in ASTM D2240 standard. The average values taken from 5 different measurement points of 3 samples for each sample code gave the Shore D hardness measurement results.

Scanning Electron Microscopy (SEM) and Electron Dispersive X-Ray (EDX) Analysis

SEM and EDX analyses (LEO 1430 VP, Carl Zeiss AG, Jena, Germany) model are essential to evaluate biocomposite's friction performance. The carbon coating process (BAL-TEC, SCD 005, Balzers, Liechtenstein) was performed before SEM analysis of biocomposites. Carbon-coated samples were analyzed at 1000X magnification.

RESULTS AND DISCUSSION

Coefficient of Friction Properties

One major issue affecting brake pads is the fact that the friction coefficient decreases during braking. This happens as a result of changes in environmental conditions and fading. Ideally, the change in the friction coefficient should be minimal and constant to provide features such as high comfort for the driver and repeatability with safe braking (Mahale *et al.* 2017; Saikrishnan *et al.* 2022). The instantaneous coefficient of friction is the most direct determinant of the friction properties of biocomposite brake pad samples (Zhang *et al.* 2019). Figure 5 shows temperature and friction coefficient graphs according to dry sliding distance for all samples. The coefficient of friction in the graphs can be evaluated in three stages. In evaluating the coefficient of friction, the stages can be named the increasing stage, decreasing stage, and constant stage. The initial phase of the experiment revealed an increase in all the friction coefficient graphs. The rise occurs until the brake pad biocomposite' maximum friction coefficient. The brake pad samples experience an increase due to gradually increasing the applied pressure force as they become accustomed to the disc.

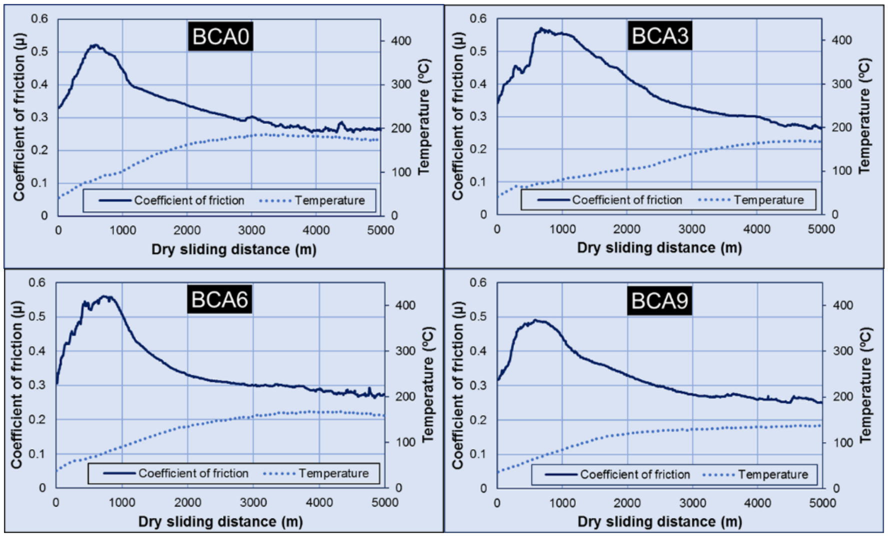

Fig. 5. Coefficient of friction and temperature graphs of biocomposites

A crucial factor in determining how well brake pad biocomposites operate is fade. As temperature rises, the fading phenomenon manifests itself as a reduction in the coefficient of friction. Fading occurs because biocomposite components suffer thermal degradation due to the disc and pad's rising temperatures. On the disc surface, materials that lose their characteristics create an adhesive film. The coefficient of friction is decreased by this adhesive coating (Gopal *et al.* 1995; Mahale *et al.* 2017). The graphics show that the friction coefficient decreased after the sliding distance of 500 to 750 m. This is the second stage of the test. Fade in brake pads result from the resin's binding ability decreasing as the temperature rises. The brake pad biocomposites' friction coefficients slightly decreased due to this property. The change in friction coefficient should ideally be slight (Mahale *et al.* 2019). The friction coefficient curve in Fig. 5 is parallel to the abscissa. After the 2000 m sliding distance, the friction coefficient curves of the biocomposites provided a similar and stable appearance to the apse.

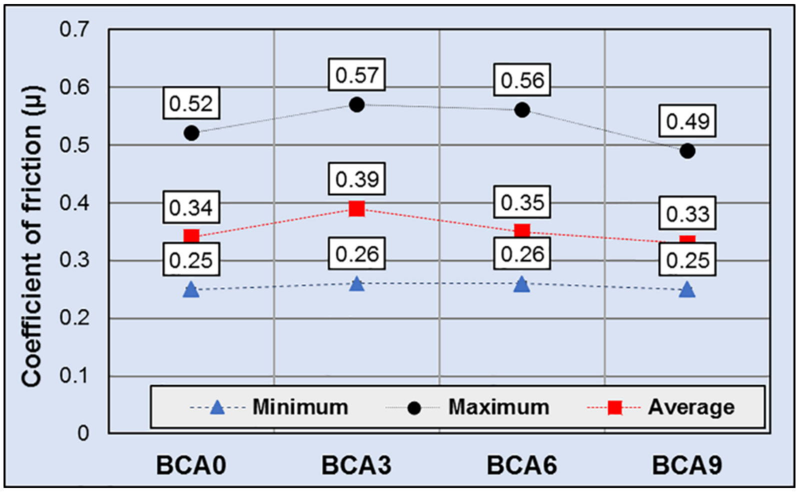

Fig. 6 Minimum, maximum, and average friction coefficients of biocomposites

Brake pads are desired to have a high and constant coefficient of friction (Zhao *et al.* 2020). The friction materials that make up the brake pad provide a thermally stable property and a sufficient friction coefficient. The thermal, physical, and chemical properties of brake pad composites can be changed continuously by numerous material selections and production processes. New properties can be achieved when different substances are added to the materials that make up the composite (Yusubov 2022). The minimum, average, and maximum friction coefficients of the biocomposites for all samples are given in Fig. 6. The maximum and average friction coefficients increased with the addition of 3% BCA to the BCA3 brake pad biocomposite. High friction coefficient brake pads are preferred. The BCA3 sample's friction coefficient, which is often more significant than the other samples at 0.39, is ideal. A desirable characteristic of brake pads is that their friction coefficient falls between 0.20 and 0.70 (*TS 555*). The friction coefficient in all brake pad samples is in the desired range. The average friction coefficient was similar to the biocomposite-based brake pad studies conducted by the researchers (Jeganmohan and Sugozu 2019; Aras and Tarakçıoğlu 2021). Providing the desired friction coefficient and stability in the developed brake pads may be due to the natural resins in the cone, as has been indicated earlier (Aras 2019).

Specific Weight Properties

The specific weight of the samples is included in the calculations to determine the brake pad wear rates. The specific weight of the developed biocomposites is shown in Fig. 7. With the addition of BCA to the sample, the specific weight of the samples increased slightly. The specific weight of all samples containing BCA was 2.46 g/cm^3.

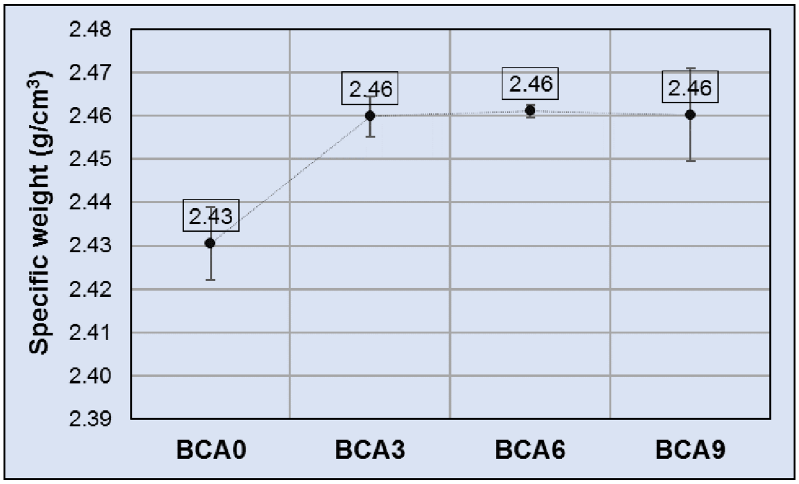

Fig. 7. Specific weight of biocomposites

Wear Rate Properties

The wear rate, a complex structure in brake pads, is governed by a combination of different mechanisms controlled by adhesion, abrasiveness, oxidation, and temperature (Vijay *et al.* 2020). Figure 8 shows wear rates for brake pad biocomposites. Brake pad wear rates were similar to those that have been reported (Akıncıoğlu *et al.* 2020; Jeganmohan and Sugözü 2019). The force applied to the brake pads has a dynamic behaviour. The tribological layer is dependent on the temperature at the interface from friction with the speed of rotation of the disc, as well as on the composite's composition.

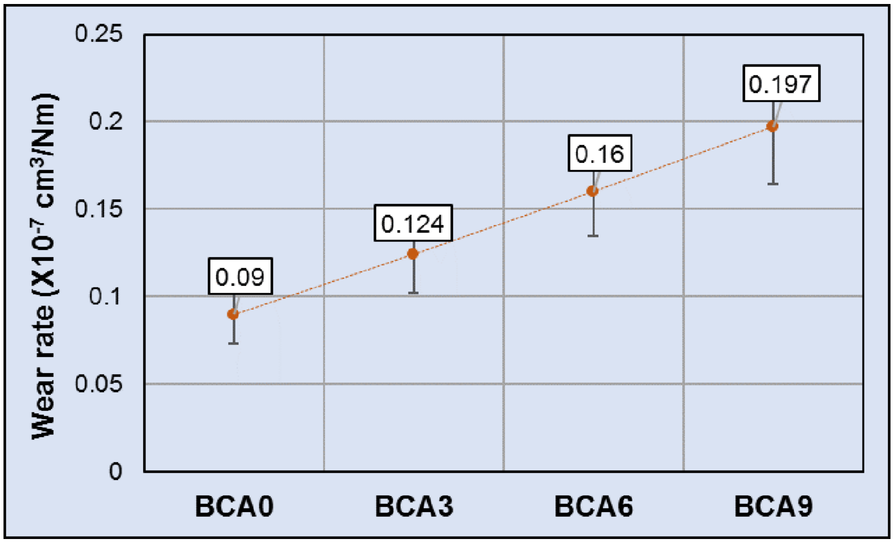

Fig. 8. Wear rates of biocomposites

Wear develops due to plastic deformation on the contact surface as a function of heat and load. Cracks, pits, and material losses occur on the brake pad contacting surface due to impact-based loads and vibrations. Because of their low thermal stability, organic compounds containing BCA deteriorate quickly at high temperatures as the amount of organic matter in the brake lining grows, leading to higher wear (Kanagaraj *et al.* 2023).

Hardness Properties

Hardness testing can be a valuable tool for identifying surface improvement even if original surface, hardened surface, friction film, and softened surface all have variable hardness. The brake pad acts as a braking pair by self-sacrificing to produce friction without harming the disc. Therefore, for the application of the coupling, lower stiffness and strength are crucial (Zhao *et al.* 2020). Wear resistance of brake pad biocomposites increases with increasing hardness (Kanagaraj *et al.* 2023). Figure 9 displays data for samples of brake pads having different hardness. Compared to the BCA0 biocomposite, the BCA9 brake pad biocomposite demonstrated approximately 1.5% less wear. As a result, the wear rate increased as the BCA cone content in the sample content increased. The hardness values were approximately 10% higher than the study in the literature (Solomon and Berhan 2007).

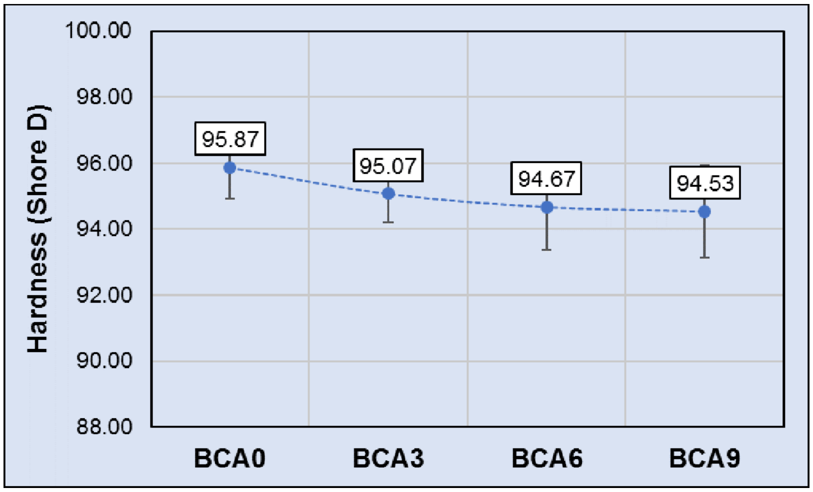

Fig. 9. Brake pad Shore D hardnesses

SEM and EDX Properties

The microscopic surface of the friction surface impacts the friction performance of brake pads (Zhang *et al.* 2020). In general, the friction surface includes tribofilm mechanisms with peeling pits that show the friction surface changes after brake tests (Zhang *et al.* 2019). Due to the reaction products that arise on the friction surface, the components making up the friction surface are complicated (Zhang *et al.* 2020). Therefore, only the prominent peaks are plotted on the left-hand EDX plots in Fig. 10. The friction surfaces in Fig. 10 obtained in this study showed a consistent appearance with the wear rate. Secondary plateaus in the BCA9 sample have a larger area than secondary plateaus in other samples. The higher wear rate of the sample may be the cause of the sizable secondary plateau area. Because of the high primary plateaus, the contact surface grows, potentially contributing to the high coefficient of friction. No micro cracks were found in SEM micrographs.

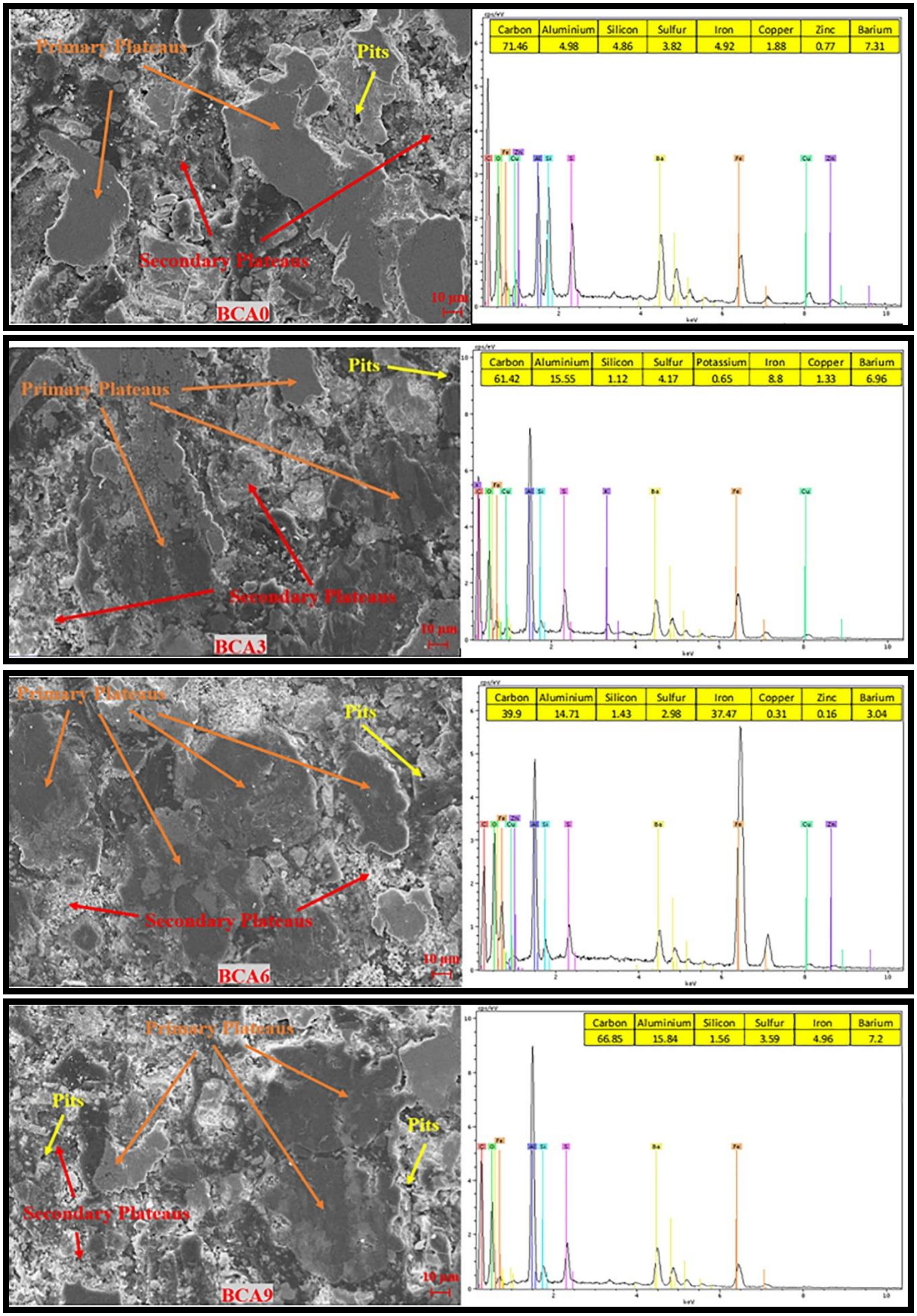

Fig. 10. SEM and EDX images of biocomposites

The absence of microcracks may indicate that the compounds that make up the biocomposite were compatible. From the EDX analyses shown on the left in Fig.10, it can be concluded that the compounds that make up the biocomposite achieved a homogeneous mixture.

Coefficient of Variance of Brake Pad Biocomposites

The coefficient of variation is a measure of distribution that does not depend on unit scales, which allows comparison of experimental results involving different variables. A low coefficient of variation indicates a low degree of uncertainty in the random variable and a high degree of uncertainty in the random variable. It is recommended that the coefficient of variation be below 30% in engineering studies (Romano *et al.* 2005; Zaharia *et al.* 2017). The coefficients of variation are shown in Table 2. The coefficient of variation for all traits was below 30%, indicating a suitably low degree of uncertainty.

Table 2. Coefficient of Variation of Brake Pad Biocomposites

Sample Code	Coefficient of Friction (μ)			Wear Rate (X10^{-7} cm^3/Nm)			Hardness (Shore D)		
	M	s	ρ %	M	s	ρ %	M	s	ρ %
BCA0	0.34	0.060	17.65	0.090	0.024	26.66	95.87	0.954	1
BCA3	0.39	0.087	22.31	0.124	0.022	17.74	95.07	0.863	0.91
BCA6	0.35	0.071	20.29	0.160	0.025	15.63	94.67	1.305	1.4
BCA9	0.33	0.060	18.18	0.197	0.033	16.75	94.53	1.407	1.5

M: Mean, s: Standard deviation, ρ: Coefficient of variation

Performance Ranking of Brake Pad Biocomposites by TOPSIS Method

The Technique for Order of Preference by Similarity to Ideal Solution (TOPSIS) technique was used to assess the performance of brake pad biocomposites. Table 3 displays the matrix for the TOPSIS application. The matrix was formed by considering three properties: coefficient of friction, wear rate, and hardness.

Table 3. Response Matrix for TOPSIS Method

Criteria / **Sample**	**Coefficient of friction**	**Wear rate (X10^{-7} cm^3/Nm)**	**Hardness (Shore D)**
BCA0	0.34	0.090	95.87
BCA3	0.39	0.124	95.07
BCA6	0.35	0.160	94.60
BCA9	0.33	0.197	94.53

The ranking was made according to the normalized evaluation given in Table 4. The highest normalized value indicates the optimum brake pad biocomposite. The results showed that BCA3 had the best performance among all brake pad biocomposites. Afterward, BCA0, BCA6, and BCA9 samples gave successively the next best performance values.

Table 4. Performance Ranking of Biocomposites

Biocomposites	**BCA0**	**BCA3**	**BCA6**	**BCA9**
Performance score	2	1	3	4
Performance ranking	0.678	0.737	0.343	0.000

CONCLUSIONS

1. In this study, 0 to 3 to 6 to 9% blue *Cupressus arizonica* (BCA) cone-added brake pad biocomposites were developed. The brake pad biocomposites containing BCA cones had the best friction coefficient and wear rate.
2. In the performance evaluation made with the TOPSIS method, the best performance was obtained in BCA3, BCA0, BCA6, and BCA9 samples. The BCA3 sample produced the highest coefficient of friction and lowest wear rate. A homogeneous mixture was formed in the EDX analyses of the samples, and the primary and secondary contact surfaces and pits were detected in the SEM analyses. It was concluded that the secondary plateau area is high in the BCA9 sample. As a result, more wear may be prevalent in this sample.
3. As a result of the experiments, it was determined that the brake pad samples developed with BCA cones meet the desired specifications in this sector and can be used in vehicles.
4. Using BCA cones in industrial studies will also positively affect environmental effects. The ability to grow BCA trees in different parts of the world will also provide a socioeconomic advantage in the brake lining industry.

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