Dimensional stability of brake pads reinforced with calcined eggshell particles under exposed to engine lubricating oil

Sunardi Sunardi[1,3], *Dody* Ariawan[1*], *Eko* Surojo[1], *Aditya Rio* Prabowo[1], *Cahyo Hadi* Wibowo[1], *Hammar Ilham* Akbar[2], *Agung* Sudrajad[3], *Harjo* Seputro[4]

[1]Department of Mechanical Engineering, Universitas Sebelas Maret, Surakarta, Indonesia
[2]Department of Mechanical Engineering, Vocational School, Universitas Sebelas Maret, Surakarta, Indonesia
[3]Department of Mechanical Engineering, Universitas Sultan Ageng Tirtayasa, Cilegon, Indonesia
[4]Department of Mechanical Engineering, Universitas 17 Agustus 1945, Surabaya, Indonesia

Abstract. Exposure to a humid environment can affect the behavior of brake lining composites. Considering the vital function of brake linings in controlling vehicle speed, environmental influences must receive serious attention. Exposure to lubricating oil that sticks to the brake lining can cause changes in performance during work. This study will investigate the dimensional stability of brake pad composites when exposed to engine oil and understand the physical behavior of material to facilitate appropriate handling. The brake pads in this study comprised 35% phenolic resin, 10% graphite, 10% bamboo fiber, 10% alumina powder, 5% zinc powder, 5% bamboo particles, and 25% eggshell particles. The manufacturing of brake linings involves three process stages: cold compaction, hot compaction, and post-curing. The samples were soaked in Federal Ultratec API SJ JASO MA engine lubricating oil for 12, 24, 36, and 48 hours. The results showed that the immersion time of the composite in lubricating oil did not significantly affect the dimensional stability of the composite. This result shows that the addition of calcined eggshell particles can improve the material's resistance to engine lubricating oil.

1 Background

Exposure to a humid environment can alter the behavior of brake lining composite materials [1]. The presence of water, which the composite is exposed to, reduces its mechanical properties [2]. Water molecules that permeate the composite over time can disrupt the interfacial bonds within the composite [3]. The hydrolysis reaction of epoxy composites at specific temperatures leads to cracks and interface failure [4].

The size of the water molecules that diffuse into the composite is affected by the type of filler, the volume fraction of the filler, and the soaking time of the composite. Fillers that react with water tend to have a high-water absorption percentage [5]. Another study demonstrates that the higher the filler volume fraction, the greater the water absorption percentage [6]. Very fine filler particles can lead to agglomeration, which may damage the interfacial bond between the fiber and the matrix [3].

The water absorption percentage of a composite is directly related to its dimensional stability. Research by Chiang et al. [7] indicates that the volume fraction, filler size, and soaking time affect the composite's thickness swelling. The rate of increase in composite thickness is higher with a higher percentage of water absorption [8]. rise in water absorption percentage is attributed to the fiber content in the composite.

When brake pads operate, they are often exposed to water and engine lubricating oil. This exposure to oil can affect the dimensional stability of the brake pad composite, which is important for the precision of the brake pads when they are installed in a vehicle's braking system. Understanding of how composites behave when exposed to lubricating oil is still limited, making this study particularly important. One way to improve the resistance of composites to oil and grease is by using thermoplastic as a matrix [9]. Another effective strategy is the use of a hybrid filler made of copper particles and glass powder, which can significantly enhance the composite's resistance to oil penetration. Risyuma and Fitri [10] have shown that composites reinforced with bamboo fiber and coconut fiber, and containing 5% copper powder and 15% glass powder, exhibited the lowest oil absorption.

In a literature review conducted by Sunardi et al. [11] it was revealed that eggshell particles have promising potential as friction materials in the automotive field. In a subsequent study, Sunardi et al. [12] further demonstrated that brake lining composites using eggshell particles achieved optimal values when the eggshell particles were calcined at a temperature of 900°C for 2 hours. This innovative use of eggshells is part of our efforts to develop materials that are not only environmentally friendly but also capable of absorbing gas emissions [13].

* Corresponding author: dodyariawan@staff.uns.ac.id

This study proposes that the addition of calcined eggshell particles can enhance dimensional stability when the material is exposed to engine oil, enabling proper handling. Consequently, there is no reduction in brake pad performance efficiency.

In this study, we will discuss the dimensional stability of brake lining composites exposed to engine lubricating oil. This is a topic of significant practical relevance given the real conditions where brake linings are in an oily environment.

2 Materials and methods

In this study, we will be developing eco-friendly brake lining composite materials. Organic materials, such as bamboo and eggshell particles, will be utilized. Bamboo will serve as reinforcement and filler for the composite, while eggshell particles will act as a filler to enhance the dimensional stability of the composite.

2.1 Materials

The brake linings are made up of various materials, each serving a specific purpose: (1) matrix: phenolic formaldehyde resin, (2) reinforcement: short bamboo fiber, (3) friction modifier: alumina, zinc, (4) solid lubricant: graphite, and (5) filler: bamboo particles, eggshell particles).

The eggshell particles used are obtained from household waste, especially from the brown laying hen variety widely found on the market, as shown in Figure 1. The eggshells are crushed until they reach a mesh size of 200 and then subjected to a classification process. The eggshell particles are then heated in an electric furnace at a temperature of 900°C for 2 hours [12].

Fig. 1. Raw eggshell obtained from household waste.

During this calcination process, a phase change from $CaCO_3$ to CaO occurs, which can be expressed chemically as shown in equation (1). The resulting calcium oxide phase is highly reactive when in contact with water, as demonstrated by equation (2).

$$CaCO_3 \rightarrow CaO + CO \quad (1)$$
$$CaO + H_2O \rightarrow Ca(OH)_2 \quad (2)$$

2.2 Manufacturing of composites

The composite mixing process consists of two stages: (1) the powdered material is mixed in a two-blade mixer machine for 2 minutes, and (2) fibre is then added and stirred for an additional 2 minutes [12, 14]. Fig. 2 illustrates the flowchart of the study regarding the dimensional stability of composites.

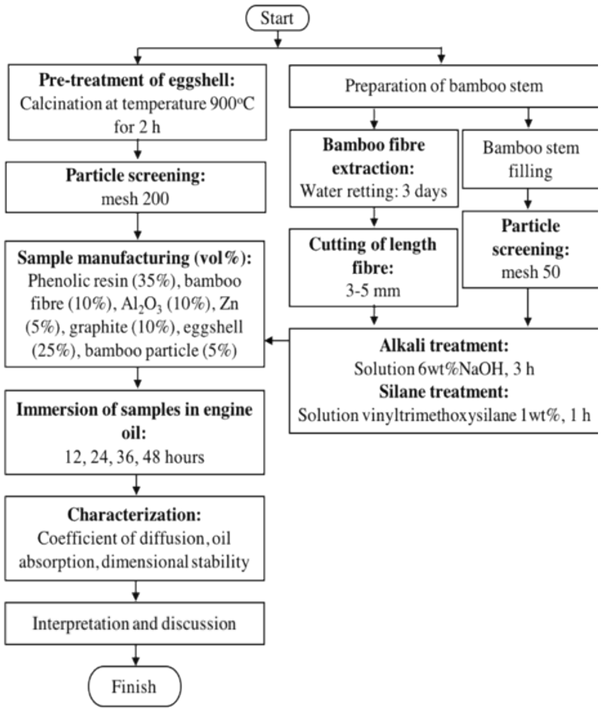

Fig. 2. Flowchart of composite dimensional stability testing.

The sample-making process involves cold-compaction, hot-compaction, and post-curing. Figure 3 illustrates the composite compaction process under both cold and hot compaction conditions. Cold-compaction is performed to create a green body. During cold-compaction, the pressure and holding time are 140 MPa for 5 minutes.

Fig. 3. Manufacturing of brake pads composite.

The hot-compaction is carried out at a temperature of 150°C and held for 10 minutes, with five gas releases in the first minute [12]. The next step is curing, which involves heating the sample continuously for 7.5 hours from ambient temperature to 180°C. After the curing process, the electric oven is opened, and the samples are allowed to cool under the influence of outside air.

2.3 Materials characterization

To evaluate the dimensional stability of the composite, we immersed it in engine lubricating oil for durations of 12, 24, 36, and 48 hours. The volume of lubricating oil used for immersion was 500 ml. The lubricating oil utilized in this study is Federal Ultratec 20W-50, which complies with API SJ and JASO MA standards, as illustrated in Figure 4. To predict the dimensional stability of composites, one needs to have knowledge of the diffusion coefficient and water absorption percentage.

Fig. 4. Lubricating oil for immersion media.

2.3.1 Coefficient of Diffusion

The diffusion coefficient (D) is the amount of substance that can diffuse into the composite and is expressed in units of cm^2/s. Equation (3) is used to calculate the magnitude of the fluid diffusion coefficient into the composite [15].

$$D = \pi \left(\frac{h}{4M_\infty}\right)^2 \left(\frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}}\right)^2 \quad (3)$$

Where M_1 and M_2 represent the weight of the sample at times t_1 and t_2, h is the thickness of the sample, and M_∞ is the weight of the sample at saturation.

Understanding the diffusion coefficient in the composite is crucial as it helps estimate the rate and amount of liquid entering the composite. The liquid that diffuses can potentially harm the bond between the matrix and the fibres and particles. This damage to the composite's bond can negatively impact the dimensional stability of material. A higher diffusion coefficient leads to a greater oil absorption percentage, which in turn reduces the dimensional stability of the composite.

2.3.2 Oil absorption

Water absorption measurements were performed according to ASTM D-570. The samples were placed in lubricating oil at room temperature and the amount of absorbed oil was determined by weighing the samples before and after 24 hours of immersion. The scales used had an accuracy of 0.001 g, and data collection was conducted by replicating four samples. The percentage of lubricating oil absorbed was calculated using Equation (4):

$$DW(\%) = \left[\frac{(W_2 - W_1)}{W_1}\right] x\ 100\% \quad (4)$$

Where W1 and W2 represent the weight of the specimen when dry and wet, respectively.

2.3.3 Dimensional stability

Dimensional stability is assessed using the volumetric swelling coefficient (VSC), which indicates the percentage change in material volume when exposed to lubricating oil. A smaller VSC value indicates higher dimensional stability of the composite material. The VSC is calculated using Equation (3):

$$DVSC = \left[\frac{(V_2 - V_1)}{V_1}\right] x\ 100\% \quad (5)$$

3 Results and discussions

During the experiment, the composite samples were submerged in engine lubricating oil for durations of 12, 24, 36, and 48 hours. Observations were made regarding any changes in the weight and dimensions of the composite. These observations can provide insight into the extent of oil absorption and its impact on the dimensional stability of the composite.

3.1 Coefficient of diffusion

Based on Equation (3), it is determined that the composite diffusion coefficient is 0.180 x 10^{-6} mm^2/sec. Kruk et al. [15] showed that the diffusion coefficients for various vegetable oils are in the range between 8.05 x 10^{-6} and 1.09 x 10^{-5} mm^2/sec. This low diffusion value is affected by the engine oil viscosity, with a kinematic viscosity of 150.08 cSt at 40°C. Viscosity is the result of the microscopic movement of molecules in a liquid. The translational diffusion coefficient is inversely proportional to the viscosity of the liquid.

The diffusion coefficient of lubricating oil is 200 times lower than that of water [15]. This indicates that the viscosity of lubricating oil is higher than that of water, resulting in slower diffusion into the composite. Additionally, lubricating oils with additives and slightly acidic substances can further slow-down the rate of fluid diffusion into the composite sample.

Engine lubricating oil, with its low absorption capacity, has a very low diffusion coefficient.

Composites benefit from this by reducing oil absorption and increasing dimensional stability.

3.2 Oil absorption

The absorption of lubricating oil into the composite continues to increase over 48 hours of soaking, as shown by Figure 5. The change in the rate of oil diffusion into the composite is relatively low due to the higher viscosity of the lubricating oil. These findings are consistent with the study conducted by Rosdi et al. [16].

The percentage of oil absorption is influenced by the immersion time, with longer immersion times resulting in higher absorption percentages. These results are in line with studies by Rajeshkumar et al. [17]. The composite gradually absorbs water molecules through pores or microdefects, which provide a pathway for the liquid to diffuse. Imperfections in the bonding between the matrix and fibers or particles can also allow water molecules to diffuse into the composite. The absorption of water will cease when equilibrium is reached.

Gbadeyan et al. [18] and Sevinc and Durgun [19] demonstrated that increasing eggshell content significantly reduces water absorption. The reduction in water absorption is due to the hydrophobic properties of the eggshell particles. The filler creates interconnecting bonds, the shell has relatively high carbon content, and the shell particles are more evenly distributed, leading to stronger bonds that resist water penetration. This condition shows that exposing the composite to engine lubricating oil will result in reduced oil absorption.

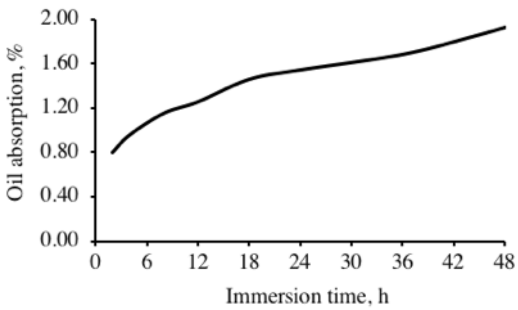

Fig. 5. The percentage of oil absorption along the time of composite immersion.

However, the study by Panchal et al. [20] found that higher eggshell content leads to higher water absorption behavior. The water absorption can increase due to the presence of amine and carboxylate functional groups in eggshell particles.

The study explores the use of organic materials, such as particles and bamboo fibres, for creating organic brake linings. Generally, organic materials are hydrophilic, meaning they have a high-water absorption percentage. One approach to reduce this is through alkali treatment, which can lower the hydrophobic properties of bamboo particles and fibres. Research has demonstrated that untreated bamboo can absorb up to 51% of water due to poor wettability, causing defects and debonding.

The composite has lower absorption of lubricating oil compared to distilled water or rainwater, largely due to the formation of a polymer layer by phenolic resin, creating a barrier to oil impregnation. Additionally, a hard-curing rate, involving conditions of 125°C for 120 minutes, can reduce the oil absorption percentage compared to a soft curing at 125°C for 90 minutes.

Engine lubricating oil comprises oil and additive materials. The oil can create a waterproof layer on the composite surface, while additives increase cohesion between particles, thereby reducing porosity. As a result, the oil absorption percentage when immersed in lubricating oil is lower.

3.3 Volumetric swelling

The detailed volumetric swelling of the composite over time due to exposure to lubricating oil is illustrated in Figure 6. The volume swelling increased steadily over time during the first 36 hours of immersion. However, after 36 hours, there was a sudden significant increase, and then the system experienced saturation. This suggests that composite interface cracks are likely to start forming after 36 hours. The composite material experiences volumetric expansion due to the diffusion of water molecule, which creates internal stress and can lead to cracks [21].

After 36 hours of exposure to lubricating oil, the sample exhibited volumetric expansion, followed by reaching an asymptotic state after 44 hours of immersion in engine lubricating oil. The presence of bamboo fibers and particles containing cellulose and hydroxyl groups contributed to the amount of air absorption [22]. Furthermore, the presence of eggshell particles acts as an inhibitor of air absorption in the composite.

Composites exposed to engine lubricating oil had the lowest volumetric swelling, indicating that CaO is not reactive to lubricating oil, and thus no chemical reaction occurs [23]. After coming into contact with water, CaO can cause cement hydration or lead to the formation of Ca(OH)2. This is supported by SEM-EDS photos, which indicate that no new phase is formed when the composite sample is submerged in engine lubricating oil, as depicted in Figure 8.

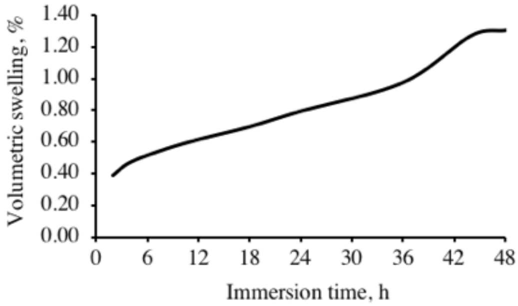

Fig. 6. The volumetric swelling coefficient along the time of composite immersion.

The volumetric swelling coefficient is closely linked to the percentage of water or lubricating oil absorbed into the brake lining composite. As the percentage of fluid absorption increases, the volumetric swelling coefficient of the composite also increases. The absorption rate of engine lubricating oil into the composite is the lowest when compared to water absorption. This is due to the high kinematic viscosity of the lubricating oil at ambient temperature, which is 154.79 cSt, a value higher than the viscosity of water (0.6579 cSt).

Correlation is a statistical method used to determine the strength and direction of correlation between two continuous variables. The correlation coefficient "r" obtained shows the magnitude of the correlation between variables. In Figure 7, the correlation between the percentage of water absorption and the volumetric swelling of the phenol polymer composite is illustrated. The higher the percentage of liquid absorption, the volumetric swelling coefficient also increases. The correlation between these two parameters is 0.9206. The results of this study align with those conducted by Masoodi and Pillai [22], indicating that the longer the composite was immersed, the higher the percentage of water absorption and thickness swelling.

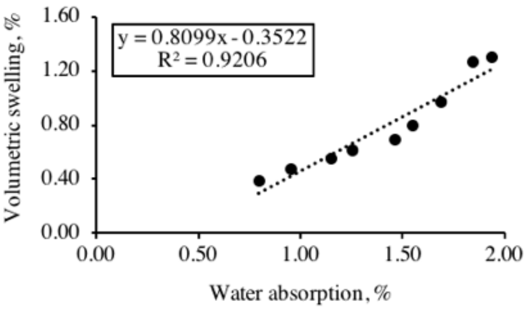

Fig. 7. The correlation between oil absorption and volumetric swelling coefficient.

Mukaka [24] shows that a correlation value of 0.70-0.90 indicates a high correlation, while 0.90-1.00 indicates a very high correlation. The correlation coefficient obtained has a positive value, indicating a unidirectional relationship between the two variables: if the water absorption is higher, the thickness and volumetric swelling will also increase.

The penetration of oil into the composite is influenced by the duration of immersion. Small molecules like gasoline, oil, and acid that permeate the epoxy resin can alter the mechanical properties of the composite [25]. The dimensional stability of the composite is connected to the qualities of the solution in contact with it. NaOH solution causes greater water absorption and thickness expansion compared to acetic acid solution [26].

3.4 Surface morphology

The significant increase in water absorption and volumetric expansion is due to the presence of porosity in the composite. Figure 8 also does not indicate any cement hydration phenomenon, confirming that there is no chemical reaction between CaO and the lubricating oil.

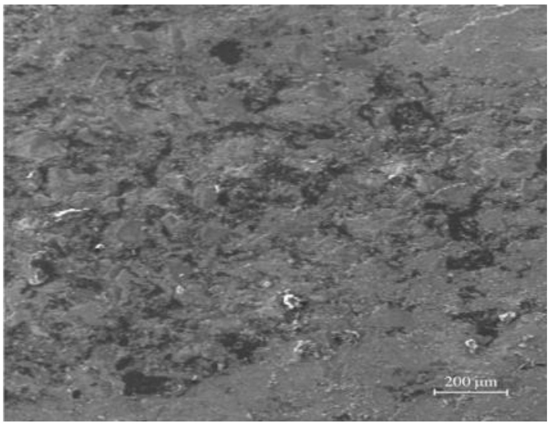

Fig. 8. Surface morphology of the sample after 36 hours of immersion.

4 Conclusion

Eggshell particles have a positive effect on brake lining composites that come into contact with engine lubricating oil. The study's findings that the oil absorption and volumetric swelling of the composite increase as the immersion time in lubricating oil lengthens. There is a strong correlation between engine lubricating oil absorption and composite dimensional stability. The greater the absorption of lubricating oil into the composite, the lower the dimensional stability. The incorporation of chicken eggshell particles into the brake pad composite can enhance its dimensional stability when exposed to engine lubricating oil. This study can help in selecting brake pad materials based on different environmental conditions. Further research can involve exposing the brake pads to rainwater or seawater, as these are abundant in Indonesia. Testing the dimensional stability of the brake pads when exposed to heat through identifying the material's coefficient of expansion.

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