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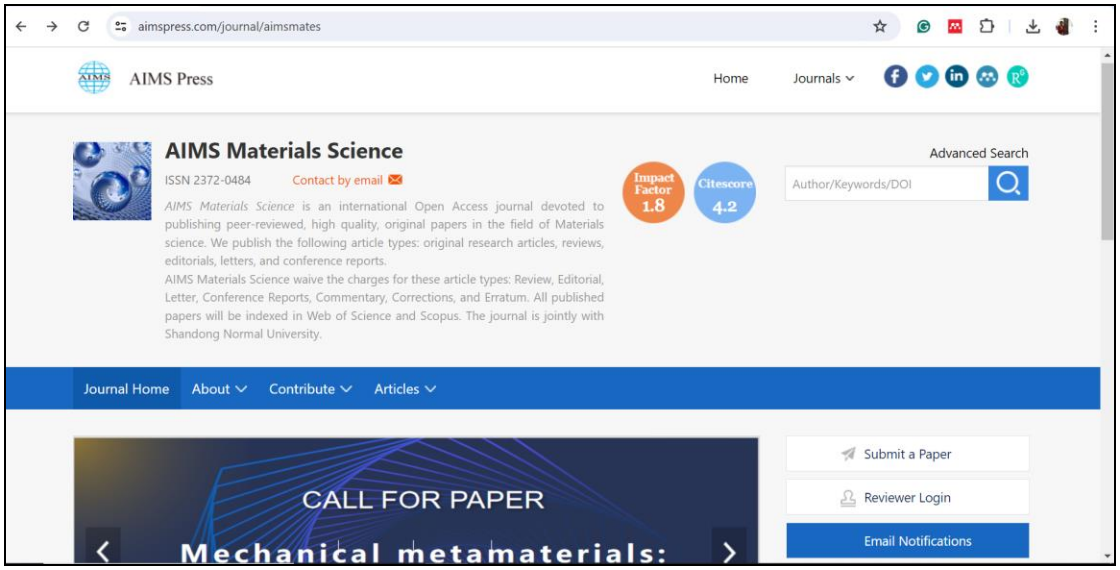

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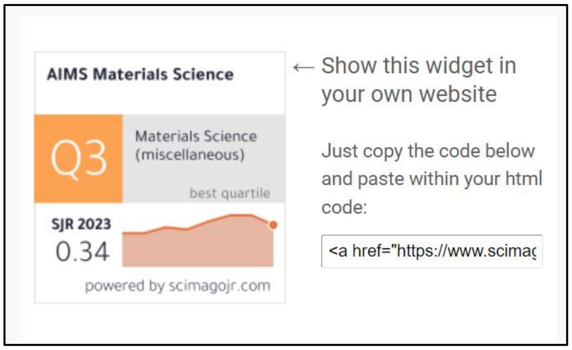

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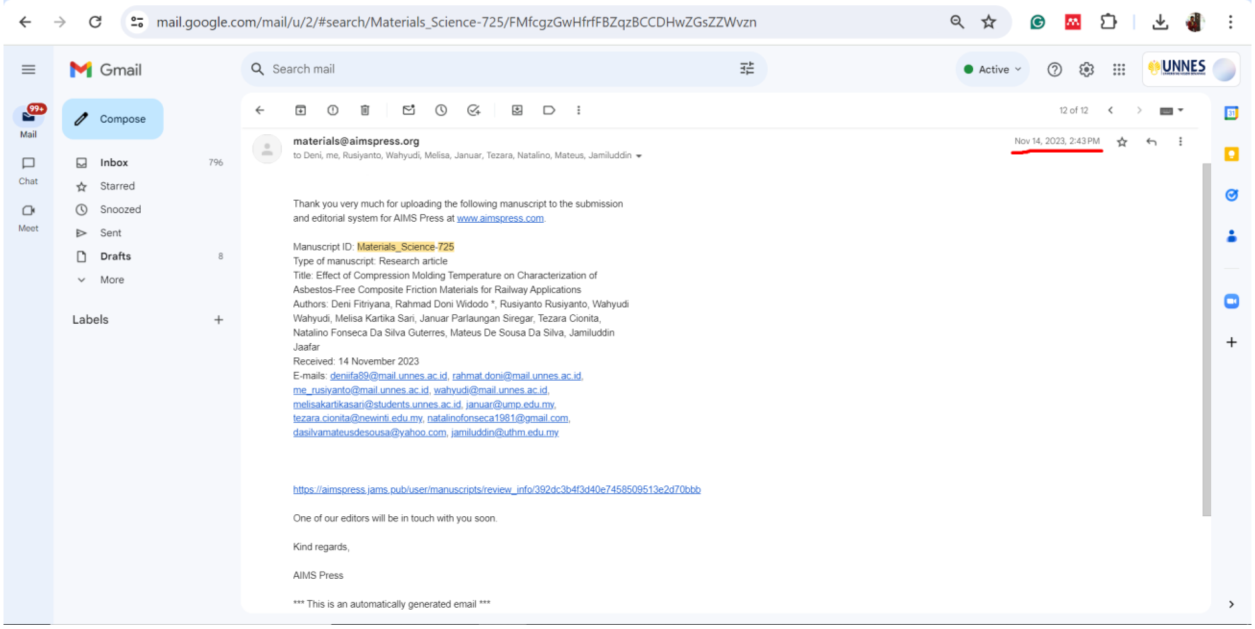

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A. Cover letter
B. Graphical Abstract
C. Draft artikel atau manuskrip

A. Cover letter

Dear Editor in-Chief,

On behalf of the authors, I would like to submit our manuscript entitled "Effect of Compression Molding Temperature on Characterization of Asbestos-Free Composite Friction Materials for Railway Applications" for publication on the **AIMS Materials Science**. We believe this research article would be a special of interest to the reader of the **AIMS Materials Science**.

This study reports a comparative study on the fabrication and characterization of non-asbestos brake pads for railways applications using hot compression molding method. Brake pads significantly affect the braking performance of railways under both normal and emergency operating conditions. In previous studies, brake pads were made using the hand lay-up method and produced the best properties on specimens with epoxy, rice husk, Al_2O_3, and Fe_2O_3 compositions of 50%, 20%, 15%, and 15%. However, the resulting density does not meet the density standard set by PT Industri Kereta Api Indonesia (PT INKA), which is 1.7 - 2.4 gr/cm^3. To date, there has been limited research into the utilization of the compression hot molding method for the production of asbestos-free composite friction materials composed of epoxy, rice husk, Al_2O_3, and Fe_2O_3 for railway applications. This study aimed to determine the effect of compression molding temperature on the characterization of composite brake pads for railway applications. The brake pad specimens were made of epoxy resin, rice husk, Al_2O_3 and Fe_2O_3 with a composition of 50%, 20%, 15% and 15%, respectively. The manufacture of composites in this study used the compression molding method with a pressure of 20 MPa for 15 minutes holding time. The mold temperature used were 80°C, 100°C, 120°C. Density, hardness, tensile, wear, TGA (Thermal Gravimetric Analysis), and DSC (Differential Scanning Calorimetry) tests were performed to evaluate the properties of the specimens obtained. The results demonstrated that an increase in molding temperature improved the characterization of the brake pads, with the best results attained at 120°C (SP-3 specimen). SP-3 specimens had the best density, hardness, tensile properties, and thermal properties compared to other specimens.

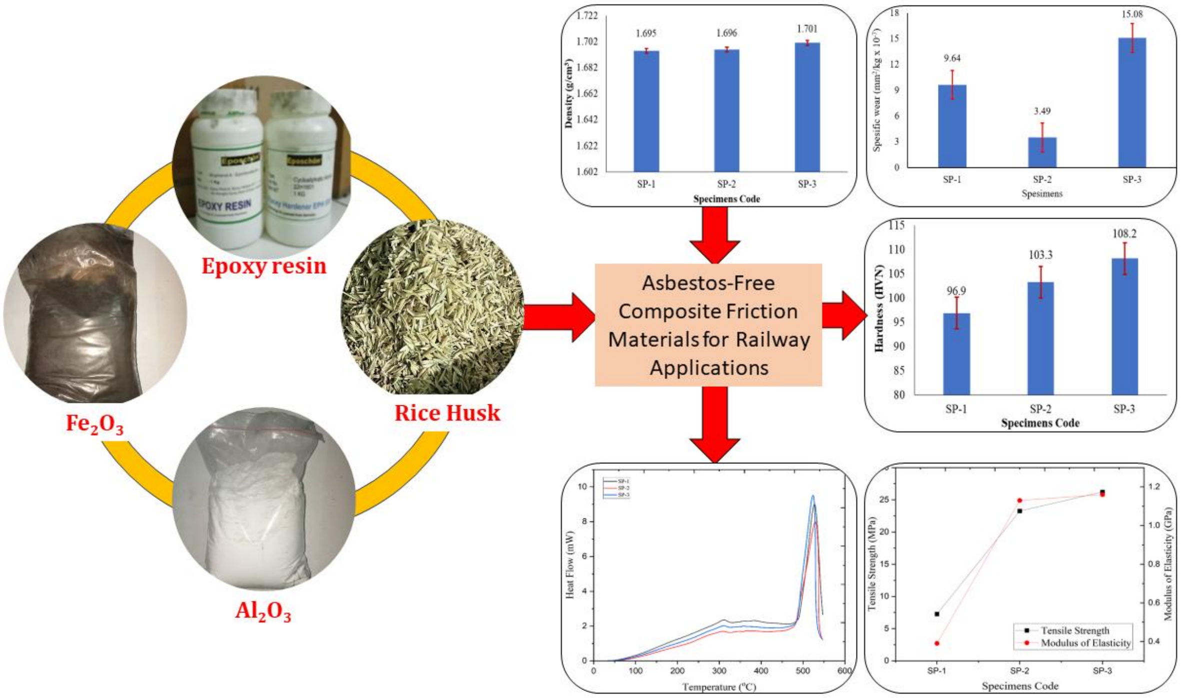
To the best of our knowledge, non-asbestos brake pads for railway application made from epoxy, rice husk, Al_2O_3, and Fe_2O_3 prepared by compression molding is still very limited. Therefore, this article is expected to contribute to enriching knowledge in the field of research on the manufacture and characterization of non-asbestos brake pads, especially in for railway application produced by the compression molding method. On this letter, we would like to declare that this research paper has never been published, and not under consideration for publication in elsewhere. We hope you will consider our article for publication on your esteemed Journal. We are looking forward to hearing your kind reply.

Thank you.

Best wishes and regards,

Dr. Rahmat Doni Widodo

B. Graphical Abstract



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Effect of Compression Molding Temperature on Characterization of Asbestos-Free Composite Friction Materials for Railway Applications

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Abstract: Brake pads significantly affect the braking performance of railways under both normal and emergency operating conditions. In previous studies, brake pads were made using the hand lay-up method and produced the best properties on specimens with epoxy, rice husk, Al_2O_3, and Fe_2O_3 compositions of 50%, 20%, 15%, and 15%. However, the resulting density does not meet the density standard set by PT Industri Kereta Api Indonesia (PT INKA), which is 1.7 - 2.4 gr/cm^3. To date, there has been limited research into the utilization of the compression hot molding method for the production of asbestos-free composite friction materials composed of epoxy, rice husk, Al_2O_3, and Fe_2O_3 for railway applications. This study aimed to determine the effect of compression molding temperature on the characterization of composite brake pads for railway applications. The brake pad specimens were made of epoxy resin, rice husk, Al_2O_3 and Fe_2O_3 with a composition of 50%, 20%, 15% and 15%, respectively. The manufacture of composites in this study used the compression molding method with a pressure of 20 MPa for 15 minutes holding time. The mold temperature used were 80°C, 100°C, 120°C.

Density, hardness, tensile, wear, TGA (Thermal Gravimetric Analysis), and DSC (Differential Scanning Calorimetry) tests were performed to evaluate the properties of the specimens obtained. The results demonstrated that an increase in molding temperature improved the characterization of the brake pads, with the best results attained at 120°C (SP-3 specimen). SP-3 specimens had the best density, hardness, tensile properties, and thermal properties compared to other specimens.

Keywords: Brake pads; railways; composites; compression molding; friction materials

1. Introduction

The effectiveness of brake pads has a significant impact on railways' capacity for stopping in an emergency as well as during normal operations [1]. Brake pads are used as components to ensure the safety of the railways during the braking process. Generally, brake pads on trains can be divided into organic and metal. Organic brake pads, which are composed of organic polymers, have been implemented in cars, trains, and other modes of transportation. Metal-based brake pads have emerged as the preferred material for high-speed trains due to their outstanding friction capability, commendable resistance to wear, outstanding thermal conductivity, and ability to withstand high operational temperatures [1,2].

Composite brake pads have been used in Indonesia since the last decade to replace cast iron brake pads for trains. Cast iron brake pads wear out faster when compared to composite brake pads [3]. In addition, metallic brake pads have a very high density which can reduce the energy efficiency of the train system [4]. In recent years, the development of composite technology has made rapid progress with various innovations in the manufacture of brake pads using natural materials that have become waste and are no longer used. Composite itself is a material composed of a mixture of two or more materials with different mechanical properties to produce a new material that has different mechanical properties from its constituent materials. The properties of composite materials are a combination of the properties of its constituents, the matrix and reinforcement or filler. The matrix serves to transfer stress to the fiber, form coherent bonds, protect the fiber and remain stable after the manufacturing process. Reinforcement or filler materials must be able to support or improve the properties of the matrix in fabricating composite materials [5].

In previous studies, brake pads for applications on trains have been successfully made using the hand lay-up method. Brake pads with the best properties were found in specimens with a composition of epoxy, rice husk, Al_2O_3, and Fe_2O_3 of 50%, 20%, 15%, and 15% respectively and a mesh size of 200. This composition produced density, hardness, tensile strength, specific wear value and degradation temperature of 1.23 g/cm^3, 81.2 HVN, 23.34 MPa, 8.67 x 10^{-7} N/mm^2, and 379°C [6]. Meanwhile, when using 100 mesh rice husk, the density, hardness, specific wear value, and degradation temperature of the resulting composite brake friction material were 1.33 g/cm^3, 83.4 HVN, 10.8 x 10^{-7} N/mm^2, and 363.99°C [7]. However, the resulting density does not meet the standard density set by PT. INKA, which is 1.7– 2.4 gr/cm^3 [8].

The hand layup method is a commonly employed technique in the fabrication of composite materials. Typically, the initial cost associated with fabricating composites using the hand layup technique is quite economical. Furthermore, this methodology enables the production of items with diverse geometries, structures, and designs.

Nevertheless, the hand layup technique employed in composite manufacturing exhibits several limitations, including a reduced production speed and a decreased volume proportion of reinforcement. Furthermore, the use of this technique results in an uneven dispersion of reinforcing and matrix substances as a consequence of the inherent imprecision associated with handling by hand. As a result, producing high-quality composites in large quantities using the hand layup method is not possible [9–11].

Based on the description above, another method is needed to meet the characteristic requirements of brake pads for applications on trains. One method of fabricating composites that can produce better characteristics is compression molding. According to the findings of a study by Nyior et al. (2018), compression molding produced composites with better mechanical properties than the manual lay-up (hand lay-up) method. Their study found that the tensile strength and Young's modulus of samples made with compression molding were 77% and 47% higher than samples made by hand lay-up, respectively. The results also showed that the impact strength of the materials made by compression molding (11.5 kJ/m^2) was significantly higher than that of the samples made by hand lay-up, which had an impact strength of 7 kJ/m^2 [12].

In this study, the composite material will be pressed and heated at a certain pressure and temperature. The fabrication of asbestos-free composite friction materials for railway applications made from epoxy, rice husk, Al_2O_3, and Fe_2O_3 by compression hot molding has not been widely studied. The pressure applied can make the composite tighter and denser. While the heating process makes the resin move to flow to fill the empty composite parts. The purpose of this study was to determine the effect of compression molding temperature on the characterization of composite brake pads for applications on railways.

2. Materials and Methods

2.1. Materials

The materials used in this study were epoxy, hardener, rice husk, aluminum oxide (Al_2O_3), and iron oxide (Fe_2O_3). The matrix used in this study was Bisphenol A-Epichlorohydrin epoxy resin as a binder, and Cycloaliphatic Amine type epoxy hardener obtained from the Justus store in Semarang. In general, epoxy has a density, tensile strength, and flexural strength of 1.18 g/cm^3, 63.7 MPa, and 8.3 MPa [13]. The rice husk used in this study was obtained from a rice mill near the campus. The composition of rice husk consists of cellulose (50%), lignin (25%-30%), silica (15%-20%), and the remainder consists of hemicellulose and water content [14]. In this study, Al_2O_3 and Fe_2O_3 were obtained from PT Merck Tbk, Indonesia.

2.2. Specimen Fabrications

In this study, the fabrication of brake pad specimens used the compression molding method. The rice husk was crushed using the FOMAC FCT-Z300 miller machine and sieved using a 200 mesh. Furthermore, the specimens were prepared by mixing the materials according to a predetermined concentration using a hand mixer in a plastic cup in stages. First, mixing epoxy and hardener with a ratio of 1:3 was carried out for 7 minutes. Then, a mixture of rice husk, aluminum oxide, iron oxide was added after being stirred for 5 minutes. Mixing was done again for 10 minutes. After all the ingredients were mixed, they were poured into the mold and left to harden at room temperature for up to 10 hours. Furthermore, the specimen was compressed with a pressure of 20 MPa for 15 minutes and a temperature of 80°C, 100°C and 120°C (Table. 1). After that, the composite specimens were cut and characterized.

Table 1: Composite brake pads specimen code and compression molding setting parameters

Specimen Code	Pressure (MPa)	Holding Time (min.)	Temp (°C)
SP-1	20	15	80
SP-2	20	15	100
SP-3	20	15	120

2.3. Testing and Characterizations

In this study, the tests carried out were density, hardness, tensile, wear using the Ogoshi method, TGA (Thermal Gravimetric Analysis), and DSC (Differential Scanning Calorimetry). Density testing was based on ASTM D792 standard. Density testing was performed using an electronic density meter DME 220 series from Vibra Canada Inc. (Mississauga, ON, USA). This test is carried out by weighing the dry mass and the mass of the test object in water (wet mass). Vickers hardness testing in this study refers to the testing method carried out by [6,15]. Hardness testing was carried out using a Microhardness Tester F-800 machine (Future-Tech Corp., Kanagawa, Japan) with a test load of 25 gf and a dwell time of 10 seconds. Tensile testing was done according to the ASTM D638 standard using a HT-2402 Computer Servo Control Material Testing Machines from Hung Ta Instrument Co., Ltd., Samutprakarn, Thailand. The tensile test results consist of max force and elongation, which will be used to calculate the tensile strength and tensile modulus. Wear testing was performed using the Oghosi High Speed Universal Wear Testing Machine (Type OAT-U). The Ogoshi wear test was carried out with the width of the wear plate (B) = 3 mm, the radius of the wear plate (r) = 13.06 mm, the distance traveled during the wear process (10) = 66.6 m, and the test load (F) = 2.12 kg. The TGA test was carried out based on the ASTM D6370 standard using the NEXTA STA test kit (Hitachi STA200RV with Real View Sample Observation). The temperature used in this test was 30°C – 550°C. The testing process was conducted at a heating rate of 10°C/minute with a 100 ml/minute nitrogen gas flow. The test results will obtain the temperature and weight loss values. The DSC test was carried out based on the ASTM D3418 standard using the NEXTA STA test tool (Hitachi STA200RV with Real View Sample Observation). The temperature used in this test was 30°C – 550°C. The testing process was carried out at a heating rate of 10°C /minute with a nitrogen gas flow of 100 ml/minute. The test results will obtain the temperature and heat flow values of the composite specimens.

3. Results and Discussions

Figure 1 shows the density of composite brake pad specimens with variations in molding temperature. The results of this study indicated that the increase in temperature affected the results of the composite density. The lowest density was shown in the SP-1 specimen, which was 1,695 g/cm^3. While the highest density was shown in the SP-3 specimen, which was 1,701 g/cm^3. The increase in density value occurs due to an increase in the temperature of composite fabrications with the compression molding method which causes a decrease in the viscosity of the resin, making it easier for the polymer to fill voids [16,17].

The fewer voids produced, the greater the density of the brake pad samples [6]. Void content is the percentage by volume of empty spaces or cavities inside composite materials [17]. When there are voids in a composite, they occupy space that would otherwise be occupied by the composite material. As a consequence, the composite's overall density decreases as the total mass is distributed over a larger volume [6,17].

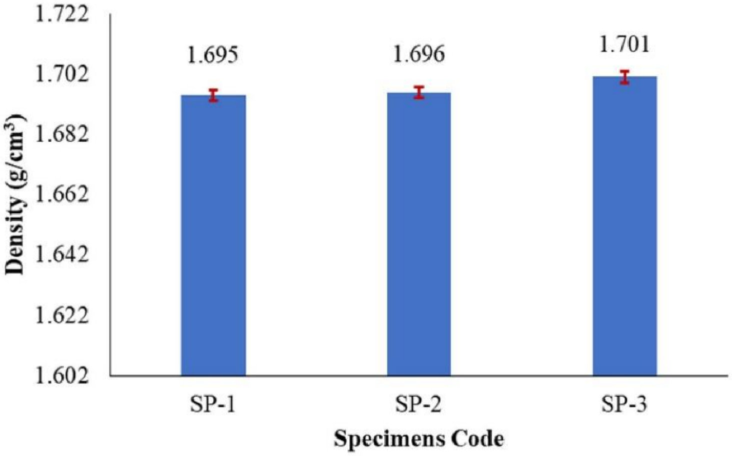

Figure 1: The effect of temperature on the density of the composites

In the manufacture of brake pad specimens using the compression molding method, an increase in molding temperature causes the viscosity of the resin to decrease so that the resin flows more easily and wets the reinforcing material. This results in a better bond between the resin, filler, and reinforcement, resulting in an increase in the density of the composite specimen [6]. This is what causes SP-3 to have a higher density than other composite specimens. Incomplete resin flow into the desiccated area of reinforcement and filler materials can give rise to porosity in composite specimens. This is typically the result of increased resin viscosity due to exposure to ambient conditions and low-temperature curing cycles, resulting in inadequate flow. The lowered molding temperature reduces the driving force of gas-induced cavity growth. So that more porosity is produced, resulting in a decrease in the specimen's mechanical properties [17–20].

The results of this investigation are consistent with Ochi et al.'s (2015) research. Their findings demonstrated a correlation between the density of long bamboo fiber/ PLA Composites and the temperature of the mold. In general, the density of composites increases from 140°C to 160°C as the molding temperature. However, when the molding temperature exceeds 160°C, the density of the composite decreases. This occurs because mold temperatures above 160°C reduce the matrix's viscosity and make it simpler for air to become trapped inside the material during the molding process, resulting in the formation of numerous voids [21]. In another study, Ochi et al. (2022) demonstrated the relationship between the density of bamboo fiber bundle-reinforced bamboo powder composite materials and the molding temperature. As the molding temperature increased, the density of the composite specimens remained relatively constant. The density of composites was between 1.41 and 1.42 g/cm^3 [22].

Based on these research results, only the SP-3 specimen met the minimum density determined by PT INKA, which was 1.7–2.4 gr/cm^3 [8]. Meanwhile, the density of the SP-2 and SP-1 specimens almost met the specified density requirements. In addition, the density obtained in this study was higher than the results of previous studies. In previous research, brake pad composite fabrication used the hand layup method with various compositions. The highest density produced in previous studies was 1.23 g/cm^3 [6], and 1.33 g/cm^3 [7]. Whereas in this study, the densities of the specimens Sp-1, SP-2, and SP-3 were 1,695 g/cm^3, 1,696 g/cm^3, and 1.701 g/cm^3, respectively.

The effect of temperature on the hardness properties of the composite is shown in Figure 2. The lowest hardness value was shown in the SP-1 specimen, which was 96.9 gf/mm^2 (HVN). While the highest density was shown in the SP-3 specimen, which was 108.2 gf/mm^2 (HVN). SP3 and SP1 specimens were the specimens with the highest and lowest densities produced in the study, respectively. In general, the hardness of composite specimens increases in proportion to their density. This is due to the fact that dense materials have strong interfacial bonding's between their matrix and reinforcement, making them more resistant to indentation and plastic deformation [6,23].

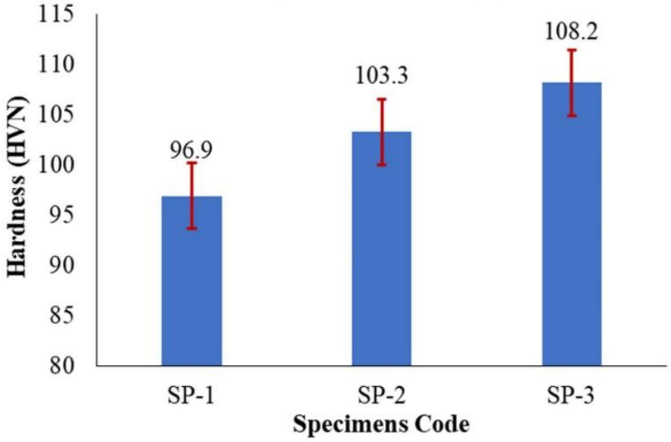

Figure 2: Effect of temperature on composite hardness

Previous research reached the same conclusion, which is that the specimen's higher density increased its hardness [24–27]. Research conducted by Fouly et al. (2021) showed that increasing the density of the composite causes an increase in the hardness of the specimen. Therefore, the average hardness also increases with increasing density. The PMHA8 specimen obtained a maximum hardness of 87.7 D-index. This happened because the PMHA8 specimen had the highest density compared to the other specimens [23]. Furthermore, an increase in the composite is hardness indicates a good interfacial bond between the matrix and the reinforcing fiber. The stronger the interfacial bond between the matrix and the reinforcing fiber, the higher the hardness of the resulting composite specimen [28,29].

The effect of temperature on the tensile strength of the composite is shown in Figure 3. The lowest tensile strength value was shown in the SP-1 specimen, which was 7.26 MPa. While the highest tensile strength was shown in the SP-3 specimen, which was 26.22 MPa. The increase in the value of tensile strength is affected by the behavior of the bond between the resin and fiber interfaces at each increasing compression molding temperature variation [30].

This is in line with research conducted by Sumesh & Kanthavel (2020). Composites with an epoxy resin matrix formed by the compression molding method produce the best mechanical properties of tensile strength at higher temperatures [31]. Heating at high temperatures facilitates the mobilization of the resin in fiber impregnation. The increase in tensile strength is also due to an increase in mold temperature which will reduce the viscosity of the matrix which has an impact on reducing voids in the composite [32].

The increase in tensile strength may also be caused by the main process that occurs at a molding temperature of 80°C-120°C for the evaporation. The higher the molding temperature, the higher the moisture content in the evaporating fiber, therefore the higher the tensile strength. Mvondo et al., (2017), stated that there was a negative relationship between the moisture content of tropical wood fiber and its tensile strength. Differences in the percentage of water content in fiber were studied. In fibers that have a lower water content produces high tensile strength, while high water content produces low tensile strength [33].

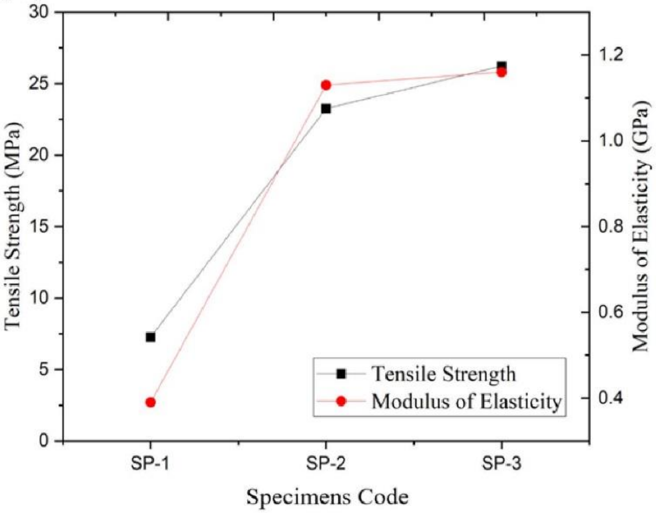

Figure 3: Effect of temperature on tensile strength and tensile modulus of composites

Figure 3 shows that the modulus of elasticity had increased with increasing molding temperature. The modulus of elasticity represents the inflexibility of a material. The higher the value of the modulus of elasticity, the less flexible the material is. At higher molding temperatures, the specimen becomes stiffer so that the modulus value is higher [18,19]. In accordance with research conducted by Ochi et al. (2022) [22], the value of the elastic modulus increased with increasing temperature used in the specimen molding process using the compression molding method.

The wear test results showed that the use of higher mold temperatures can cause a decrease in the wear resistance of the composite (Figure 4). A higher specific wear value indicated a lower wear resistance property. In the study, the SP-3 specimen had the highest specific wear value of 15.08×10^{-7} mm^2/kg while the SP-2 specimen had the lowest specific wear value. So, it can be said that the highest wear resistance was found in the SP-2 specimen. According Günay et al. (2020), a negative correlation exists between a material's hardness value and the specific wear value it exhibits. Materials possessing high hardness exhibit enhanced resistance to wear and tear, hence endowing the test specimens with excellent wear resistance.

The reason for this phenomenon is that the wear value acquired during Ogoshi wear testing exhibits an inverse relationship with the wear resistance qualities of the material being tested. A material's wear resistance qualities are improved when its specific wear value decreases [36]. Nevertheless, this investigation revealed that SP-3 had the highest specific wear value. It can be asserted that SP-3 exhibits the lowest level of wear resistance. This phenomenon can be attributed to the positive correlation between the temperature applied and the surface roughness observed in the composite specimen. The findings of a study conducted by Jan et al. (2020) indicate that elevating the temperature of the mold leads to a corresponding increase in the roughness of the composite material. Their findings indicated that elevating the mold temperature led to a corresponding increase in the surface roughness of the brake pad [37]. The occurrence of increased surface roughness in the specimen can be attributed to the roughness of the composite surface when it comes into contact with the surface of the hard steel disc (used as test equipment). This contact leads to the formation of cracks on both the surface and subsurface of the specimen, which results in matrix delamination or wear [38]. This results in the removal of material in large flakes and creates various irregular edge shapes resulting in higher friction and wear [38].

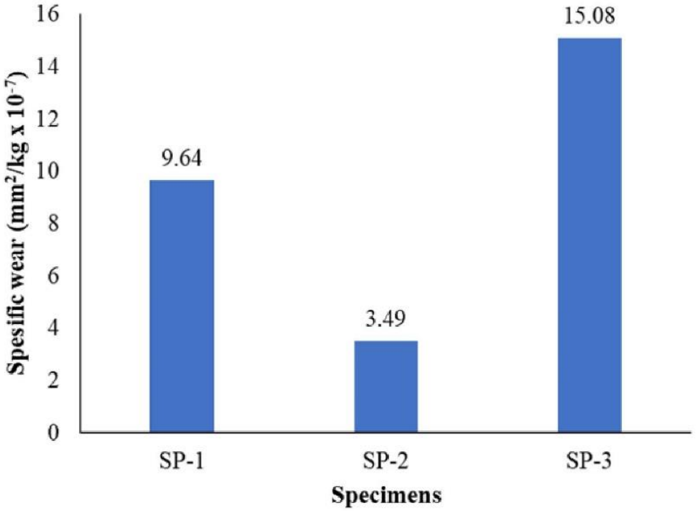

Figure 4: Effect of temperature on the specific wear of the composite

TGA and DSC testing in this study was conducted to determine the thermal properties of the composite. Figure 5 shows that the composite brake pads specimen had several temperature variations during thermal decomposition that occurred in the temperature range of 30°C – 550°C. Weight loss on initial heating (30°C – 200°C) was caused by the evaporation of water on the rice husk. This happened because water was not chemically bound to the fiber. The reduction of fiber mass was further related to the degradation of hemicellulose. In the second range (200°C – 400°C), the weight loss that occurred was mostly due to the decomposition of hemicellulose and cellulose fibers and was followed by the decomposition of epoxy resin. In the last range (400°C – 550°C), the weight loss that occurred was caused by fiber degradation and decomposition of epoxy resin and filler.

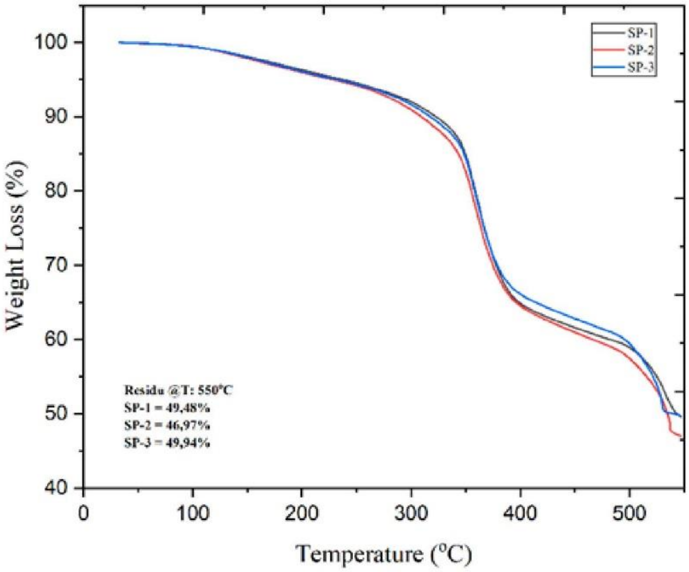

Figure 5: Effect of compression molding temperature on composite weight loss

This is in accordance with research conducted by Chen et al., (2020), rice husk experiences weight loss which is divided into three stages. At a temperature of 35°C – 150°C, rice husk undergoes evaporation of water in the fiber. At temperatures of 150°C – 380°C decomposition of hemicellulose, cellulose and lignin occurs and at temperature stages of 380°C – 600°C fiber degradation occurs [39]. The TGA test with a temperature scale of 30°C – 550°C resulted in a residue of 49.48% in the SP-1 specimen, 46.97% in the SP-2 specimen and 49.94% in the SP-3 specimen. The residual results in this study are in line with research by Li et al., (2020), the residual weight of the composite showed a tendency to increase with increasing molding temperature. The results of the TG curve analysis show that the right molding temperature will help the curing reaction and cross-linking of resin and fiber. The increased crosslink density of the composite will strengthen the thermal stability of the composite [40].

Figure 6 shows the DTG (Derivative Thermal Gravimetry) curve for each composite brake pads specimen that experienced a maximum decomposition phase (Tmax) concerning temperature which was shown at the main peak. SP-1 specimen occurred at 359.98°C, SP-2 specimen at 356.13°C, and SP-3 specimen at 360.94°C. Maximum decomposition is the maximum weight loss on the specimen that occurs at a certain temperature (Tmax) which can be used as the most important indicator in determining the thermal stability of a material [41]. The results showed that there was an effect of the use of specimen molding temperature. The specimen with a molding temperature of 120°C had a larger Tmax and residue than other specimens. According to research by Li et al., (2020), by increasing the molding temperature, the durability of the epoxy resin and fiber activity tends to increase, which benefits the chemical bond between the fiber and the resin which results in increased composite cross-linking so that the degradation temperature will be higher [40].

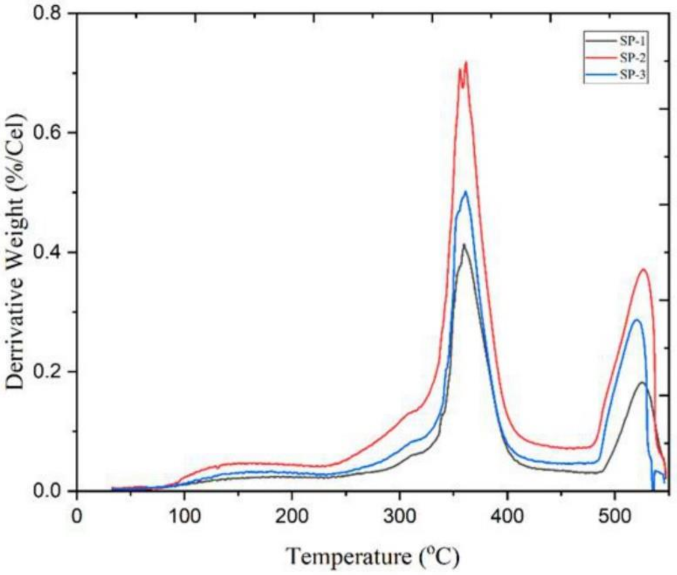

Figure 6: The effect of compression molding temperature on the derivative weight of the composite

Based on Figure 7, the exothermic phase occurred when heated to a temperature of 30°C – 550°C. On the DSC curve, Tg can be known from each specimen. The glass temperature is the transition temperature where the behavior of the composite transitions from hard glass to soft rubber [42]. The SP-1 specimen had a Tg of 311.25°C. The SP-2 specimen had a Tg of 309.18°C. The SP-3 specimen had a Tg of 310.06°C. After passing through the Tg material, each specimen began to form crystals. Specimens will experience cold crystallization; cold crystallization is a unique phenomenon in which crystallization that accompanies an exothermic anomaly occurs when a material is heated to a temperature below its melting point but above the temperature of its glass.

On the curve, the peak point of the curve after going through the Tg phase is Tc. Temperature crystallization is a transition temperature where the formation of a crystal structure occurs due to heating [43]. The SP-1 specimen has the highest Tc of 526.17°C. The SP-2 specimen has the highest Tc of 529.83°C. The SP-3 specimen has the highest Tc of 523.17°C. The three specimens did not experience an endothermic phase, in which the specimens did not melt up to 550°C. On the DSC curve, Tm (Temperature melting) can be observed at the turning point of the curve. This is in line with Li et al., (2020) which stated that the first exothermic peak is closely related to the glass transition. Increased Tg and decreased peaks can be attributed to increased crosslinking and amorphism. The decreasing exothermic peak value of the composite indicates that the crosslink density of the mixture is increasing. It can be concluded that a reasonable increase in molding temperature will be conducive to increasing the crosslink density [40].

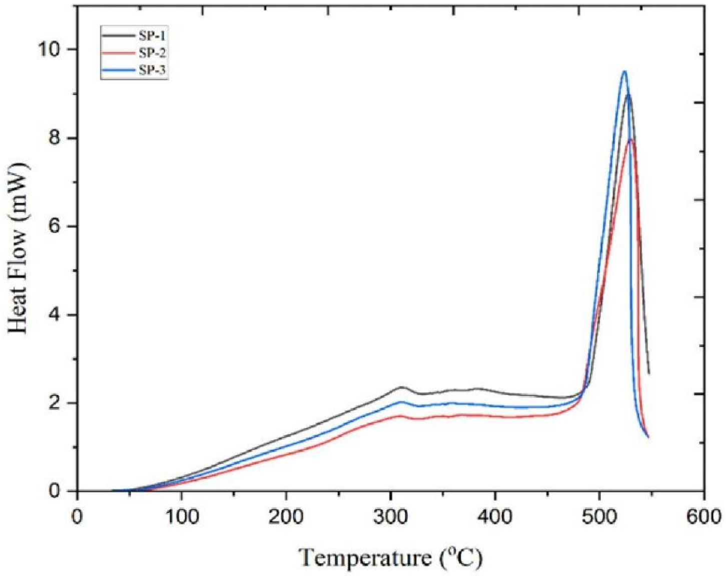

Figure 7: Effect of compression molding temperature on composite heat flow

4. Conclusions

The current study investigated the impact of temperature on the compression molding process on the characterization of composite brake pads. The results demonstrated that an increase in molding temperature improved the characterization of the brake pads, with the best results attained at 120°C (SP-3 specimen). At this temperature, the produced brake pads had densities, Vickers hardness, tensile strengths, and tensile modulus of 1.70 g/cm^3, 108.2 HVN, 26.25 MPa, and 1.16 GPa, respectively. The increasing temperature during the compression molding process caused the viscosity of the resin to decrease so that the resin flows more easily and wets the reinforcing material. This results in a better bond between the resin, filler, and reinforcement, resulting in an increase in the mechanical properties of the composite specimen. Furthermore, the mold temperature in the compression hot molding process has a significant effect on the specific wear on the composite specimen. Specific wear (x 10^{-7} mm^2/kg) on SP-1, SP-2, and SP-3 specimens was 9.64, 3.49, and 15.08, respectively.

The thermal properties of composite brake pads were determined through TGA and DSC testing in this investigation. The study discovered that composite brake pads undergo temperature variations during thermal decomposition, with weight loss occurring between 30°C and 550°C. It is essential for determining thermal stability that the residual weight of the composite increased as the molding temperature increased. The study found that increasing the molding temperature led to an increase in the thermal resistance of the brake pads. The highest thermal properties were generated by brake pads molded at 120°C (SP-3 specimen), with total residues, Tmax, Tg, and Tc values of 49.94%, 360.94°C, 310.06°C, and 517.17°C, respectively. The results of the study indicated that it is possible to produce high-quality composite brake pads that can substitute cast iron brake pads on trains using the compression molding method.

This could contribute to the establishment of a domestic brake pad industry in Indonesia, reducing the country's reliance on imports. In addition, the utilization of locally accessible materials, such as rice husk, could contribute to the Sustainable Development Goals (SDGs) and environmentally responsible brake pad industry in Indonesia.

Use of AI tools declaration

The authors declare they have not used Artificial Intelligence (AI) tools in the creation of this article.

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Conflict of Interest

The authors declare no conflict of interest

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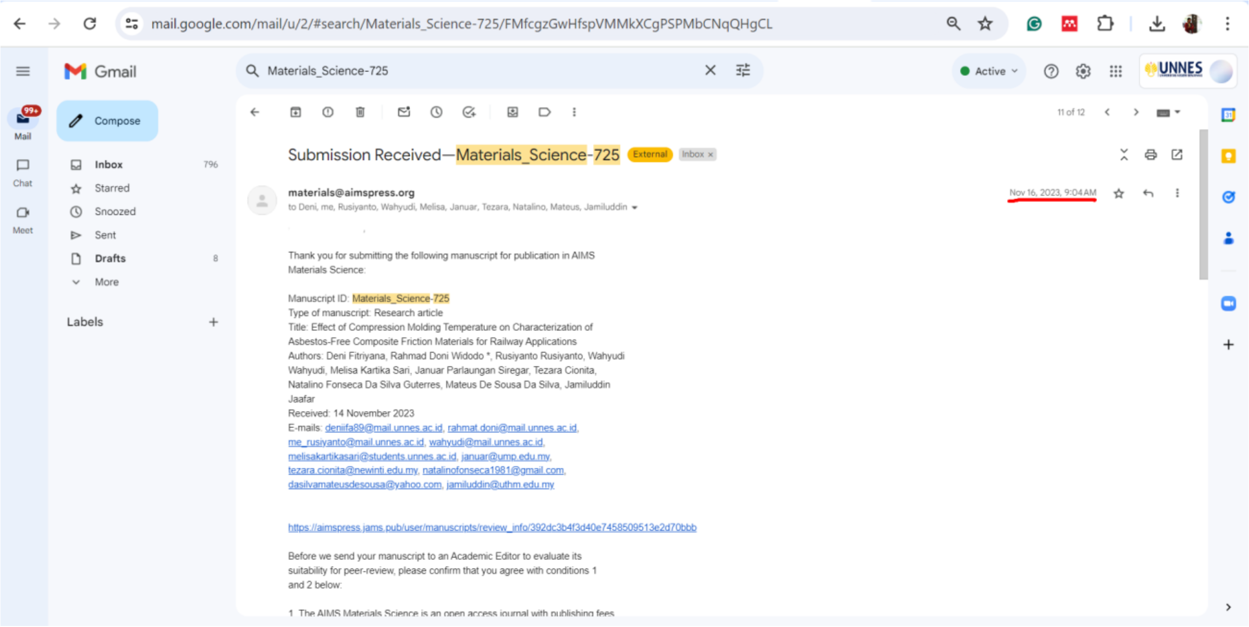

Submission Received—Materials_Science-725

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Manuscript ID: Materials_Science-725
Type of manuscript: Research article
Title: Effect of Compression Molding Temperature on Characterization of Asbestos-Free Composite Friction Materials for Railway Applications
Authors: Deni Fitriyana, Rahmad Doni Widodo *, Rusiyanto Rusiyanto, Wahyudi Wahyudi, Melisa Kartika Sari, Januar Parlaungan Siregar, Tezara Cionita, Natalino Fonseca Da Silva Guterres, Mateus De Sousa Da Silva, Jamiluddin Jaafar
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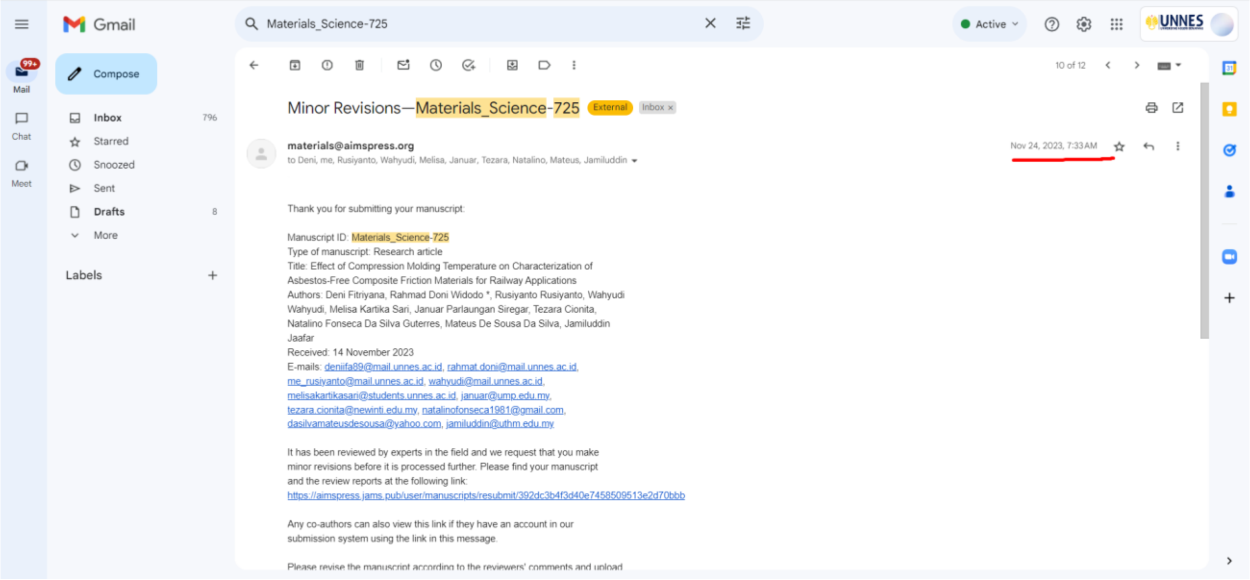

Minor Revisions—Materials_Science-725

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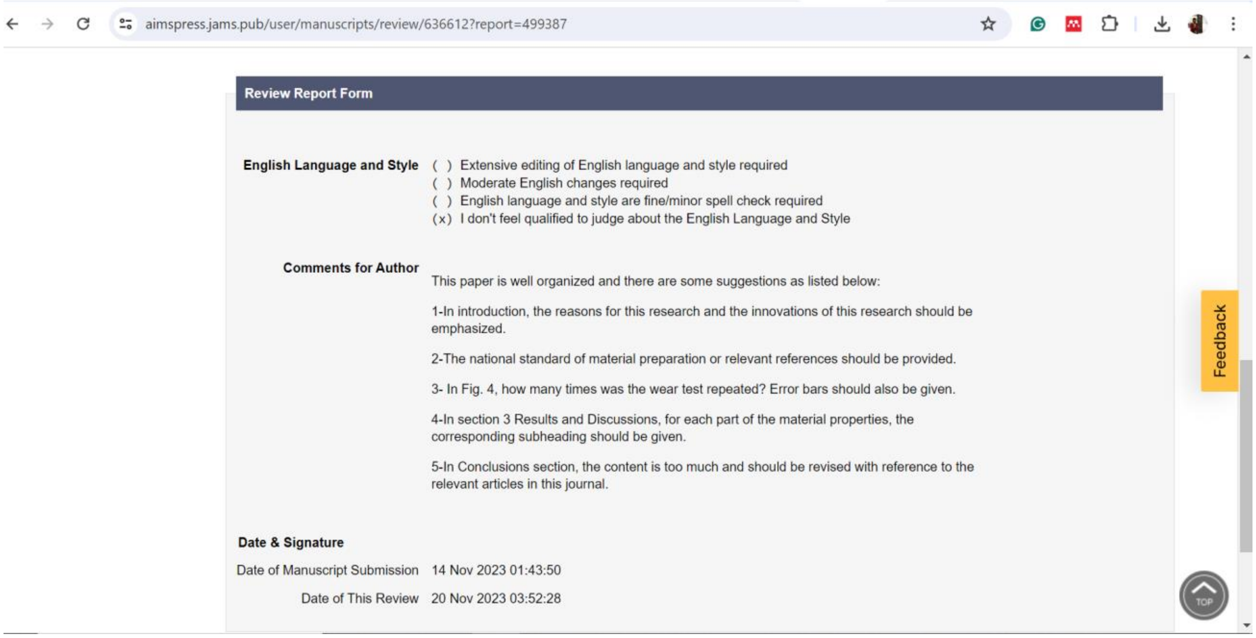

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Comments for Author

This paper is well organized and there are some suggestions as listed below:

1-In introduction, the reasons for this research and the innovations of this research should be emphasized.

2-The national standard of material preparation or relevant references should be provided.

3- In Fig. 4, how many times was the wear test repeated? Error bars should also be given.

4-In section 3 Results and Discussions, for each part of the material properties, the corresponding subheading should be given.

5-In Conclusions section, the content is too much and should be revised with reference to the relevant articles in this journal.

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Date of Manuscript Submission	14 Nov 2023 01:43:50
Date of This Review	20 Nov 2023 03:52:28

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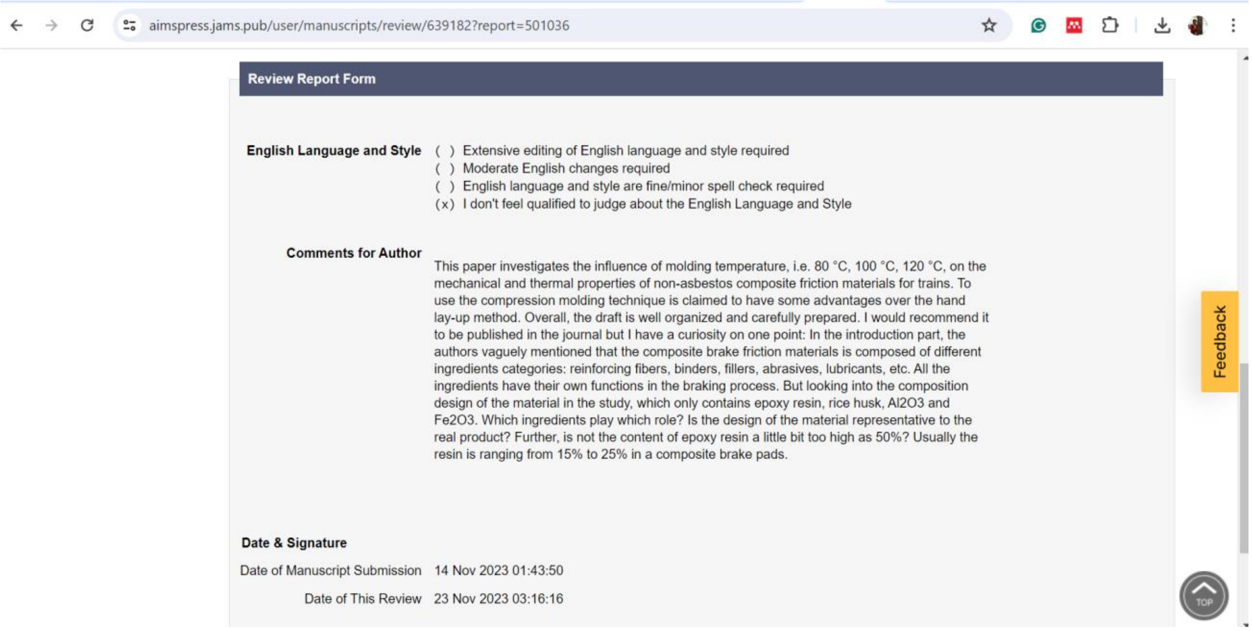

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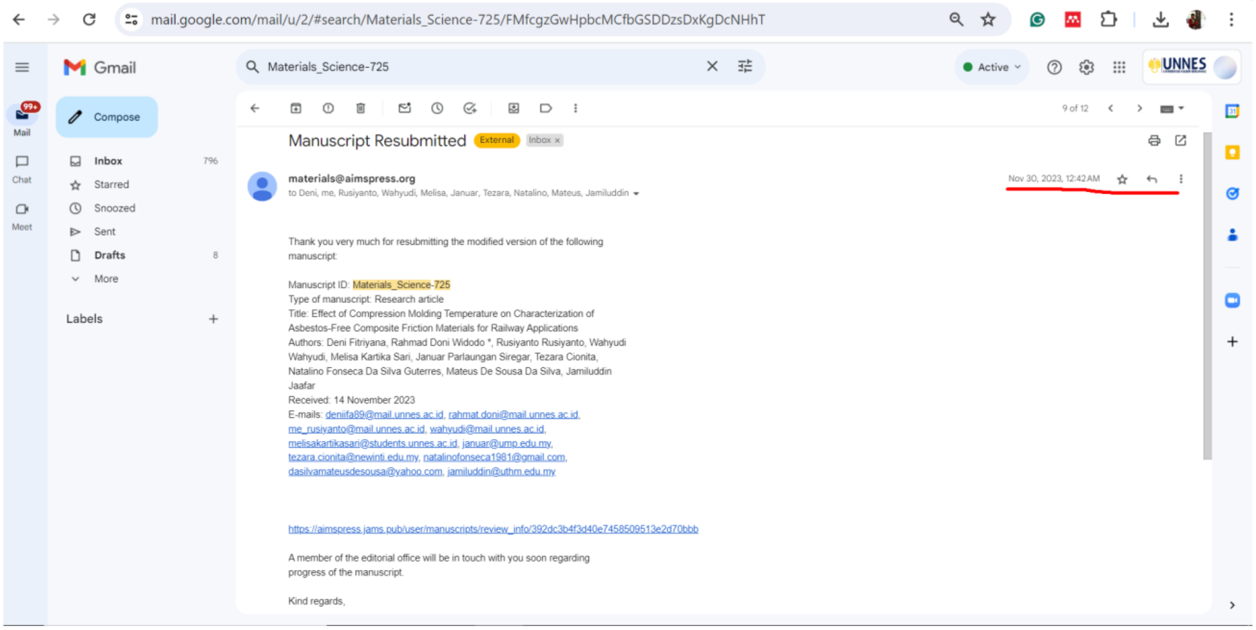
Comments for Author

This paper investigates the influence of molding temperature, i.e. 80 °C, 100 °C, 120 °C, on the mechanical and thermal properties of non-asbestos composite friction materials for trains. To use the compression molding technique is claimed to have some advantages over the hand lay-up method. Overall, the draft is well organized and carefully prepared. I would recommend it to be published in the journal but I have a curiosity on one point: In the introduction part, the authors vaguely mentioned that the composite brake friction materials is composed of different ingredients categories: reinforcing fibers, binders, fillers, abrasives, lubricants, etc. All the ingredients have their own functions in the braking process. But looking into the composition design of the material in the study, which only contains epoxy resin, rice husk, Al_2O_3 and Fe_2O_3. Which ingredients play which role? Is the design of the material representative to the real product? Further, is not the content of epoxy resin a little bit too high as 50%? Usually the resin is ranging from 15% to 25% in a composite brake pads.

Date & Signature

Date of Manuscript Submission	14 Nov 2023 01:43:50
Date of This Review	23 Nov 2023 03:16:16

4. Mengirimkan response letter dan artikel yang telah diperbaiki pada tanggal 30 Nopember 2023. Pada tahap ini, Submission telah direview dan perlu dilakukan perbaikan. File yang diperlukan pada tahap ini adalah :



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materials@aimspress.org
to Deni, me, Rusiyanto, Wahyudi, Melisa, Januar, Tezara, Natalino, Mateus, Jamiluddin
Nov 30, 2023, 12:42 AM

Thank you very much for resubmitting the modified version of the following manuscript:

Manuscript ID: Materials_Science-725
Type of manuscript: Research article
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Authors: Deni Fitriyana, Rahmad Doni Widodo *, Rusiyanto Rusiyanto, Wahyudi Wahyudi, Melisa Kartika Sari, Januar Parlaungan Siregar, Tezara Cionita, Natalino Fonseca Da Silva Guterres, Mateus De Sousa Da Silva, Jamiluddin Jaafar
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A member of the editorial office will be in touch with you soon regarding progress of the manuscript.

Kind regards,

A. Response letter untuk reviewer 1

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Effect of Compression Molding Temperature on Characterization of Asbestos-Free Composite Friction Materials for Railway Applications

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Response to reviewer's comments

We thank the reviewer for the comments and suggestions. We have now addressed them as discussed below. All changes have been shown in a marked version of the manuscript (in 'red font'), which is attached with this submission. Also, all responses are indicated in blue font under each comment while the reviewer's comments remain in black font.

Reviewers 1

This paper is well organized and there are some suggestions as listed below:

1. In introduction, the reasons for this research and the innovations of this research should be emphasized.

Thank you for the reviewer's suggestion. The author has improved the last paragraph in the Introduction section.

2. The national standard of material preparation or relevant references should be provided.

   Thank you for the reviewer's suggestion. The author has added an explanation to the Specimen Fabrications section.

3. In Fig. 4, how many times was the wear test repeated? Error bars should also be given.

   Thank you for the reviewer's suggestion. the author has made revisions to this section

4. In section 3 Results and Discussions, for each part of the material properties, the corresponding subheading should be given.

   Thank you for the reviewer's suggestion. The author has made revisions to this section

5. In Conclusions section, the content is too much and should be revised with reference to the relevant articles in this journal.

   Thank you for the reviewer's suggestion. The author has made revisions to this section.

B. Response letter untuk reviewer 2

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Reviewers 2

This paper investigates the influence of molding temperature, i.e. 80 °C, 100 °C, 120 °C, on the mechanical and thermal properties of non-asbestos composite friction materials for trains. To use the compression molding technique is claimed to have some advantages over the hand lay-up method. Overall, the draft is well organized and carefully prepared. I would recommend it to be published in the journal but I have a curiosity on one point:

- In the introduction part, the authors vaguely mentioned that the composite brake friction materials is composed of different ingredients categories: reinforcing fibers, binders, fillers, abrasives, lubricants, etc. All the ingredients have their own functions in the braking process. But looking into the composition design of the material in the study, which only contains epoxy resin, rice husk, Al2O3 and Fe2O3. Which ingredients play which role? Is the design of the material representative to the real product?

  Thank you for the reviewer's suggestion. The author has improved the last paragraph in the Introduction section.

- Further, is not the content of epoxy resin a little bit too high as 50%? Usually, the resin is ranging from 15% to 25% in a composite brake pad.

  The use of epoxy resin with a content of 50% is based on our literature review. We have also added a reference to our revised manuscript (Reference no 13).

  Irawan, A.P.; Fitriyana, D.F.; Tezara, C.; Siregar, J.P.; Laksmidewi, D.; Baskara, G.D.; Abdullah, M.Z.; Junid, R.; Hadi, A.E.; Hamdan, M.H.M.; et al. Overview of the Important Factors Influencing the Performance of Eco-Friendly Brake Pads. Polymers (Basel). 2022, 14, 1–22, doi:10.3390/polym14061180.

C. Manuskrip yang diperbaiki dengan track change

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Abstract: Brake pads significantly affect the braking performance of railways under both normal and emergency operating conditions. In previous studies, brake pads were made using the hand lay-up method and produced the best properties on specimens with epoxy, rice husk, Al_2O_3, and Fe_2O_3 compositions of 50%, 20%, 15%, and 15%. However, the resulting density does not meet the density standard set by PT Industri Kereta Api Indonesia (PT INKA), which is 1.7 - 2.4 gr/cm^3. To date, there has been limited research into the utilization of the compression hot molding method for the production of asbestos-free composite friction materials composed of epoxy, rice husk, Al_2O_3, and Fe_2O_3 for railway applications. This study aimed to determine the effect of compression molding temperature on the characterization of composite brake pads for railway applications. The brake pad specimens were made of epoxy resin, rice husk, Al_2O_3 and Fe_2O_3 with a composition of 50%, 20%, 15% and 15%, respectively. The manufacture of composites in this study used the compression molding method with a pressure of 20 MPa for 15 minutes holding time. The mold temperature used were 80°C, 100°C, 120°C.

Density, hardness, tensile, wear, TGA (Thermal Gravimetric Analysis), and DSC (Differential Scanning Calorimetry) tests were performed to evaluate the properties of the specimens obtained. The results demonstrated that an increase in molding temperature improved the characterization of the brake pads, with the best results attained at 120°C (SP-3 specimen). SP-3 specimens had the best density, hardness, tensile properties, and thermal properties compared to other specimens.

Keywords: Brake pads; railways; composites; compression molding; friction materials

1. Introduction

The effectiveness of brake pads has a significant impact on railways' capacity for stopping in an emergency as well as during normal operations [1]. Brake pads are used as components to ensure the safety of the railways during the braking process. Generally, brake pads on trains can be divided into organic and metal. Organic brake pads, which are composed of organic polymers, have been implemented in cars, trains, and other modes of transportation. Metal-based brake pads have emerged as the preferred material for high-speed trains due to their outstanding friction capability, commendable resistance to wear, outstanding thermal conductivity, and ability to withstand high operational temperatures [1,2].

Composite brake pads have been used in Indonesia since the last decade to replace cast iron brake pads for trains. Cast iron brake pads wear out faster when compared to composite brake pads [3]. In addition, metallic brake pads have a very high density which can reduce the energy efficiency of the train system [4]. In recent years, the development of composite technology has made rapid progress with various innovations in the manufacture of brake pads using natural materials that have become waste and are no longer used. Composite itself is a material composed of a mixture of two or more materials with different mechanical properties to produce a new material that has different mechanical properties from its constituent materials. The properties of composite materials are a combination of the properties of its constituents, the matrix and reinforcement or filler. The matrix serves to transfer stress to the fiber, form coherent bonds, protect the fiber and remain stable after the manufacturing process. Reinforcement or filler materials must be able to support or improve the properties of the matrix in fabricating composite materials [5].

In previous studies, brake pads for applications on trains have been successfully made using the hand lay-up method. Brake pads with the best properties were found in specimens with a composition of epoxy, rice husk, Al_2O_3, and Fe_2O_3 of 50%, 20%, 15%, and 15% respectively and a mesh size of 200. This composition produced density, hardness, tensile strength, specific wear value and degradation temperature of 1.23 g/cm^3, 81.2 HVN, 23.34 MPa, 8.67 x 10^{-7} N/mm^2, and 379°C [6]. Meanwhile, when using 100 mesh rice husk, the density, hardness, specific wear value, and degradation temperature of the resulting composite brake friction material were 1.33 g/cm^3, 83.4 HVN, 10.8 x 10^{-7} N/mm^2, and 363.99°C [7]. However, the resulting density does not meet the standard density set by PT. INKA, which is 1.7– 2.4 gr/cm^3 [8].

The hand layup method is a commonly employed technique in the fabrication of composite materials. Typically, the initial cost associated with fabricating composites using the hand layup technique is quite economical. Furthermore, this methodology enables the production of items with diverse geometries, structures, and designs.

Nevertheless, the hand layup technique employed in composite manufacturing exhibits several limitations, including a reduced production speed and a decreased volume proportion of reinforcement. Furthermore, the use of this technique results in an uneven dispersion of reinforcing and matrix substances as a consequence of the inherent imprecision associated with handling by hand. As a result, producing high-quality composites in large quantities using the hand layup method is not possible [9–11].

Based on the description above, another method is needed to meet the characteristic requirements of brake pads for applications on trains. One method of fabricating composites that can produce better characteristics is compression molding. According to the findings of a study by Nyior et al. (2018), compression molding produced composites with better mechanical properties than the manual lay-up (hand lay-up) method. Their study found that the tensile strength and Young's modulus of samples made with compression molding were 77% and 47% higher than samples made by hand lay-up, respectively. The results also showed that the impact strength of the materials made by compression molding (11.5 kJ/m^2) was significantly higher than that of the samples made by hand lay-up, which had an impact strength of 7 kJ/m^2 [12].

In general, the components used in brake pads to create friction materials are the matrix or adhesive, reinforcements, fillers, and abrasives. The proper combination of these components is critical to ensuring the efficient and reliable functioning of brake pads in various applications, such as brake pads for railways [13]. The high-speed trains usually use special brake block materials consisting of steel wool fiber, resin, aramid fiber, graphite, barium sulfate, magnesium, friction powder, mineral wool fiber, calcium carbonate powder, butyronitrile rubber powder, antimony sulfide, and argil [14]. On the other hand, brake pads designed for high-speed and heavy trains are produced using phenolic resin, composite fibers, graphite, barium sulfate, iron powder, butyronitrile, zirconite, rubber powder, feldspar powder, and alumina [15]. Meanwhile, the composite brake block for trains made by Green Power Runde Industry Co. Limited. consists of phenolic resin, nitrile rubber, steel fiber, reduced iron powder, graphite, etc [16]. In this study, the fabrication of asbestos-free composite friction material specimens for railway applications utilizes a more straightforward combination of materials that include epoxy, rice husk, Al_2O_3, and Fe_2O_3. The roles of epoxy resin, rice husk, Al_2O_3, and Fe_2O_3 are as matrix [17], reinforcement [18], filler [19], and abrasive [20,21], respectively. The composite material as friction materials will be pressed and heated at a certain pressure and temperature. Furthermore, the fabrication of asbestos-free composite friction materials for railway applications made from epoxy, rice husk, Al_2O_3, and Fe_2O_3 by compression hot molding has not been widely studied. The pressure applied can make the composite tighter and denser. While the heating process makes the resin move to flow to fill the empty composite parts. The purpose of this study was to determine the effect of compression molding temperature on the characterization of composite brake pads for applications on railways.

2. Materials and Methods

2.1. Materials

The materials used in this study were epoxy, hardener, rice husk, aluminum oxide (Al_2O_3), and iron oxide (Fe_2O_3). The matrix used in this study was Bisphenol A-Epichlorohydrin epoxy resin as a binder, and Cycloaliphatic Amine type epoxy hardener obtained from the Justus store in Semarang, Indonesia.

In general, epoxy has a density, tensile strength, and flexural strength of 1.18 g/cm^3, 63.7 MPa, and 8.3 MPa [22]. The rice husk used in this study was obtained from a rice mill near the campus. The composition of rice husk consists of cellulose (50%), lignin (25%-30%), silica (15%-20%), and the remainder consists of hemicellulose and water content [23]. In this study, Al_2O_3 and Fe_2O_3 were obtained from PT Merck Tbk, Indonesia.

2.2. Specimen Fabrications

In this study, the fabrication of brake pad specimens used the compression molding method. The preparation of raw materials for the fabrication of brake pad specimens refers to previous research [6,7]. The rice husk was crushed using the FOMAC FCT-Z300 miller machine and sieved using a 200 mesh. Furthermore, the specimens were prepared by mixing the materials according to a predetermined concentration using a hand mixer in a plastic cup in stages. First, mixing epoxy and hardener with a ratio of 1:3 was carried out for 7 minutes. Then, a mixture of rice husk, aluminum oxide, iron oxide was added after being stirred for 5 minutes. Mixing was done again for 10 minutes. After all the ingredients were mixed, they were poured into the mold and left to harden at room temperature for up to 10 hours. Furthermore, the specimen was compressed with a pressure of 20 MPa for 15 minutes and a temperature of 80°C, 100°C and 120°C (Table. 1). After that, the composite specimens were cut and characterized.

Table 1: Composite brake pads specimen code and compression molding setting parameters

Specimen Code	Pressure (MPa)	Holding Time (min.)	Temp (°C)
SP-1	20	15	80
SP-2	20	15	100
SP-3	20	15	120

2.3. Testing and Characterizations

In this study, the tests carried out were density, hardness, tensile, wear using the Ogoshi method, TGA (Thermal Gravimetric Analysis), and DSC (Differential Scanning Calorimetry). Density testing was based on ASTM D792 standard. Density testing was performed using an electronic density meter DME 220 series from Vibra Canada Inc. (Mississauga, ON, USA). This test is carried out by weighing the dry mass and the mass of the test object in water (wet mass). Vickers hardness testing in this study refers to the testing method carried out by [6,24]. Hardness testing was carried out using a Microhardness Tester F-800 machine (Future-Tech Corp., Kanagawa, Japan) with a test load of 25 gf and a dwell time of 10 seconds. Tensile testing was done according to the ASTM D638 standard using a HT-2402 Computer Servo Control Material Testing Machines from Hung Ta Instrument Co., Ltd., Samutprakarn, Thailand. The tensile test results consist of max force and elongation, which will be used to calculate the tensile strength and tensile modulus. Wear testing was performed using the Oghosi High Speed Universal Wear Testing Machine (Type OAT-U). The Ogoshi wear test was carried out with the width of the wear plate (B) = 3 mm, the radius of the wear plate (r) = 13.06 mm, the distance traveled during the wear process (10) = 66.6 m, and the test load (F) = 2.12 kg. The TGA test was carried out based on the ASTM D6370 standard using the NEXTA STA test kit (Hitachi STA200RV with Real View Sample Observation). The temperature used in this test was 30°C – 550°C. The testing process was conducted at a heating rate of 10°C/minute with a 100 ml/minute nitrogen gas flow.

The test results will obtain the temperature and weight loss values. The DSC test was carried out based on the ASTM D3418 standard using the NEXTA STA test tool (Hitachi STA200RV with Real View Sample Observation). The temperature used in this test was 30°C – 550°C. The testing process was carried out at a heating rate of 10°C /minute with a nitrogen gas flow of 100 ml/minute. The test results will obtain the temperature and heat flow values of the composite specimens.

3. Results and Discussions

3.1. Effect of Compression Molding Temperature on the density of brake pad composite specimens

Figure 1 shows the density of composite brake pad specimens with variations in molding temperature. The results of this study indicated that the increase in temperature affected the results of the composite density. The lowest density was shown in the SP-1 specimen, which was 1,695 g/cm^3. While the highest density was shown in the SP-3 specimen, which was 1,701 g/cm^3. The increase in density value occurs due to an increase in the temperature of composite fabrications with the compression molding method which causes a decrease in the viscosity of the resin, making it easier for the polymer to fill voids [25,26]. The fewer voids produced, the greater the density of the brake pad samples [6]. Void content is the percentage by volume of empty spaces or cavities inside composite materials [26]. When there are voids in a composite, they occupy space that would otherwise be occupied by the composite material. As a consequence, the composite's overall density decreases as the total mass is distributed over a larger volume [6,26].

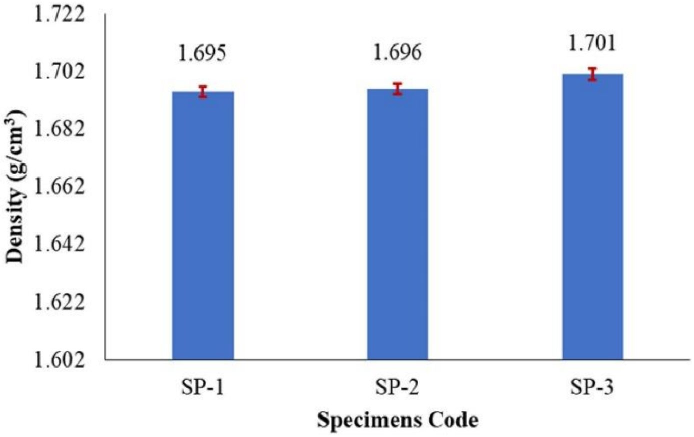

Figure 1: **Effect of Compression Molding Temperature on the density of brake pad composite specimens**

In the manufacture of brake pad specimens using the compression molding method, an increase in molding temperature causes the viscosity of the resin to decrease so that the resin flows more easily and wets the reinforcing material. This results in a better bond between the resin, filler, and reinforcement, resulting in an increase in the density of the composite specimen [6]. This is what causes SP-3 to have a higher density than other composite specimens. Incomplete resin flow into the desiccated area of reinforcement and filler materials can give rise to porosity in composite specimens.

This is typically the result of increased resin viscosity due to exposure to ambient conditions and low-temperature curing cycles, resulting in inadequate flow. The lowered molding temperature reduces the driving force of gas-induced cavity growth. So that more porosity is produced, resulting in a decrease in the specimen's mechanical properties [26–29].

The results of this investigation are consistent with Ochi et al.'s (2015) research. Their findings demonstrated a correlation between the density of long bamboo fiber/ PLA Composites and the temperature of the mold. In general, the density of composites increases from 140°C to 160°C as the molding temperature. However, when the molding temperature exceeds 160°C, the density of the composite decreases. This occurs because mold temperatures above 160°C reduce the matrix's viscosity and make it simpler for air to become trapped inside the material during the molding process, resulting in the formation of numerous voids [30]. In another study, Ochi et al. (2022) demonstrated the relationship between the density of bamboo fiber bundle-reinforced bamboo powder composite materials and the molding temperature. As the molding temperature increased, the density of the composite specimens remained relatively constant. The density of composites was between 1.41 and 1.42 g/cm^3 [31].

Based on these research results, only the SP-3 specimen met the minimum density determined by PT INKA, which was 1.7–2.4 gr/cm^3 [8]. Meanwhile, the density of the SP-2 and SP-1 specimens almost met the specified density requirements. In addition, the density obtained in this study was higher than the results of previous studies. In previous research, brake pad composite fabrication used the hand layup method with various compositions. The highest density produced in previous studies was 1.23 g/cm^3 [6], and 1.33 g/cm^3 [7]. Whereas in this study, the densities of the specimens Sp-1, SP-2, and SP-3 were 1,695 g/cm^3, 1,696 g/cm^3, and 1.701 g/cm^3, respectively.

3.2. Effect of Compression Molding Temperature on the hardness of brake pad composite specimens

The effect of temperature on the hardness of the composite is shown in Figure 2. The lowest hardness value was shown in the SP-1 specimen, which was 96.9 gf/mm^2 (HVN). While the highest density was shown in the SP-3 specimen, which was 108.2 gf/mm^2 (HVN). SP3 and SP1 specimens were the specimens with the highest and lowest densities produced in the study, respectively. In general, the hardness of composite specimens increases in proportion to their density. This is due to the fact that dense materials have strong interfacial bonding's between their matrix and reinforcement, making them more resistant to indentation and plastic deformation [6,32].

Previous research reached the same conclusion, which is that the specimen's higher density increased its hardness [33–36]. Research conducted by Fouly et al. (2021) showed that increasing the density of the composite causes an increase in the hardness of the specimen. Therefore, the average hardness also increases with increasing density. The PMHA8 specimen obtained a maximum hardness of 87.7 D-index. This happened because the PMHA8 specimen had the highest density compared to the other specimens [32]. Furthermore, an increase in the composite is hardness indicates a good interfacial bond between the matrix and the reinforcing fiber. The stronger the interfacial bond between the matrix and the reinforcing fiber, the higher the hardness of the resulting composite specimen [37,38]. Yawas et al. obtained the maximum hardness and density of non-asbestos brake pad samples by using a compaction load and sintering temperature of 15 tonnes and 150 °C, respectively. This happens because using these variations results in a more uniform distribution of frictional filler material particles in the matrix. Furthermore, this variation increases the surface area, which improves the matrix's bonding ability with frictional filler material [39].

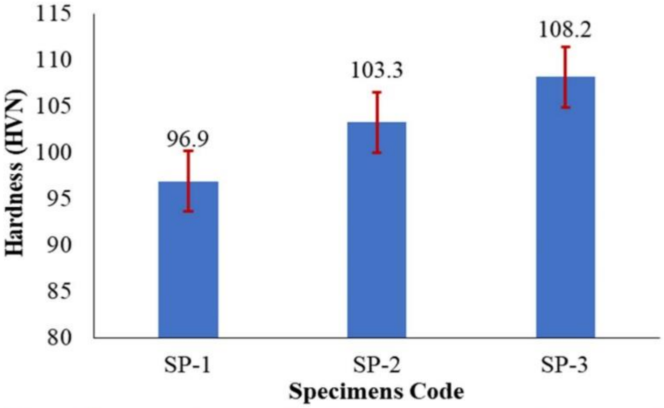

Figure 2: **Effect of Compression Molding Temperature on the hardness of brake pad composite specimens**

3.3. Effect of Compression Molding Temperature on the tensile strength of brake pad composite specimens

The effect of temperature on the tensile strength of the composite is shown in Figure 3. The lowest tensile strength value was shown in the SP-1 specimen, which was 7.26 MPa. While the highest tensile strength was shown in the SP-3 specimen, which was 26.22 MPa.

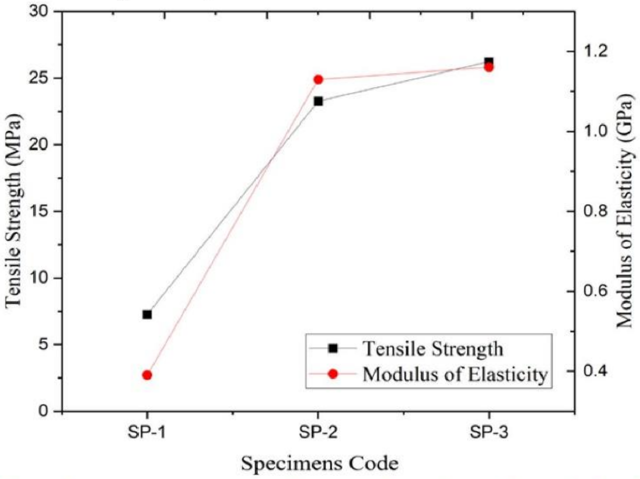

Figure 3: **Effect of temperature on tensile strength and tensile modulus of brake pad composite specimens**

The increase in the value of tensile strength is affected by the behavior of the bond between the resin and fiber interfaces at each increasing compression molding temperature variation [40]. This is in line with research conducted by Sumesh & Kanthavel (2020). Composites with an epoxy resin matrix formed by the compression molding method produce the best mechanical properties of tensile strength at higher temperatures [41].

Heating at high temperatures facilitates the mobilization of the resin in fiber impregnation. The increase in tensile strength is also due to an increase in mold temperature which will reduce the viscosity of the matrix which has an impact on reducing voids in the composite [42].

The increase in tensile strength may also be caused by the main process that occurs at a molding temperature of 80˚C-120˚C for the evaporation. The higher the molding temperature, the higher the moisture content in the evaporating fiber, therefore the higher the tensile strength. Mvondo et al., (2017), stated that there was a negative relationship between the moisture content of tropical wood fiber and its tensile strength. Differences in the percentage of water content in fiber were studied. In fibers that have a lower water content produces high tensile strength, while high water content produces low tensile strength [43]. Figure 3 shows that the modulus of elasticity had increased with increasing molding temperature. The modulus of elasticity represents the inflexibility of a material. The higher the value of the modulus of elasticity, the less flexible the material is. At higher molding temperatures, the specimen becomes stiffer so that the modulus value is higher [18,19]. In accordance with research conducted by Ochi et al. (2022) [31], the value of the elastic modulus increased with increasing temperature used in the specimen molding process using the compression molding method.

3.4. Effect of Compression Molding Temperature on the specific wear of brake pad composite specimens

The wear test results showed that the use of higher mold temperatures can cause a decrease in the wear resistance of the composite (Figure 4). A higher specific wear value indicated a lower wear resistance property. In the study, the SP-3 specimen had the highest specific wear value of 15.08×10^{-7} mm^2/kg while the SP-2 specimen had the lowest specific wear value. So, it can be said that the highest wear resistance was found in the SP-2 specimen. According Günay et al. (2020), a negative correlation exists between a material's hardness value and the specific wear value it exhibits. Materials possessing high hardness exhibit enhanced resistance to wear and tear, hence endowing the test specimens with excellent wear resistance.

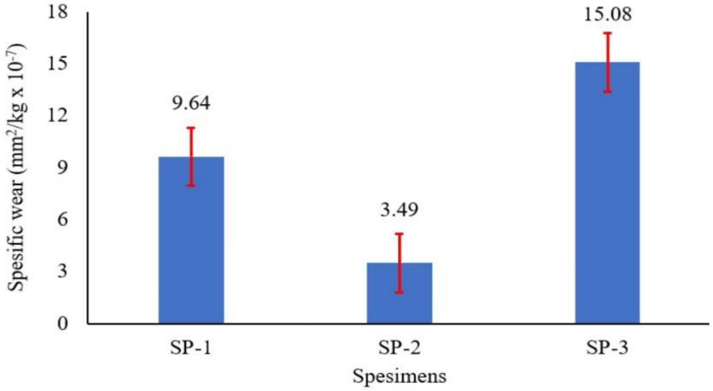

Figure 4: **Effect of Compression Molding Temperature on the specific wear of brake pad composite specimens**

The reason for this phenomenon is that the wear value acquired during Ogoshi wear testing exhibits an inverse relationship with the wear resistance qualities of the material being tested. A material's wear resistance qualities are improved when its specific wear value decreases [46].

Nevertheless, this investigation revealed that SP-3 had the highest specific wear value. It can be asserted that SP-3 exhibits the lowest level of wear resistance. This phenomenon can be attributed to the positive correlation between the temperature applied and the surface roughness observed in the composite specimen. The findings of a study conducted by Jan et al. (2020) indicate that elevating the temperature of the mold leads to a corresponding increase in the roughness of the composite material. Their findings indicated that elevating the mold temperature led to a corresponding increase in the surface roughness of the brake pad [47]. The occurrence of increased surface roughness in the specimen can be attributed to the roughness of the composite surface when it comes into contact with the surface of the hard steel disc (used as test equipment). This contact leads to the formation of cracks on both the surface and subsurface of the specimen, which results in matrix delamination or wear [48]. This results in the removal of material in large flakes and creates various irregular edge shapes resulting in higher friction and wear [48].

3.5. Effect of Compression Molding Temperature on the thermal properties of brake pad composite specimens

TGA and DSC testing in this study was conducted to determine the thermal properties of the composite. Figure 5 shows that the composite brake pads specimen had several temperature variations during thermal decomposition that occurred in the temperature range of 30°C – 550°C. Weight loss on initial heating (30°C – 200°C) was caused by the evaporation of water on the rice husk. This happened because water was not chemically bound to the fiber. The reduction of fiber mass was further related to the degradation of hemicellulose. In the second range (200°C – 400°C), the weight loss that occurred was mostly due to the decomposition of hemicellulose and cellulose fibers and was followed by the decomposition of epoxy resin. In the last range (400°C – 550°C), the weight loss that occurred was caused by fiber degradation and decomposition of epoxy resin and filler.

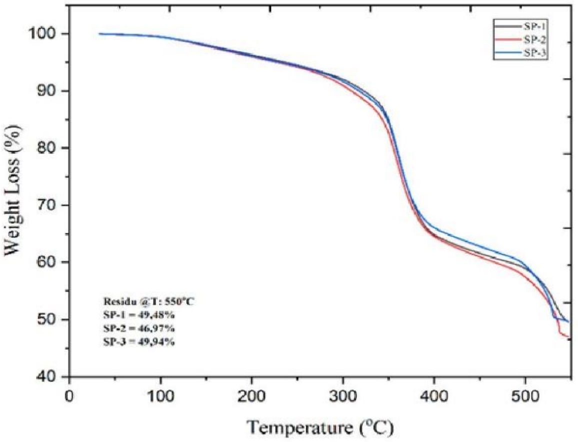

Figure 5: Effect of compression molding temperature on composite weight loss

This is in accordance with research conducted by Chen et al., (2020), rice husk experiences weight loss which is divided into three stages. At a temperature of 35°C – 150°C, rice husk undergoes evaporation of water in the fiber. At temperatures of 150°C – 380°C decomposition of hemicellulose, cellulose and lignin occurs and at temperature stages of 380°C – 600°C fiber degradation occurs [49].

The TGA test with a temperature scale of 30°C – 550°C resulted in a residue of 49.48% in the SP-1 specimen, 46.97% in the SP-2 specimen and 49.94% in the SP-3 specimen. The residual results in this study are in line with research by Li et al., (2020), the residual weight of the composite showed a tendency to increase with increasing molding temperature. The results of the TG curve analysis show that the right molding temperature will help the curing reaction and cross-linking of resin and fiber. The increased crosslink density of the composite will strengthen the thermal stability of the composite [50].

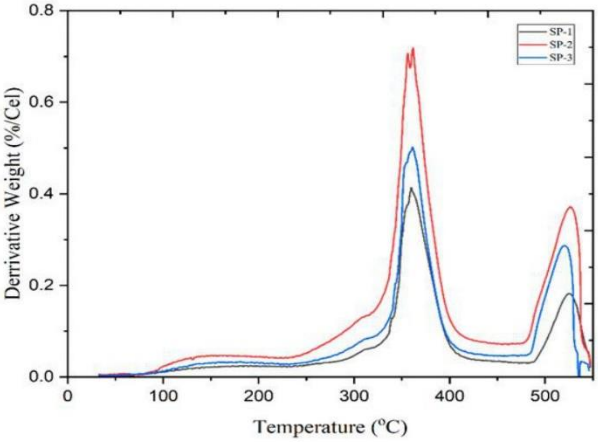

Figure 6: The effect of compression molding temperature on the derivative weight of the composite

Figure 6 shows the DTG (Derivative Thermal Gravimetry) curve for each composite brake pads specimen that experienced a maximum decomposition phase (Tmax) concerning temperature which was shown at the main peak. SP-1 specimen occurred at 359.98°C, SP-2 specimen at 356.13°C, and SP-3 specimen at 360.94°C. Maximum decomposition is the maximum weight loss on the specimen that occurs at a certain temperature (Tmax) which can be used as the most important indicator in determining the thermal stability of a material [51]. The results showed that there was an effect of the use of specimen molding temperature. The specimen with a molding temperature of 120°C had a larger Tmax and residue than other specimens. According to research by Li et al., (2020), by increasing the molding temperature, the durability of the epoxy resin and fiber activity tends to increase, which benefits the chemical bond between the fiber and the resin which results in increased composite cross-linking so that the degradation temperature will be higher [50].

Based on Figure 7, the exothermic phase occurred when heated to a temperature of 30°C – 550°C. On the DSC curve, Tg can be known from each specimen. The glass temperature is the transition temperature where the behavior of the composite transitions from hard glass to soft rubber [52]. The SP-1 specimen had a Tg of 311.25°C. The SP-2 specimen had a Tg of 309.18°C. The SP-3 specimen had a Tg of 310.06°C. After passing through the Tg material, each specimen began to form crystals. Specimens will experience cold crystallization; cold crystallization is a unique phenomenon in which crystallization that accompanies an exothermic anomaly occurs when a material is heated to a temperature below its melting point but above the temperature of its glass. On the curve, the peak point of the curve after going through the Tg phase is Tc. Temperature crystallization is a transition temperature where the formation of a crystal structure occurs due to heating [53].

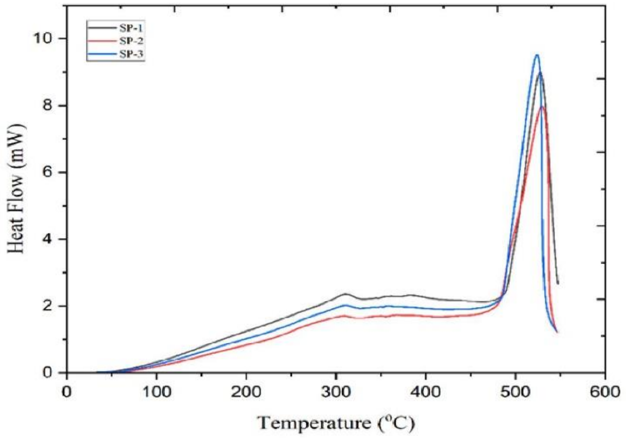

Figure 7: Effect of compression molding temperature on composite heat flow

The SP-1 specimen has the highest Tc of 526.17°C. The SP-2 specimen has the highest Tc of 529.83°C. The SP-3 specimen has the highest Tc of 523.17°C. The three specimens did not experience an endothermic phase, in which the specimens did not melt up to 550°C. On the DSC curve, Tm (Temperature melting) can be observed at the turning point of the curve. This is in line with Li et al., (2020) which stated that the first exothermic peak is closely related to the glass transition. Increased Tg and decreased peaks can be attributed to increased crosslinking and amorphism. The decreasing exothermic peak value of the composite indicates that the crosslink density of the mixture is increasing. It can be concluded that a reasonable increase in molding temperature will be conducive to increasing the crosslink density [50].

4. Conclusions

The effect of compression molding temperature on the characterization of asbestos-free composite friction materials for railway applications has been discussed in this study. Density, hardness, tensile, wear, TGA (Thermal Gravimetric Analysis), and DSC (Differential Scanning Calorimetry) tests were performed to evaluate the properties of the obtained specimens. According to the findings of this study, increasing the temperature during the compression molding process reduces the viscosity of the resin, allowing the resin to flow more easily and wet the reinforcement, filler, and abrasive materials. This leads to better bonding between the constituent materials, so the density of the composite specimen increases. The mechanical and thermal properties of the composite specimens increase as density increases. The results of this study show that Specimen SP-3 has better characteristics compared to other specimens. The density, Vickers hardness, tensile strength, and tensile modulus of SP-3 specimens are 1.70 g/cm^3, 108.2 HVN, 26.25 MPa, and 1.16 GPa, respectively. The highest thermal properties were generated by SP-3 specimen, with total residues, Tmax, Tg, and Tc values of 49.94%, 360.94°C, 310.06°C, and 517.17°C, respectively. Furthermore, the molding temperature has a significant effect on the specific wear on the composite specimen. Specific wear (x 10^{-7} mm^2/kg) on SP-1, SP-2, and SP-3 specimens was 9.64, 3.49, and 15.08, respectively.

Use of AI tools declaration

The authors declare they have not used Artificial Intelligence (AI) tools in the creation of this article.

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Conflict of Interest

The authors declare no conflict of interest

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Effect of Compression Molding Temperature on Characterization of Asbestos-Free Composite Friction Materials for Railway Applications

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Abstract: Brake pads significantly affect the braking performance of railways under both normal and emergency operating conditions. In previous studies, brake pads were made using the hand lay-up method and produced the best properties on specimens with epoxy, rice husk, Al_2O_3, and Fe_2O_3 compositions of 50%, 20%, 15%, and 15%. However, the resulting density does not meet the density standard set by PT Industri Kereta Api Indonesia (PT INKA), which is 1.7 - 2.4 gr/cm^3. To date, there has been limited research into the utilization of the compression hot molding method for the production of asbestos-free composite friction materials composed of epoxy, rice husk, Al_2O_3, and Fe_2O_3 for railway applications. This study aimed to determine the effect of compression molding temperature on the characterization of composite brake pads for railway applications. The brake pad specimens were made of epoxy resin, rice husk, Al_2O_3 and Fe_2O_3 with a composition of 50%, 20%, 15% and 15%, respectively. The manufacture of composites in this study used the compression molding method with a pressure of 20 MPa for 15 minutes holding time. The mold temperature used were 80°C, 100°C, 120°C.

Density, hardness, tensile, wear, TGA (Thermal Gravimetric Analysis), and DSC (Differential Scanning Calorimetry) tests were performed to evaluate the properties of the specimens obtained. The results demonstrated that an increase in molding temperature improved the characterization of the brake pads, with the best results attained at 120°C (SP-3 specimen). SP-3 specimens had the best density, hardness, tensile properties, and thermal properties compared to other specimens.

Keywords: Brake pads; railways; composites; compression molding; friction materials

1. Introduction

The effectiveness of brake pads has a significant impact on railways' capacity for stopping in an emergency as well as during normal operations [1]. Brake pads are used as components to ensure the safety of the railways during the braking process. Generally, brake pads on trains can be divided into organic and metal. Organic brake pads, which are composed of organic polymers, have been implemented in cars, trains, and other modes of transportation. Metal-based brake pads have emerged as the preferred material for high-speed trains due to their outstanding friction capability, commendable resistance to wear, outstanding thermal conductivity, and ability to withstand high operational temperatures [1,2].

Composite brake pads have been used in Indonesia since the last decade to replace cast iron brake pads for trains. Cast iron brake pads wear out faster when compared to composite brake pads [3]. In addition, metallic brake pads have a very high density which can reduce the energy efficiency of the train system [4]. In recent years, the development of composite technology has made rapid progress with various innovations in the manufacture of brake pads using natural materials that have become waste and are no longer used. Composite itself is a material composed of a mixture of two or more materials with different mechanical properties to produce a new material that has different mechanical properties from its constituent materials. The properties of composite materials are a combination of the properties of its constituents, the matrix and reinforcement or filler. The matrix serves to transfer stress to the fiber, form coherent bonds, protect the fiber and remain stable after the manufacturing process. Reinforcement or filler materials must be able to support or improve the properties of the matrix in fabricating composite materials [5].

In previous studies, brake pads for applications on trains have been successfully made using the hand lay-up method. Brake pads with the best properties were found in specimens with a composition of epoxy, rice husk, Al_2O_3, and Fe_2O_3 of 50%, 20%, 15%, and 15% respectively and a mesh size of 200. This composition produced density, hardness, tensile strength, specific wear value and degradation temperature of 1.23 g/cm^3, 81.2 HVN, 23.34 MPa, 8.67 x 10^{-7} N/mm^2, and 379°C [6]. Meanwhile, when using 100 mesh rice husk, the density, hardness, specific wear value, and degradation temperature of the resulting composite brake friction material were 1.33 g/cm^3, 83.4 HVN, 10.8 x 10^{-7} N/mm^2, and 363.99°C [7]. However, the resulting density does not meet the standard density set by PT. INKA, which is 1.7– 2.4 gr/cm^3 [8].

The hand layup method is a commonly employed technique in the fabrication of composite materials. Typically, the initial cost associated with fabricating composites using the hand layup technique is quite economical. Furthermore, this methodology enables the production of items with diverse geometries, structures, and designs.

Nevertheless, the hand layup technique employed in composite manufacturing exhibits several limitations, including a reduced production speed and a decreased volume proportion of reinforcement. Furthermore, the use of this technique results in an uneven dispersion of reinforcing and matrix substances as a consequence of the inherent imprecision associated with handling by hand. As a result, producing high-quality composites in large quantities using the hand layup method is not possible [9–11].

Based on the description above, another method is needed to meet the characteristic requirements of brake pads for applications on trains. One method of fabricating composites that can produce better characteristics is compression molding. According to the findings of a study by Nyior et al. (2018), compression molding produced composites with better mechanical properties than the manual lay-up (hand lay-up) method. Their study found that the tensile strength and Young's modulus of samples made with compression molding were 77% and 47% higher than samples made by hand lay-up, respectively. The results also showed that the impact strength of the materials made by compression molding (11.5 kJ/m^2) was significantly higher than that of the samples made by hand lay-up, which had an impact strength of 7 kJ/m^2 [12].

In general, the components used in brake pads to create friction materials are the matrix or adhesive, reinforcements, fillers, and abrasives. The proper combination of these components is critical to ensuring the efficient and reliable functioning of brake pads in various applications, such as brake pads for railways [13]. The high-speed trains usually use special brake block materials consisting of steel wool fiber, resin, aramid fiber, graphite, barium sulfate, magnesium, friction powder, mineral wool fiber, calcium carbonate powder, butyronitrile rubber powder, antimony sulfide, and argil [14]. On the other hand, brake pads designed for high-speed and heavy trains are produced using phenolic resin, composite fibers, graphite, barium sulfate, iron powder, butyronitrile, zirconite, rubber powder, feldspar powder, and alumina [15]. Meanwhile, the composite brake block for trains made by Green Power Runde Industry Co. Limited. consists of phenolic resin, nitrile rubber, steel fiber, reduced iron powder, graphite, etc [16]. In this study, the fabrication of asbestos-free composite friction material specimens for railway applications utilizes a more straightforward combination of materials that include epoxy, rice husk, Al_2O_3, and Fe_2O_3. The roles of epoxy resin, rice husk, Al_2O_3, and Fe_2O_3 are as matrix [17], reinforcement [18], filler [19], and abrasive [20,21], respectively. The composite material as friction materials will be pressed and heated at a certain pressure and temperature. Furthermore, the fabrication of asbestos-free composite friction materials for railway applications made from epoxy, rice husk, Al_2O_3, and Fe_2O_3 by compression hot molding has not been widely studied. The pressure applied can make the composite tighter and denser. While the heating process makes the resin move to flow to fill the empty composite parts. The purpose of this study was to determine the effect of compression molding temperature on the characterization of composite brake pads for applications on railways.

2. Materials and Methods

2.1. Materials

The materials used in this study were epoxy, hardener, rice husk, aluminum oxide (Al_2O_3), and iron oxide (Fe_2O_3). The matrix used in this study was Bisphenol A-Epichlorohydrin epoxy resin as a binder, and Cycloaliphatic Amine type epoxy hardener obtained from the Justus store in Semarang, Indonesia.

In general, epoxy has a density, tensile strength, and flexural strength of 1.18 g/cm^3, 63.7 MPa, and 8.3 MPa [22]. The rice husk used in this study was obtained from a rice mill near the campus. The composition of rice husk consists of cellulose (50%), lignin (25%-30%), silica (15%-20%), and the remainder consists of hemicellulose and water content [23]. In this study, Al_2O_3 and Fe_2O_3 were obtained from PT Merck Tbk, Indonesia.

2.2. Specimen Fabrications

In this study, the fabrication of brake pad specimens used the compression molding method. The preparation of raw materials for the fabrication of brake pad specimens refers to previous research [6,7]. The rice husk was crushed using the FOMAC FCT-Z300 miller machine and sieved using a 200 mesh. Furthermore, the specimens were prepared by mixing the materials according to a predetermined concentration using a hand mixer in a plastic cup in stages. First, mixing epoxy and hardener with a ratio of 1:3 was carried out for 7 minutes. Then, a mixture of rice husk, aluminum oxide, iron oxide was added after being stirred for 5 minutes. Mixing was done again for 10 minutes. After all the ingredients were mixed, they were poured into the mold and left to harden at room temperature for up to 10 hours. Furthermore, the specimen was compressed with a pressure of 20 MPa for 15 minutes and a temperature of 80°C, 100°C and 120°C (Table. 1). After that, the composite specimens were cut and characterized.

Table 1: Composite brake pads specimen code and compression molding setting parameters

Specimen Code	Pressure (MPa)	Holding Time (min.)	Temp (°C)
SP-1	20	15	80
SP-2	20	15	100
SP-3	20	15	120

2.3. Testing and Characterizations

In this study, the tests carried out were density, hardness, tensile, wear using the Ogoshi method, TGA (Thermal Gravimetric Analysis), and DSC (Differential Scanning Calorimetry). Density testing was based on ASTM D792 standard. Density testing was performed using an electronic density meter DME 220 series from Vibra Canada Inc. (Mississauga, ON, USA). This test is carried out by weighing the dry mass and the mass of the test object in water (wet mass). Vickers hardness testing in this study refers to the testing method carried out by [6,24]. Hardness testing was carried out using a Microhardness Tester F-800 machine (Future-Tech Corp., Kanagawa, Japan) with a test load of 25 gf and a dwell time of 10 seconds. Tensile testing was done according to the ASTM D638 standard using a HT-2402 Computer Servo Control Material Testing Machines from Hung Ta Instrument Co., Ltd., Samutprakarn, Thailand. The tensile test results consist of max force and elongation, which will be used to calculate the tensile strength and tensile modulus. Wear testing was performed using the Oghosi High Speed Universal Wear Testing Machine (Type OAT-U). The Ogoshi wear test was carried out with the width of the wear plate (B) = 3 mm, the radius of the wear plate (r) = 13.06 mm, the distance traveled during the wear process (10) = 66.6 m, and the test load (F) = 2.12 kg. The TGA test was carried out based on the ASTM D6370 standard using the NEXTA STA test kit (Hitachi STA200RV with Real View Sample Observation). The temperature used in this test was 30°C – 550°C. The testing process was conducted at a heating rate of 10°C/minute with a 100 ml/minute nitrogen gas flow.

The test results will obtain the temperature and weight loss values. The DSC test was carried out based on the ASTM D3418 standard using the NEXTA STA test tool (Hitachi STA200RV with Real View Sample Observation). The temperature used in this test was 30°C – 550°C. The testing process was carried out at a heating rate of 10°C /minute with a nitrogen gas flow of 100 ml/minute. The test results will obtain the temperature and heat flow values of the composite specimens.

3. Results and Discussions

3.1. Effect of Compression Molding Temperature on the density of brake pad composite specimens

Figure 1 shows the density of composite brake pad specimens with variations in molding temperature. The results of this study indicated that the increase in temperature affected the results of the composite density. The lowest density was shown in the SP-1 specimen, which was 1,695 g/cm^3. While the highest density was shown in the SP-3 specimen, which was 1,701 g/cm^3. The increase in density value occurs due to an increase in the temperature of composite fabrications with the compression molding method which causes a decrease in the viscosity of the resin, making it easier for the polymer to fill voids [25,26]. The fewer voids produced, the greater the density of the brake pad samples [6]. Void content is the percentage by volume of empty spaces or cavities inside composite materials [26]. When there are voids in a composite, they occupy space that would otherwise be occupied by the composite material. As a consequence, the composite's overall density decreases as the total mass is distributed over a larger volume [6,26].

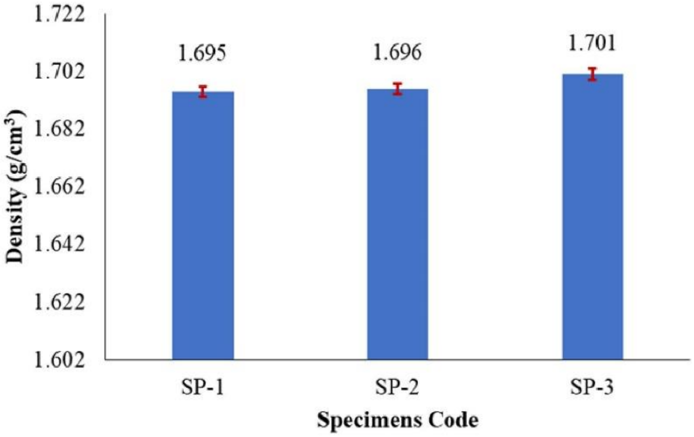

Figure 1: Effect of Compression Molding Temperature on the density of brake pad composite specimens

In the manufacture of brake pad specimens using the compression molding method, an increase in molding temperature causes the viscosity of the resin to decrease so that the resin flows more easily and wets the reinforcing material. This results in a better bond between the resin, filler, and reinforcement, resulting in an increase in the density of the composite specimen [6]. This is what causes SP-3 to have a higher density than other composite specimens. Incomplete resin flow into the desiccated area of reinforcement and filler materials can give rise to porosity in composite specimens.

This is typically the result of increased resin viscosity due to exposure to ambient conditions and low-temperature curing cycles, resulting in inadequate flow. The lowered molding temperature reduces the driving force of gas-induced cavity growth. So that more porosity is produced, resulting in a decrease in the specimen's mechanical properties [26–29].

The results of this investigation are consistent with Ochi et al.'s (2015) research. Their findings demonstrated a correlation between the density of long bamboo fiber/ PLA Composites and the temperature of the mold. In general, the density of composites increases from 140°C to 160°C as the molding temperature. However, when the molding temperature exceeds 160°C, the density of the composite decreases. This occurs because mold temperatures above 160°C reduce the matrix's viscosity and make it simpler for air to become trapped inside the material during the molding process, resulting in the formation of numerous voids [30]. In another study, Ochi et al. (2022) demonstrated the relationship between the density of bamboo fiber bundle-reinforced bamboo powder composite materials and the molding temperature. As the molding temperature increased, the density of the composite specimens remained relatively constant. The density of composites was between 1.41 and 1.42 g/cm^3 [31].

Based on these research results, only the SP-3 specimen met the minimum density determined by PT INKA, which was 1.7–2.4 gr/cm^3 [8]. Meanwhile, the density of the SP-2 and SP-1 specimens almost met the specified density requirements. In addition, the density obtained in this study was higher than the results of previous studies. In previous research, brake pad composite fabrication used the hand layup method with various compositions. The highest density produced in previous studies was 1.23 g/cm^3 [6], and 1.33 g/cm^3 [7]. Whereas in this study, the densities of the specimens Sp-1, SP-2, and SP-3 were 1,695 g/cm^3, 1,696 g/cm^3, and 1.701 g/cm^3, respectively.

3.2. Effect of Compression Molding Temperature on the hardness of brake pad composite specimens

The effect of temperature on the hardness of the composite is shown in Figure 2. The lowest hardness value was shown in the SP-1 specimen, which was 96.9 gf/mm^2 (HVN). While the highest density was shown in the SP-3 specimen, which was 108.2 gf/mm^2 (HVN). SP3 and SP1 specimens were the specimens with the highest and lowest densities produced in the study, respectively. In general, the hardness of composite specimens increases in proportion to their density. This is due to the fact that dense materials have strong interfacial bonding's between their matrix and reinforcement, making them more resistant to indentation and plastic deformation [6,32].

Previous research reached the same conclusion, which is that the specimen's higher density increased its hardness [33–36]. Research conducted by Fouly et al. (2021) showed that increasing the density of the composite causes an increase in the hardness of the specimen. Therefore, the average hardness also increases with increasing density. The PMHA8 specimen obtained a maximum hardness of 87.7 D-index. This happened because the PMHA8 specimen had the highest density compared to the other specimens [32]. Furthermore, an increase in the composite is hardness indicates a good interfacial bond between the matrix and the reinforcing fiber. The stronger the interfacial bond between the matrix and the reinforcing fiber, the higher the hardness of the resulting composite specimen [37,38]. Yawas et al. obtained the maximum hardness and density of non-asbestos brake pad samples by using a compaction load and sintering temperature of 15 tonnes and 150 °C, respectively. This happens because using these variations results in a more uniform distribution of frictional filler material particles in the matrix. Furthermore, this variation increases the surface area, which improves the matrix's bonding ability with frictional filler material [39].

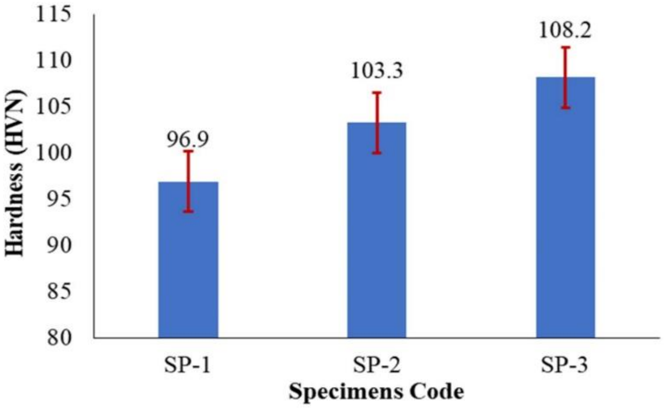

Figure 2: Effect of Compression Molding Temperature on the hardness of brake pad composite specimens

3.3. Effect of Compression Molding Temperature on the tensile strength of brake pad composite specimens

The effect of temperature on the tensile strength of the composite is shown in Figure 3. The lowest tensile strength value was shown in the SP-1 specimen, which was 7.26 MPa. While the highest tensile strength was shown in the SP-3 specimen, which was 26.22 MPa.

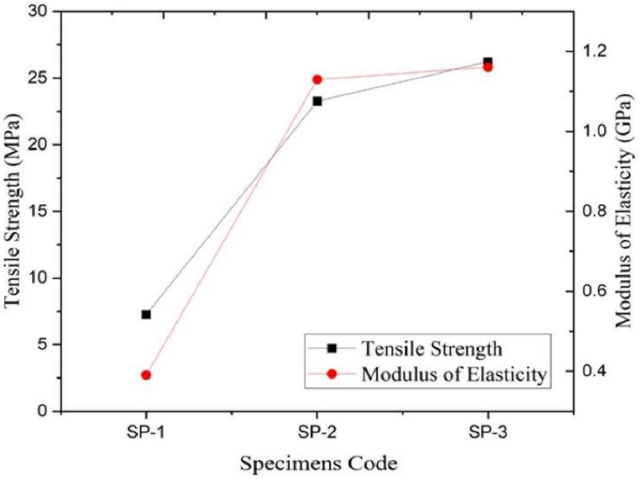

Figure 3: Effect of temperature on tensile strength and tensile modulus of brake pad composite specimens

The increase in the value of tensile strength is affected by the behavior of the bond between the resin and fiber interfaces at each increasing compression molding temperature variation [40]. This is in line with research conducted by Sumesh & Kanthavel (2020). Composites with an epoxy resin matrix formed by the compression molding method produce the best mechanical properties of tensile strength at higher temperatures [41].

Heating at high temperatures facilitates the mobilization of the resin in fiber impregnation. The increase in tensile strength is also due to an increase in mold temperature which will reduce the viscosity of the matrix which has an impact on reducing voids in the composite [42].

The increase in tensile strength may also be caused by the main process that occurs at a molding temperature of 80°C-120°C for the evaporation. The higher the molding temperature, the higher the moisture content in the evaporating fiber, therefore the higher the tensile strength. Mvondo et al., (2017), stated that there was a negative relationship between the moisture content of tropical wood fiber and its tensile strength. Differences in the percentage of water content in fiber were studied. In fibers that have a lower water content produces high tensile strength, while high water content produces low tensile strength [43]. Figure 3 shows that the modulus of elasticity had increased with increasing molding temperature. The modulus of elasticity represents the inflexibility of a material. The higher the value of the modulus of elasticity, the less flexible the material is. At higher molding temperatures, the specimen becomes stiffer so that the modulus value is higher [18,19]. In accordance with research conducted by Ochi et al. (2022) [31], the value of the elastic modulus increased with increasing temperature used in the specimen molding process using the compression molding method.

3.4. Effect of Compression Molding Temperature on the specific wear of brake pad composite specimens

The wear test results showed that the use of higher mold temperatures can cause a decrease in the wear resistance of the composite (Figure 4). A higher specific wear value indicated a lower wear resistance property. In the study, the SP-3 specimen had the highest specific wear value of 15.08×10^{-7} mm^2/kg while the SP-2 specimen had the lowest specific wear value. So, it can be said that the highest wear resistance was found in the SP-2 specimen. According Günay et al. (2020), a negative correlation exists between a material's hardness value and the specific wear value it exhibits. Materials possessing high hardness exhibit enhanced resistance to wear and tear, hence endowing the test specimens with excellent wear resistance.

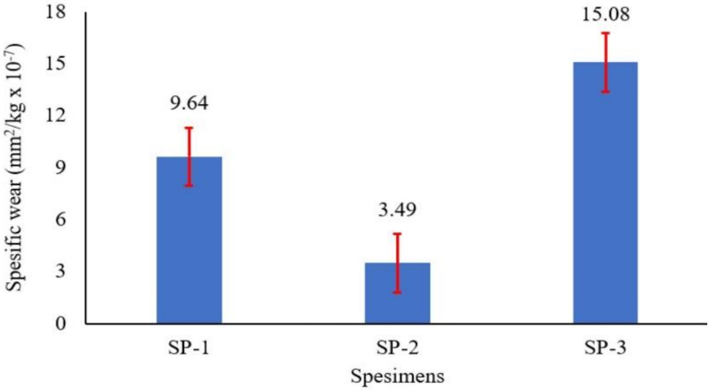

Figure 4: Effect of Compression Molding Temperature on the specific wear of brake pad composite specimens

The reason for this phenomenon is that the wear value acquired during Ogoshi wear testing exhibits an inverse relationship with the wear resistance qualities of the material being tested. A material's wear resistance qualities are improved when its specific wear value decreases [46].

Nevertheless, this investigation revealed that SP-3 had the highest specific wear value. It can be asserted that SP-3 exhibits the lowest level of wear resistance. This phenomenon can be attributed to the positive correlation between the temperature applied and the surface roughness observed in the composite specimen. The findings of a study conducted by Jan et al. (2020) indicate that elevating the temperature of the mold leads to a corresponding increase in the roughness of the composite material. Their findings indicated that elevating the mold temperature led to a corresponding increase in the surface roughness of the brake pad [47]. The occurrence of increased surface roughness in the specimen can be attributed to the roughness of the composite surface when it comes into contact with the surface of the hard steel disc (used as test equipment). This contact leads to the formation of cracks on both the surface and subsurface of the specimen, which results in matrix delamination or wear [48]. This results in the removal of material in large flakes and creates various irregular edge shapes resulting in higher friction and wear [48].

3.5. Effect of Compression Molding Temperature on the thermal properties of brake pad composite specimens

TGA and DSC testing in this study was conducted to determine the thermal properties of the composite. Figure 5 shows that the composite brake pads specimen had several temperature variations during thermal decomposition that occurred in the temperature range of 30°C – 550°C. Weight loss on initial heating (30°C – 200°C) was caused by the evaporation of water on the rice husk. This happened because water was not chemically bound to the fiber. The reduction of fiber mass was further related to the degradation of hemicellulose. In the second range (200°C – 400°C), the weight loss that occurred was mostly due to the decomposition of hemicellulose and cellulose fibers and was followed by the decomposition of epoxy resin. In the last range (400°C – 550°C), the weight loss that occurred was caused by fiber degradation and decomposition of epoxy resin and filler.

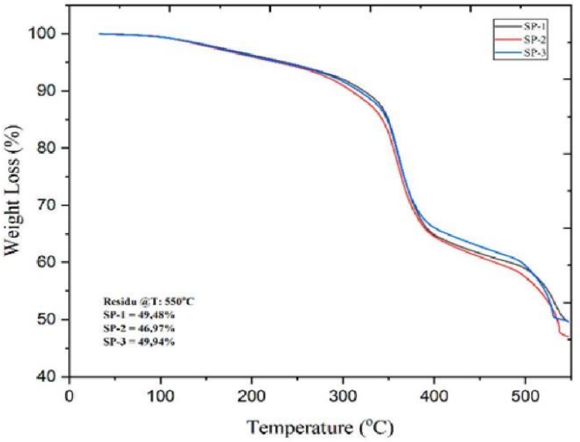

Figure 5: Effect of compression molding temperature on composite weight loss

This is in accordance with research conducted by Chen et al., (2020), rice husk experiences weight loss which is divided into three stages. At a temperature of 35°C – 150°C, rice husk undergoes evaporation of water in the fiber. At temperatures of 150°C – 380°C decomposition of hemicellulose, cellulose and lignin occurs and at temperature stages of 380°C – 600°C fiber degradation occurs [49].

The TGA test with a temperature scale of 30°C – 550°C resulted in a residue of 49.48% in the SP-1 specimen, 46.97% in the SP-2 specimen and 49.94% in the SP-3 specimen. The residual results in this study are in line with research by Li et al., (2020), the residual weight of the composite showed a tendency to increase with increasing molding temperature. The results of the TG curve analysis show that the right molding temperature will help the curing reaction and cross-linking of resin and fiber. The increased crosslink density of the composite will strengthen the thermal stability of the composite [50].

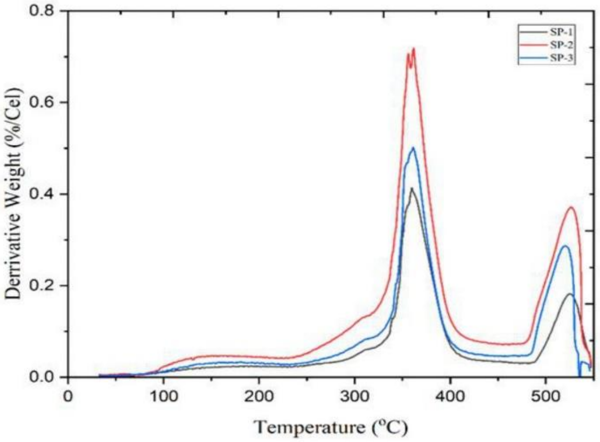

Figure 6: The effect of compression molding temperature on the derivative weight of the composite

Figure 6 shows the DTG (Derivative Thermal Gravimetry) curve for each composite brake pads specimen that experienced a maximum decomposition phase (Tmax) concerning temperature which was shown at the main peak. SP-1 specimen occurred at 359.98°C, SP-2 specimen at 356.13°C, and SP-3 specimen at 360.94°C. Maximum decomposition is the maximum weight loss on the specimen that occurs at a certain temperature (Tmax) which can be used as the most important indicator in determining the thermal stability of a material [51]. The results showed that there was an effect of the use of specimen molding temperature. The specimen with a molding temperature of 120°C had a larger Tmax and residue than other specimens. According to research by Li et al., (2020), by increasing the molding temperature, the durability of the epoxy resin and fiber activity tends to increase, which benefits the chemical bond between the fiber and the resin which results in increased composite cross-linking so that the degradation temperature will be higher [50].

Based on Figure 7, the exothermic phase occurred when heated to a temperature of 30°C – 550°C. On the DSC curve, Tg can be known from each specimen. The glass temperature is the transition temperature where the behavior of the composite transitions from hard glass to soft rubber [52]. The SP-1 specimen had a Tg of 311.25°C. The SP-2 specimen had a Tg of 309.18°C. The SP-3 specimen had a Tg of 310.06°C. After passing through the Tg material, each specimen began to form crystals. Specimens will experience cold crystallization; cold crystallization is a unique phenomenon in which crystallization that accompanies an exothermic anomaly occurs when a material is heated to a temperature below its melting point but above the temperature of its glass. On the curve, the peak point of the curve after going through the Tg phase is Tc. Temperature crystallization is a transition temperature where the formation of a crystal structure occurs due to heating [53].

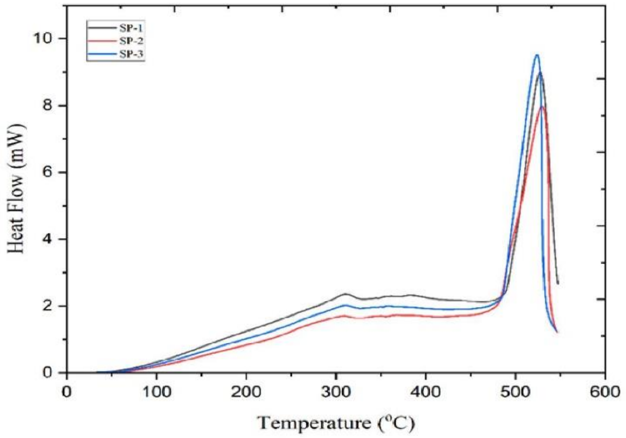

Figure 7: Effect of compression molding temperature on composite heat flow

The SP-1 specimen has the highest Tc of 526.17°C. The SP-2 specimen has the highest Tc of 529.83°C. The SP-3 specimen has the highest Tc of 523.17°C. The three specimens did not experience an endothermic phase, in which the specimens did not melt up to 550°C. On the DSC curve, Tm (Temperature melting) can be observed at the turning point of the curve. This is in line with Li et al., (2020) which stated that the first exothermic peak is closely related to the glass transition. Increased Tg and decreased peaks can be attributed to increased crosslinking and amorphism. The decreasing exothermic peak value of the composite indicates that the crosslink density of the mixture is increasing. It can be concluded that a reasonable increase in molding temperature will be conducive to increasing the crosslink density [50].

4. Conclusions

The effect of compression molding temperature on the characterization of asbestos-free composite friction materials for railway applications has been discussed in this study. Density, hardness, tensile, wear, TGA (Thermal Gravimetric Analysis), and DSC (Differential Scanning Calorimetry) tests were performed to evaluate the properties of the obtained specimens. According to the findings of this study, increasing the temperature during the compression molding process reduces the viscosity of the resin, allowing the resin to flow more easily and wet the reinforcement, filler, and abrasive materials. This leads to better bonding between the constituent materials, so the density of the composite specimen increases. The mechanical and thermal properties of the composite specimens increase as density increases. The results of this study show that Specimen SP-3 has better characteristics compared to other specimens. The density, Vickers hardness, tensile strength, and tensile modulus of SP-3 specimens are 1.70 g/cm^3, 108.2 HVN, 26.25 MPa, and 1.16 GPa, respectively. The highest thermal properties were generated by SP-3 specimen, with total residues, Tmax, Tg, and Tc values of 49.94%, 360.94°C, 310.06°C, and 517.17°C, respectively. Furthermore, the molding temperature has a significant effect on the specific wear on the composite specimen. Specific wear (x 10^{-7} mm^2/kg) on SP-1, SP-2, and SP-3 specimens was 9.64, 3.49, and 15.08, respectively.

Use of AI tools declaration

The authors declare they have not used Artificial Intelligence (AI) tools in the creation of this article.

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Conflict of Interest

The authors declare no conflict of interest

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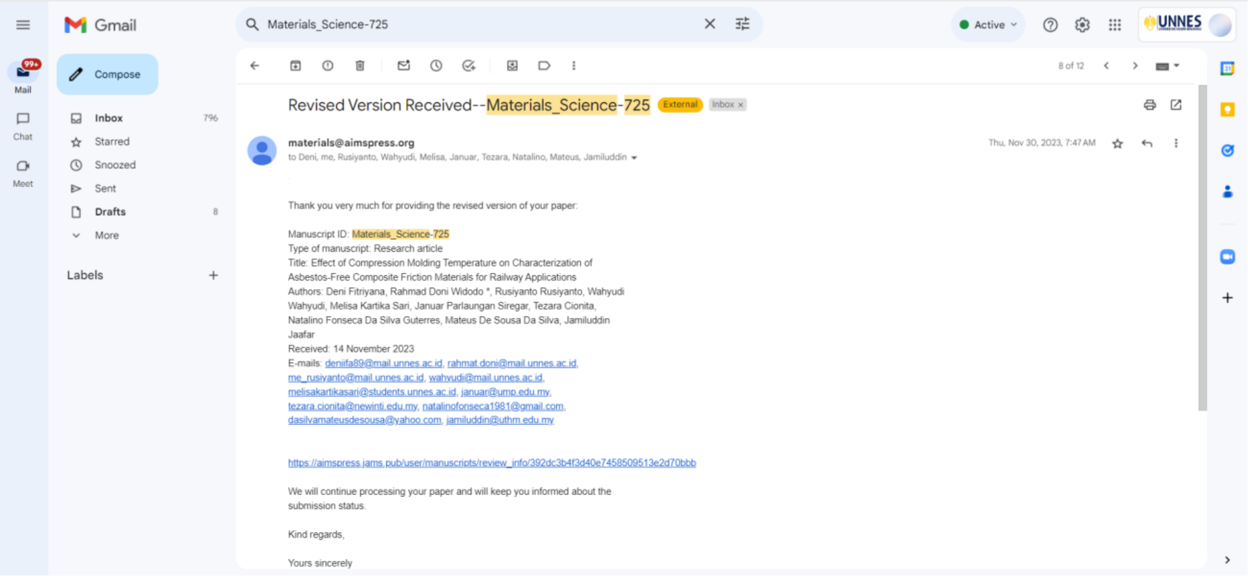

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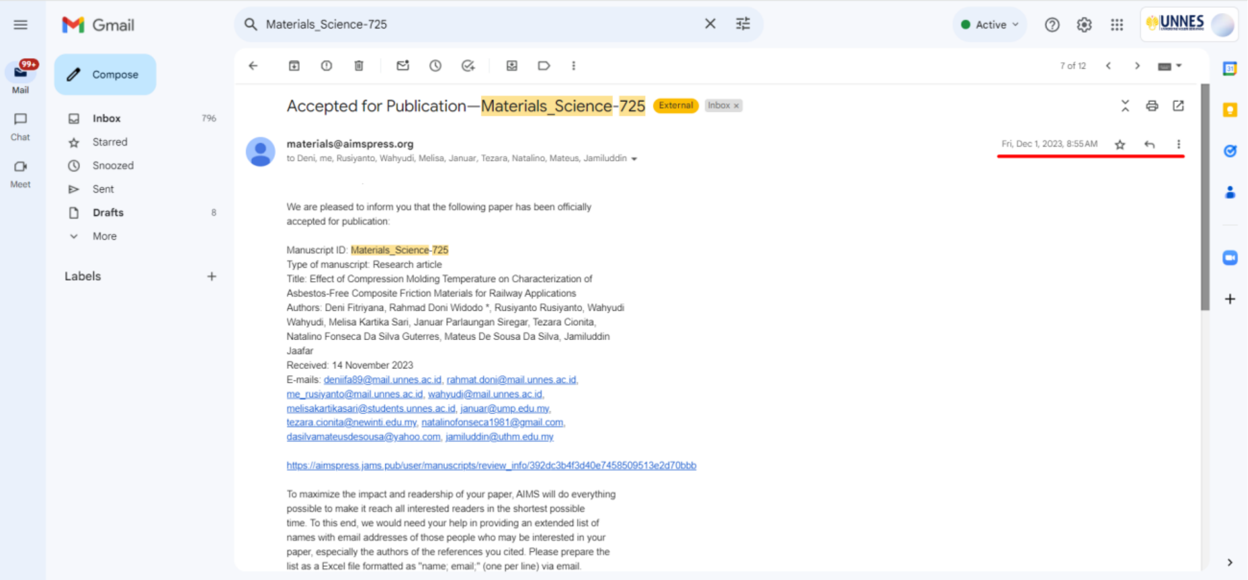

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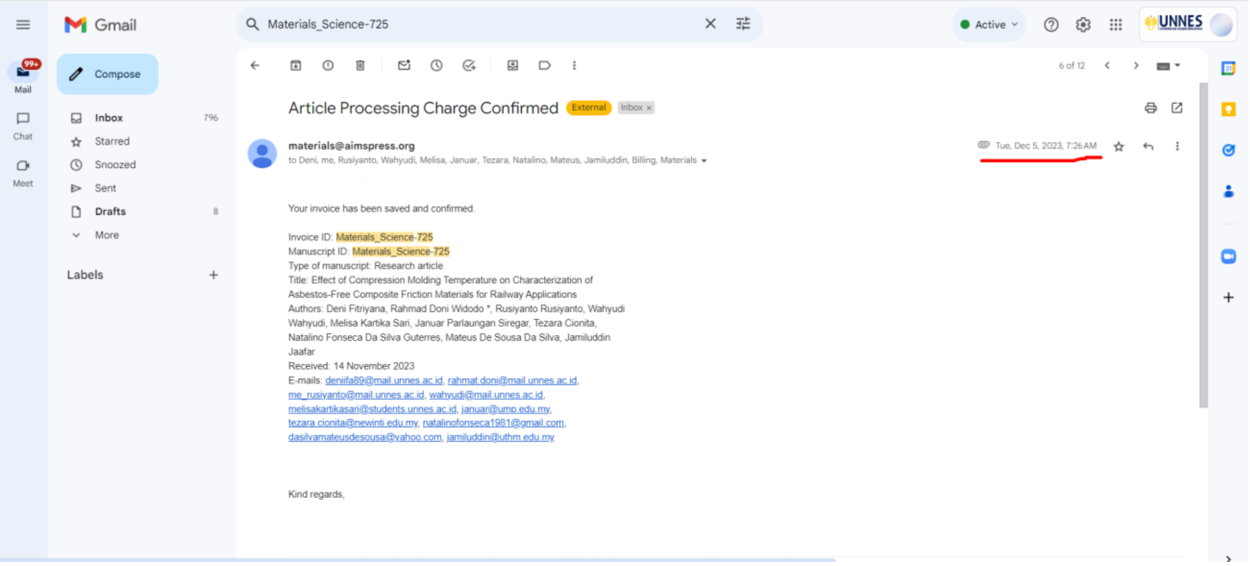

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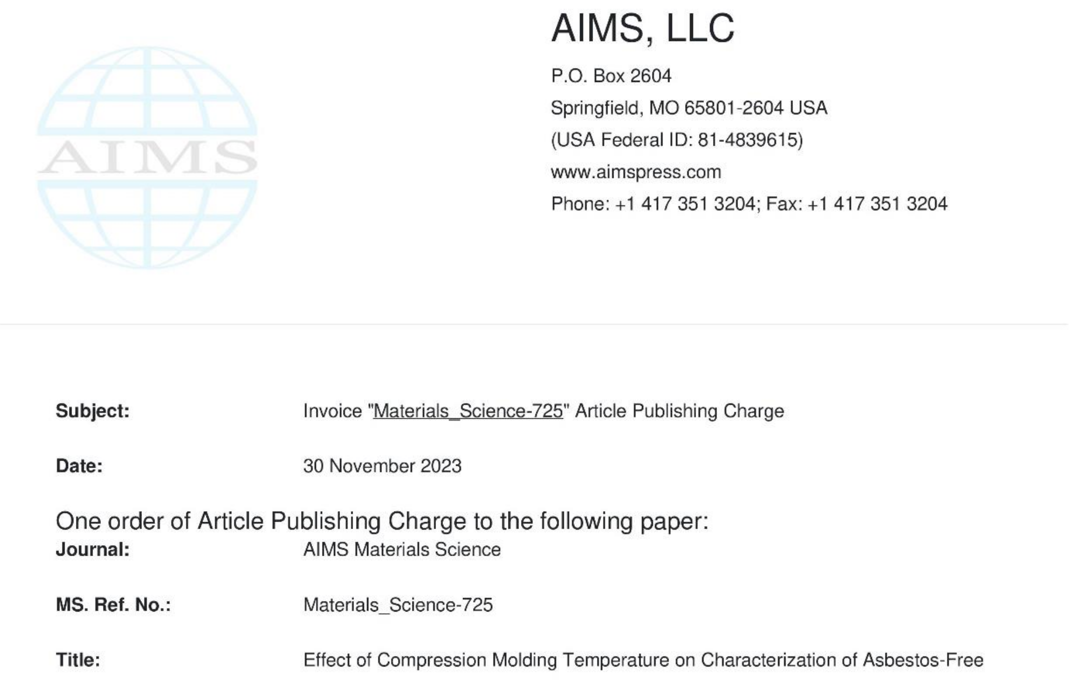

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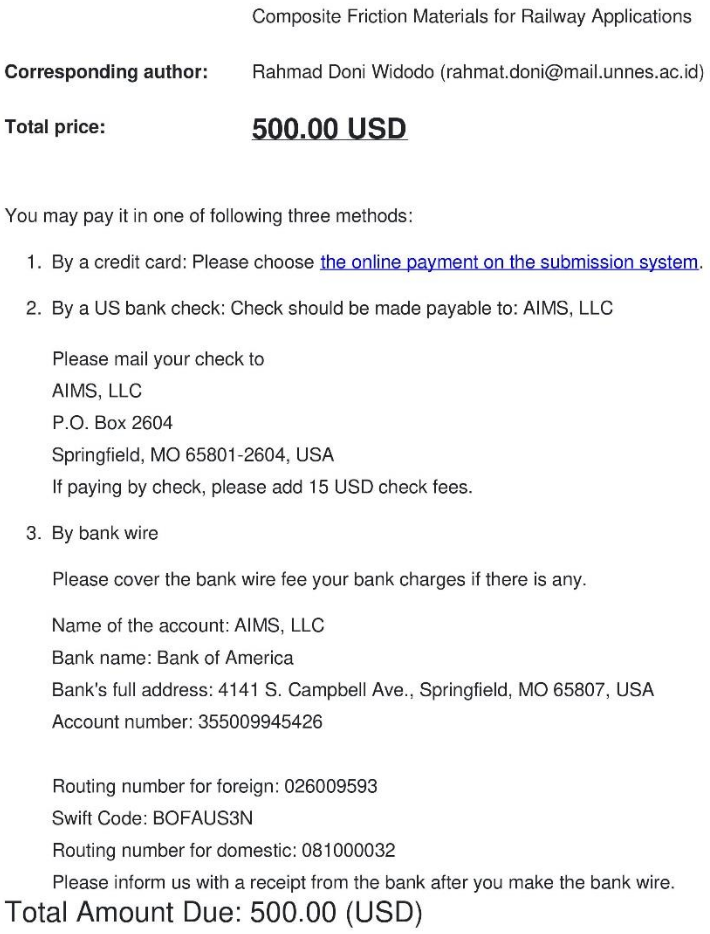

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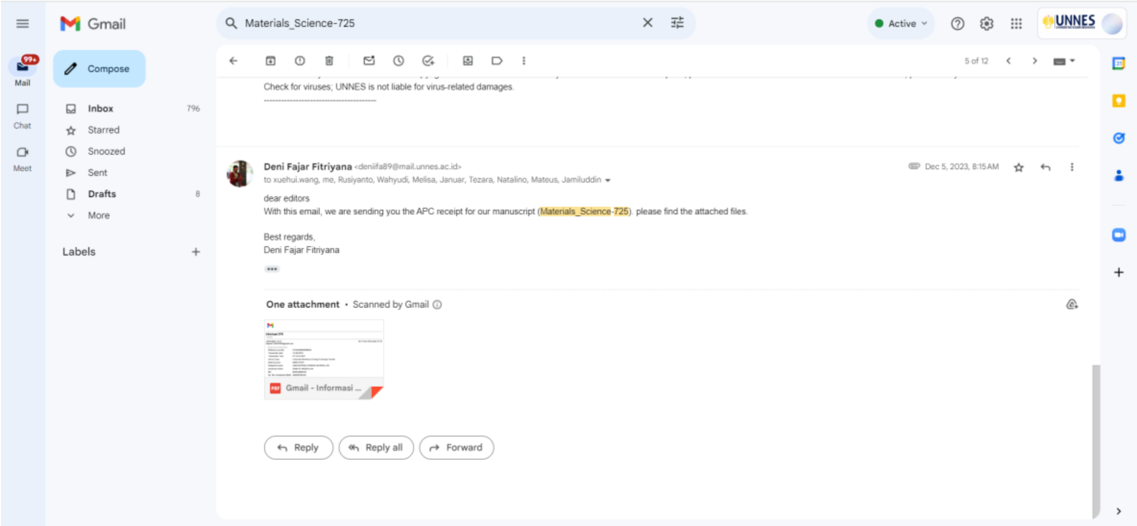

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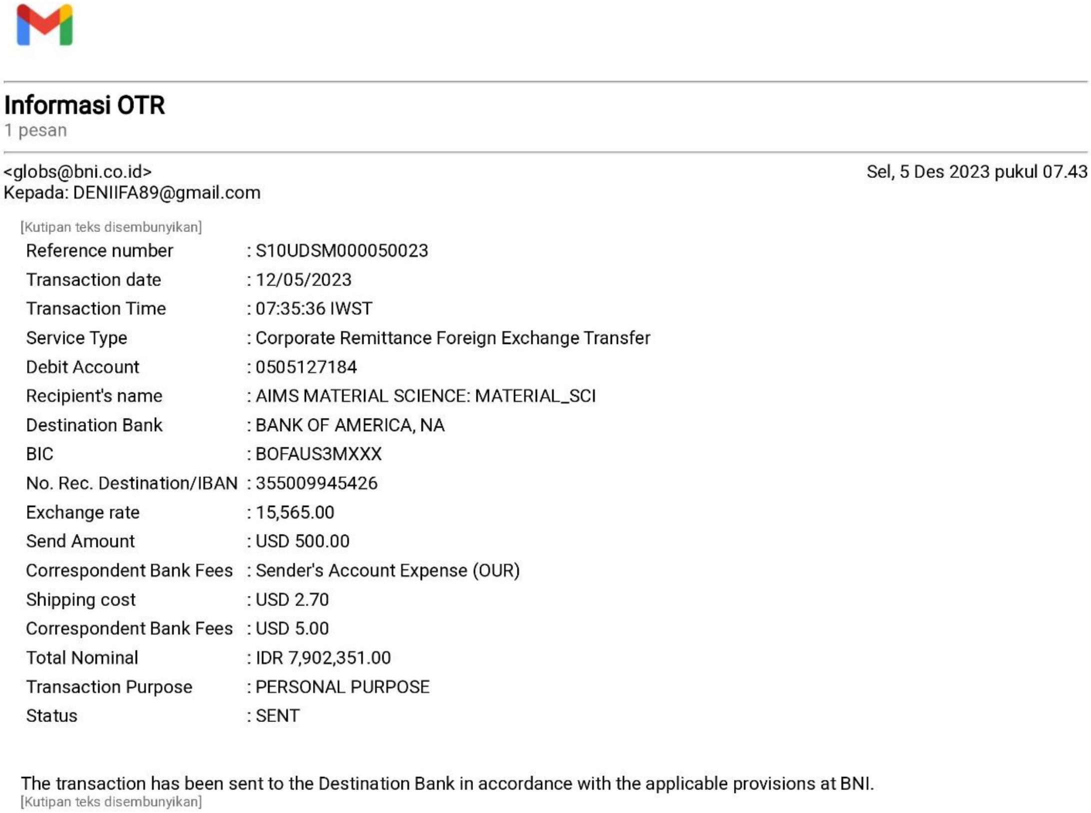

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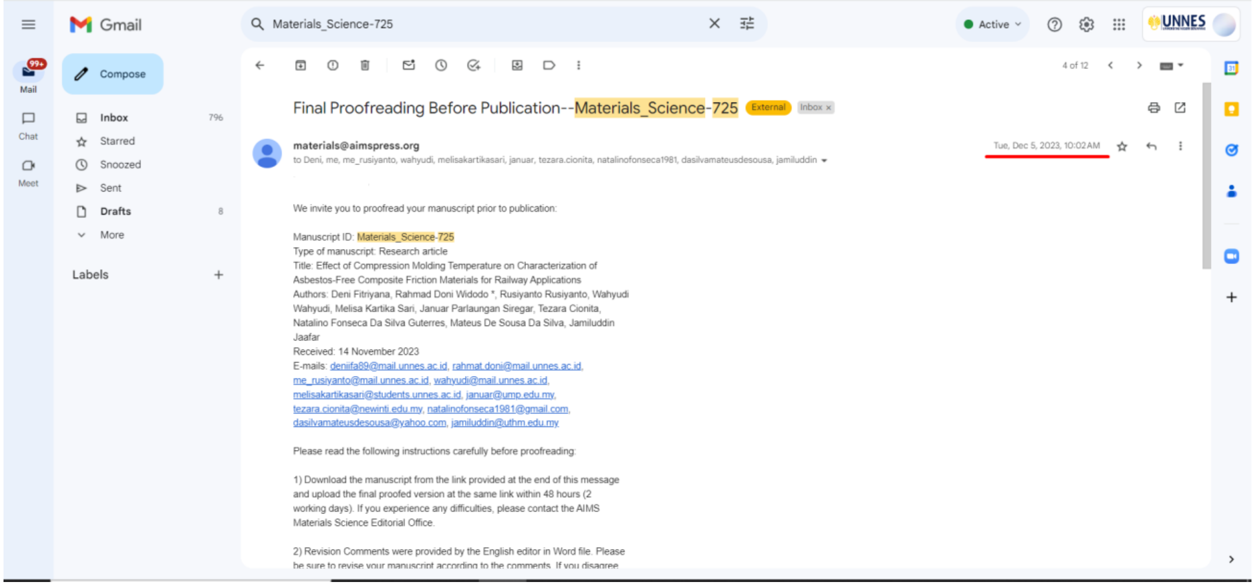

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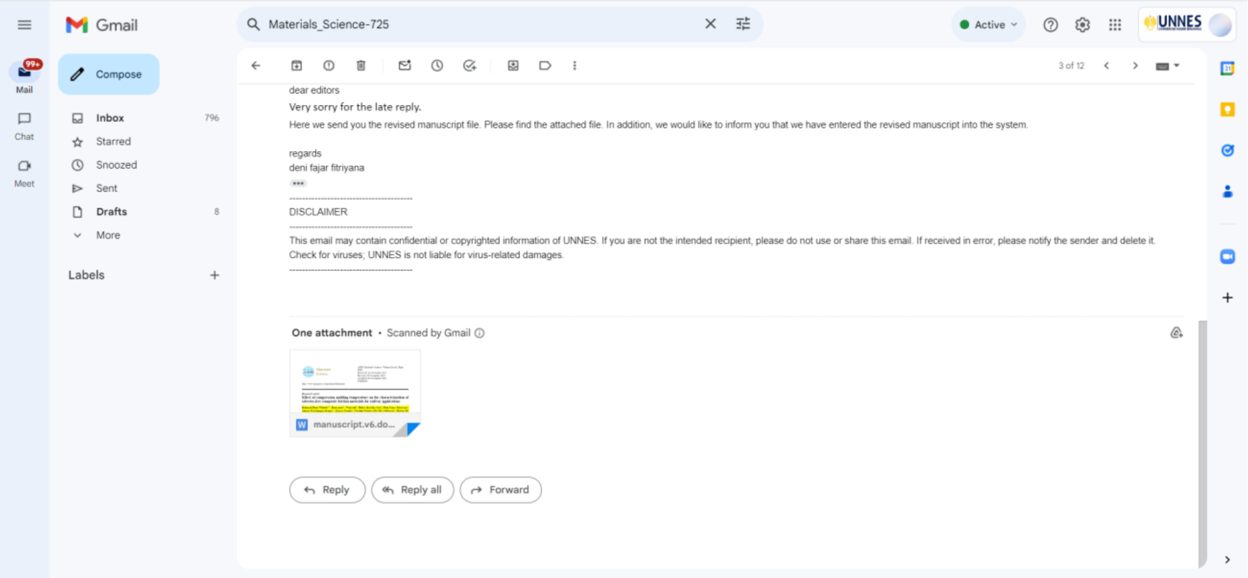

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Research article

Effect of compression molding temperature on the characterization of asbestos-free composite friction materials for railway applications

Rahmad Doni Widodo[1,*], Rusiyanto[21], Wahyudi[1], Melisa Kartika Sari[1], Deni Fajar Fitriyana[1], Januar Parlaungan Siregar [2], Tezara Cionita[3], Natalino Fonseca Da Silva Guterres[4], Mateus De Sousa Da Silva[4] and Jamiluddin Jaafar[5]

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Abstract: Brake pads significantly affect the braking performance of railways under both normal and emergency operating conditions. In previous studies, brake pads were made using the hand lay-up method and produced the best properties on specimens with epoxy, rice husk, Al_2O_3 and Fe_2O_3 compositions of 50%, 20%, 15% and 15%. However, the resulting density does not meet the density standard set by PT Industri Kereta Api Indonesia (PT INKA), which is 1.7–2.4 g~~r~~/cm^3. To date, there has been limited research into the utilization of the compression hot molding method for the production of asbestos-free composite friction materials composed of epoxy, rice husk, Al_2O_3 and Fe_2O_3 for railway applications. In this study, we aimed to determine the effect of compression molding temperature on the characterization of composite brake pads for railway applications. The brake pad specimens were made of epoxy resin, rice husk, Al_2O_3 and Fe_2O_3 with a composition of 50%, 20%, 15% and 15%, respectively. The manufacture of composites in this study used the compression molding method with a pressure of 20 MPa for 15 min~~utes~~ holding time. The mold temperature used were 80, 100, 120 °C.

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Density, hardness, tensile, wear, thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC) tests were performed to evaluate the properties of the specimens obtained. The results demonstrated that an increase in molding temperature improved the characterization of the brake pads, with the best results achieved at a molding temperature of ~~attained at~~ 120 °C (SP-3 specimen). SP-3 specimens had the best density, hardness, tensile properties and thermal properties compared to other specimens.

Keywords: brake pads; railways; composites; compression molding; friction materials

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1. Introduction

The effectiveness of brake pads has a significant impact on railways' capacity for stopping in an emergency as well as during normal operations [1]. Brake pads are used as components to ensure the safety of the railways during the braking process. Generally, brake pads on trains can be divided into organic and metal. Organic brake pads, which are composed of organic polymers, have been implemented in cars, trains and other modes of transportation. Metal-based brake pads have emerged as the preferred material for high-speed trains due to their outstanding friction capability, commendable resistance to wear, outstanding thermal conductivity and ability to withstand high operational temperatures [1,2].

Composite brake pads have been used in Indonesia since the last decade to replace cast iron brake pads for trains. Cast iron brake pads wear out faster when compared to composite brake pads [3]. In addition, metallic brake pads have a very high density, which can reduce the energy efficiency of the train system [4]. In recent years, the development of composite technology has made rapid progress with various innovations in the manufacture of brake pads using natural materials that have become waste and are no longer used. Composite itself is a material composed of a mixture of two or more materials with different mechanical properties to produce a new material that has different mechanical properties from its constituent materials. The properties of composite materials are a combination of the properties of its constituents, the matrix and reinforcement or filler. The matrix serves to transfer stress to the fiber, form coherent bonds, protect the fiber and remain stable after the manufacturing process. Reinforcement or filler materials must be able to support or improve the properties of the matrix in fabricating composite materials [5].

In previous studies, brake pads for applications on trains have been successfully made using the hand lay-up method. Brake pads with the best properties were found in specimens with a composition of epoxy, rice husk, Al_2O_3 and Fe_2O_3 of 50%, 20%, 15% and 15% respectively and a mesh size of 200. This composition produced density, hardness, tensile strength, specific wear value and degradation temperature of 1.23 g/cm^3, 81.2 HV~~N~~, 23.34 MPa, 8.67 × 10^{-7} N/mm^2 and 379 °C [6]. Moreover, when using 100 mesh rice husk, the density, hardness, specific wear value, and degradation temperature of the resulting composite brake friction material were 1.33 g/cm^3, 83.4 HV~~N~~, 10.8 ~~x~~× 10^{-7} N/mm^2 and 363.99 °C~~°C~~ [7]. However, the resulting density does not meet the standard density set by PT. ~~-~~Industri Kereta Api (INKA)~~,~~ which is 1.7–2.4 g~~r~~/cm^3 [8].

The hand layup method is a commonly employed technique in the fabrication of composite materials. Typically, the initial cost associated with fabricating composites using the hand layup technique is quite economical. Furthermore, this methodology enables the production of items with

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diverse geometries, structures and designs.

Nevertheless, the hand layup technique employed in composite manufacturing exhibits several limitations, including a reduced production speed and a decreased volume proportion of reinforcement. Furthermore, the use of this technique results in an uneven dispersion of reinforcing and matrix substances as a consequence of the inherent imprecision associated with handling by hand. As a result, producing high-quality composites in large quantities using the hand layup method is not possible [9–11].

Based on the description above, another method is needed to meet the characteristic requirements of brake pads for applications on trains. One method of fabricating composites that can produce better characteristics is compression molding. According to the findings of a study by Nyior et al. (2018), compression molding produced composites with better mechanical properties than the manual lay-up (hand lay-up) method. Their study found that the tensile strength and Young's modulus of samples made with compression molding were 77% and 47% higher than samples made by hand lay-up, respectively. The results also showed that the impact strength of the materials made by compression molding (11.5 kJ/m^2) was significantly higher than that of the samples made by hand lay-up, which had an impact strength of 7 kJ/m^2 [12].

In general, the components used in brake pads to create friction materials are the matrix or adhesive, reinforcements, fillers and abrasives. The proper combination of these components is critical to ensuring the efficient and reliable functioning of brake pads in various applications, such as brake pads for railways [13]. The high-speed trains usually use special brake block materials consisting of steel wool fiber, resin, aramid fiber, graphite, barium sulfate, magnesium, friction powder, mineral wool fiber, calcium carbonate powder, butyronitrile rubber powder, antimony sulfide and argil [14]. On the other hand, brake pads designed for high-speed and heavy trains are produced using phenolic resin, composite fibers, graphite, barium sulfate, iron powder, butyronitrile, zirconite, rubber powder, feldspar powder and alumina [15]. Moreover, the composite brake block for trains made by Green Power Runde Industry Co. Limited. consists of phenolic resin, nitrile rubber, steel fiber, reduced iron powder, graphite, etc. [16]. In this study, the fabrication of asbestos-free composite friction material specimens for railway applications utilizes a more straightforward combination of materials that include epoxy, rice husk, Al_2O_3 and Fe_2O_3. The roles of epoxy resin, rice husk, Al_2O_3 and Fe_2O_3 are as matrix [17], reinforcement [18], filler [19] and abrasive [20,21], respectively. The composite material as friction materials will be pressed and heated at a certain pressure and temperature. Furthermore, the fabrication of asbestos-free composite friction materials for railway applications made from epoxy, rice husk, Al_2O_3 and Fe_2O_3 by compression hot molding has not been widely studied. The pressure applied can make the composite tighter and denser. While the heating process makes the resin move to flow to fill the empty composite parts. Our purpose of this study was to determine the effect of compression molding temperature on the characterization of composite brake pads for applications on railways.

2. Materials and methods

2.1. Materials

The materials used in this study were epoxy, hardener, rice husk, aluminum oxide (Al_2O_3) and iron oxide (Fe_2O_3). The matrix used in this study was Bisphenol A-Epichlorohydrin epoxy resin as a binder, and Cycloaliphatic Amine type epoxy hardener obtained from the Justus store in Semarang, Indonesia.

In general, epoxy has a density, tensile strength and flexural strength of 1.18 g/cm^3, 63.7 and 8.3 MPa [22]. The rice husk used in this study was obtained from a rice mill near the campus. The composition of rice husk consists of cellulose (50%), lignin (25%–30%), silica (15%–20%) and the remainder consists of hemicellulose and water content [23]. In this study, Al_2O_3 and Fe_2O_3 were obtained from PT Merck Tbk, Indonesia.

2.2. Specimen fabrications

In this study, the fabrication of brake pad specimens used the compression molding method. The preparation of raw materials for the fabrication of brake pad specimens refers to previous research [6,7]. The rice husk was crushed using the FOMAC FCT-Z300 miller machine and sieved using a 200 mesh. Furthermore, the specimens were prepared by mixing the materials according to a predetermined concentration using a hand mixer in a plastic cup in stages. First, mixing epoxy and hardener with a ratio of 1:3 was carried out for 7 ~~minutes~~min. Then, a mixture of rice husk, aluminum oxide, iron oxide was added after being stirred for 5 ~~minutes~~min. Mixing was done again for 10 ~~minutes~~min. After all the ingredients were mixed, they were poured into the mold and left to harden at room temperature for up to 10 ~~hours~~h. Furthermore, the specimen was compressed with a pressure of 20 MPa for 15 ~~minutes~~ min and a temperature of 80, 100 and 120 °C (Table. 1). After that, the composite specimens were cut and characterized.

Table 1. Composite brake pads specimen code and compression molding setting parameters.

Specimen code	Pressure (MPa)	Holding time (min)	Temperature (°C)
SP-1	20	15	80
SP-2	20	15	100
SP-3	20	15	120

2.3. Testing and characterizations

In this study, the tests carried out were density, hardness, tensile and wear using the Ogoshi method, thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC). Density testing was based on ASTM D792 standard. Density testing was performed using an electronic density meter DME 220 series from Vibra Canada Inc. (Mississauga, ON, USA). This test is carried out by weighing the dry mass and the mass of the test object in water (wet mass). Vickers hardness testing in this study refers to the testing method carried out by [6,24]. Hardness testing was carried out using a Microhardness Tester F-800 machine (Future-Tech Corp., Kanagawa, Japan) with a test load of 25 gf and a dwell time of 10 ~~seconds~~s. Tensile testing was done according to the ASTM D638 standard using a HT-2402 computer servo control material testing machines from Hung Ta Instrument Co., Ltd., Samutprakarn, Thailand. The tensile test results consist of max force and elongation, which will be used to calculate the tensile strength and tensile modulus. Wear testing was performed using the O~~o~~ghosi high speed universal wear testing machine (Type OAT-U). The Ogoshi wear test was carried out with the width of the wear plate (B) = 3 mm, the radius of the wear plate (r) = 13.06 mm, the distance traveled during the wear process (10) = 66.6 m and the test load (F) = 2.12 kg. The TGA test

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was carried out based on the ASTM D6370 standard using the NEXTA STA test kit (Hitachi STA200RV with Real View Sample Observation). The temperature used in this test was 30–550 °C ~~°C~~. The testing process was conducted at a heating rate of 10 °C ~~°C~~/~~minute~~ min with a 100 m~~l~~L/~~minute~~ min nitrogen gas flow.

The test results obtain the temperature and weight loss values. The DSC test was carried out based on the ASTM D3418 standard using the NEXTA STA test tool (Hitachi STA200RV with Real View Sample Observation). The temperature used in this test was 30–550 °C ~~°C~~. The testing process was carried out at a heating rate of 10 °C /~~minute~~ min with a nitrogen gas flow of 100 mL~~l~~/~~minute~~min. The test results obtain the temperature and heat flow values of the composite specimens.

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3. Results and discussion

3.1. Effect of compression molding temperature on the density of brake pad composite specimens

Figure 1 shows the density of composite brake pad specimens with variations in molding temperature. The results of this study indicated that the increase in temperature affected the results of the composite density. The lowest density was shown in the SP-1 specimen, which was 1.~~,~~695 g/cm^3. While the highest density was shown in the SP-3 specimen, which was 1.~~,~~701 g/cm^3. The increase in density value occurs due to an increase in the temperature of composite fabrications with the compression molding method which causes a decrease in the viscosity of the resin, making it easier for the polymer to fill voids [25,26]. The fewer voids produced, the greater the density of the brake pad samples [6]. Void content is the percentage by volume of empty spaces or cavities inside composite materials [26]. When there are voids in a composite, they occupy space that would otherwise be occupied by the composite material. As a consequence, the composite's overall density decreases as the total mass is distributed over a larger volume [6,26].

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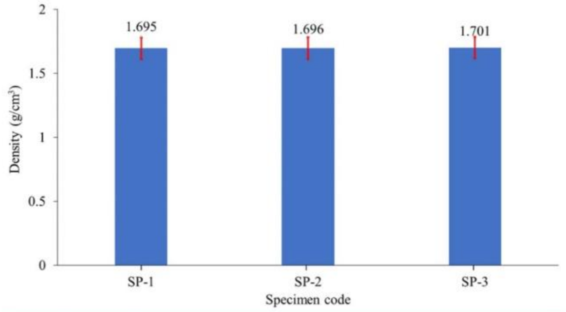

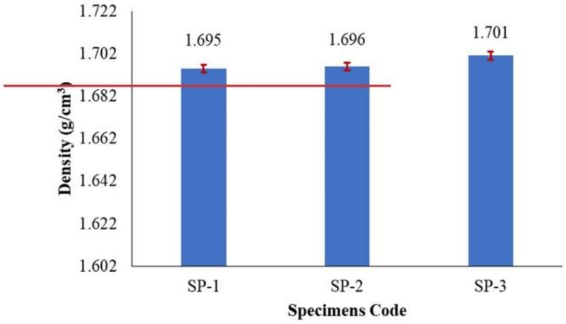

Figure 1. Effect of compression molding temperature on the density of brake pad composite specimens.

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In the manufacture of brake pad specimens using the compression molding method, an increase in molding temperature causes the viscosity of the resin to decrease so that the resin flows more easily and wets the reinforcing material. This results in a better bond between the resin, filler and reinforcement, resulting in an increase in the density of the composite specimen [6]. This is what causes SP-3 to have a higher density than other composite specimens. Incomplete resin flow into the desiccated area of reinforcement and filler materials can give rise to porosity in composite specimens. This is typically the result of increased resin viscosity due to exposure to ambient conditions and low-temperature curing cycles, resulting in inadequate flow. The lowered molding temperature reduces the driving force of gas-induced cavity growth, so that more porosity is produced, resulting in a decrease in the specimen's mechanical properties [26–29].

The results of this investigation are consistent with Ochi et al.'s (2015) research. Their findings demonstrated a correlation between the density of long bamboo fiber/ PLA Composites and the temperature of the mold. In general, the density of composites increases from 140 to 160 °C~~°C~~ as the molding temperature. However, when the molding temperature exceeds 160 °C~~°C~~, the density of the composite decreases. This occurs because mold temperatures above 160 °C reduce the matrix's viscosity and make it simpler for air to become trapped inside the material during the molding process, resulting in the formation of numerous voids [30]. In another study, Ochi et al. (2022) demonstrated the relationship between the density of bamboo fiber bundle-reinforced bamboo powder composite materials and the molding temperature. As the molding temperature increased, the density of the composite specimens remained relatively constant. The density of composites was between 1.41 and 1.42 g/cm^3 [31].

Based on these research results, only the SP-3 specimen met the minimum density determined by PT INKA, which was 1.7–2.4 g~~r~~/cm^3 [8]. Furthermore, the density of the SP-2 and SP-1 specimens almost met the specified density requirements. In addition, the density obtained in this study was higher than the results of previous studies. In previous research, brake pad composite fabrication used the hand layup method with various compositions. The highest densities produced in previous studies were

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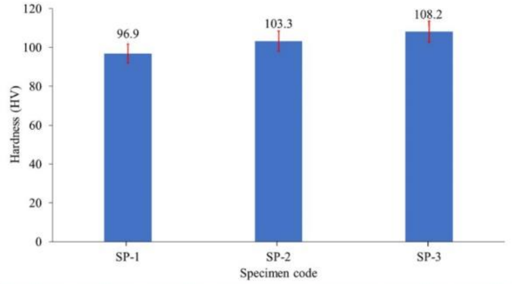
1.23 g/cm^3 [6] and 1.33 g/cm^3 [7]; whereas in this study, the densities of the specimens Sp-1, SP-2 and SP-3 were 1.~~,~~695, 1.~~,~~696 and 1.701 g/cm^3, respectively.

3.2. Effect of compression molding temperature on the hardness of brake pad composite specimens

The effect of temperature on the hardness of the composite is shown in Figure 2. The lowest hardness value was shown in the SP-1 specimen, which was 96.9 gf/mm^2 (HVN), while the highest density was shown in the SP-3 specimen, which was 108.2 gf/mm^2 (HVN). SP-3 and SP-1 specimens were the specimens with the highest and lowest densities produced in the study, respectively. In general, the hardness of composite specimens increases in proportion to their density. This is due to the fact that dense materials have strong interfacial bonding's between their matrix and reinforcement, making them more resistant to indentation and plastic deformation [6,32].

Previous research reached the same conclusion, which is that the specimen's higher density increased its hardness [33–36]. Research conducted by Fouly et al. (2021) showed that increasing the density of the composite causes an increase in the hardness of the specimen. Therefore, the average hardness also increases with increasing density. The poly(methyl methacrylate) (PMMA) nanocomposites specimen reinforced with 8 wt.% hydroxyapatite (PMHA8) specimen obtained a maximum hardness of 87.7 D-index. This happened because the PMHA8 specimen had the highest density compared to the other specimens [32]. Furthermore, an increase in the composite is hardness indicates a good interfacial bond between the matrix and the reinforcing fiber. The stronger the interfacial bond between the matrix and the reinforcing fiber, the higher the hardness of the resulting composite specimen [37,38]. Yawas et al. obtained the maximum hardness and density of non-asbestos brake pad samples using a compaction load and sintering temperature of 15t ~~tonnes~~ and 150 °C~~ºC~~, respectively. This happens because using these variations results in a more uniform distribution of frictional filler material particles in the matrix. Furthermore, this variation increases the surface area, which improves the matrix's bonding ability with frictional filler material [39].



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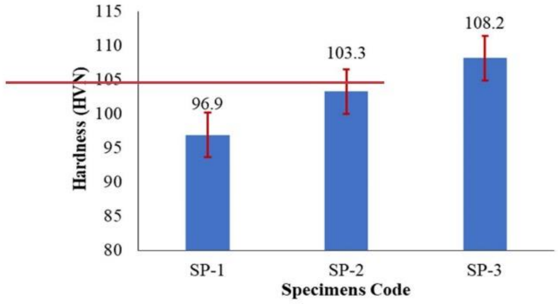

Figure 2. Effect of compression molding temperature on the hardness of brake pad composite specimens.

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3.3. Effect of compression molding temperature on the tensile strength of brake pad composite specimens

The effect of temperature on the tensile strength of the composite is shown in Figure 3. The lowest tensile strength value was shown in the SP-1 specimen, which was 7.26 MPa, while the highest tensile strength was shown in the SP-3 specimen, which was 26.22 MPa.

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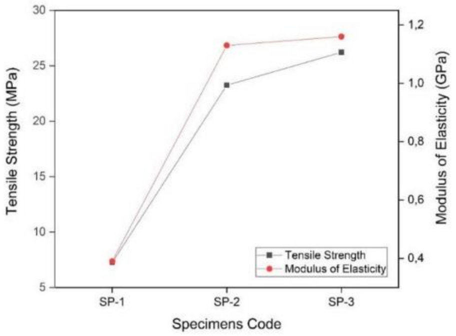

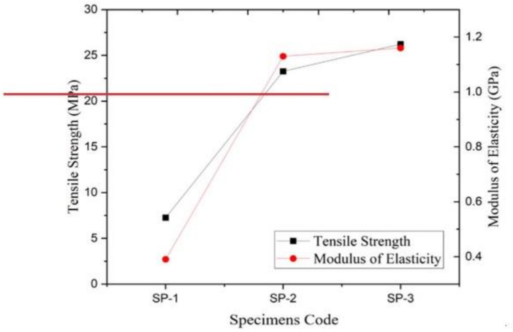

Figure 3. Effect of temperature on tensile strength and tensile modulus of brake pad composite specimens.

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The increase in the value of tensile strength is affected by the behavior of the bond between the resin and fiber interfaces at each increasing compression molding temperature variation [40]. This is in line with research conducted by ~~Sumesh & Kanthavel (2020)~~ Sumesh et al. (2020). Composites with an epoxy resin matrix formed by the compression molding method produce the best mechanical properties of tensile strength at higher temperatures [41].

Heating at high temperatures facilitates the mobilization of the resin in fiber impregnation. The increase in tensile strength is also due to an increase in mold temperature, which reduces the viscosity of the matrix which has an impact on reducing voids in the composite [42].

The increase in tensile strength may also be caused by the main process that occurs at a molding temperature of 80–120 °C for the evaporation. The higher the molding temperature, the higher the moisture content in the evaporating fiber, therefore the higher the tensile strength. Mvondo et al.~~,~~ (2017)~~,~~ stated that there was a negative relationship between the moisture content of tropical wood fiber and its tensile strength. Differences in the percentage of water content in fiber were studied. In fibers that have a lower water content produces high tensile strength, while high water content produces low tensile strength [43]. Figure 3 shows that the modulus of elasticity had increased with increasing molding temperature. The modulus of elasticity represents the inflexibility of a material [44,45]. The higher the value of the modulus of elasticity is, the less flexible the material. At higher molding temperatures, the specimen becomes stiffer so that the modulus value is higher [18,19]. In accordance with research conducted by Ochi et al. (2022) [31], the value of the elastic modulus increased with increasing temperature used in the specimen molding process using the compression molding method.

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3.4. Effect of compression molding temperature on the specific wear of brake pad composite specimens

The wear test results showed that the use of higher mold temperatures can cause a decrease in the wear resistance of the composite (Figure 4). A higher specific wear value indicated a lower wear resistance property. In the study, the SP-3 specimen had the highest specific wear value of 15.08 × ~~x~~

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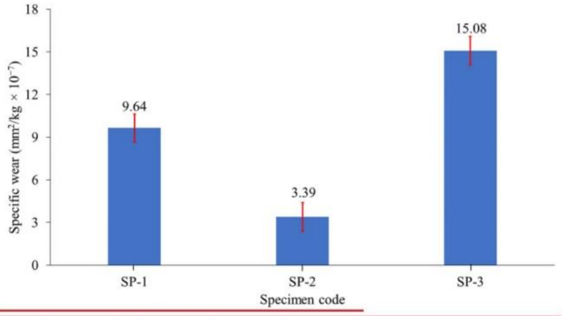
10^{-7} mm^2/kg while the SP-2 specimen had the lowest specific wear value. Thus, it can be said that the highest wear resistance was found in the SP-2 specimen. According Günay et al. (2020), a negative correlation exists between a material's hardness value and the specific wear value it exhibits. Materials possessing high hardness exhibit enhanced resistance to wear and tear, hence endowing the test specimens with excellent wear resistance.



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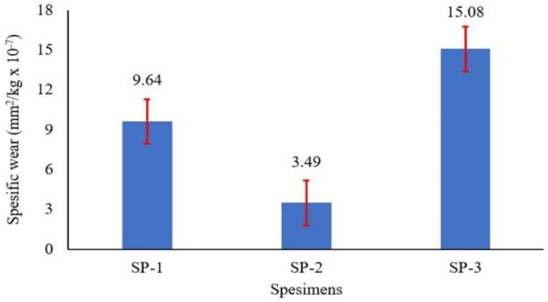

Figure 4. Effect of compression molding temperature on the specific wear of brake pad composite specimens.

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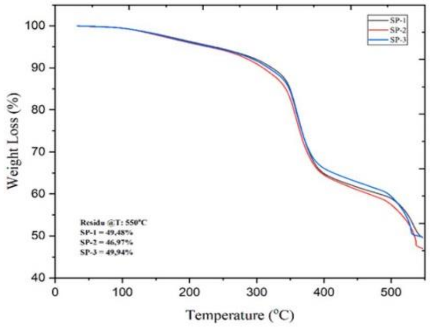
The reason for this phenomenon is that the wear value acquired during Ogoshi wear testing exhibits an inverse relationship with the wear resistance qualities of the material being tested. A material's wear resistance qualities are improved when its specific wear value decreases [46].

Nevertheless, this investigation revealed that SP-3 had the highest specific wear value. It can be asserted that SP-3 exhibits the lowest level of wear resistance. This phenomenon can be attributed to the positive correlation between the temperature applied and the surface roughness observed in the composite specimen. The findings of a study conducted by Jan et al. (2020) indicate that elevating the temperature of the mold leads to a corresponding increase in the roughness of the composite material. Their findings indicated that elevating the mold temperature led to a corresponding increase in the surface roughness of the brake pad [47]. The occurrence of increased surface roughness in the specimen can be attributed to the roughness of the composite surface when it comes into contact with the surface of the hard steel disc (used as test equipment). This contact leads to the formation of cracks on both the surface and subsurface of the specimen, which results in matrix delamination or wear [48]. This results in the removal of material in large flakes and creates various irregular edge shapes resulting in higher friction and wear [48].

3.5. Effect of compression molding temperature on the thermal properties of brake pad composite specimens

TGA and DSC testing in this study was conducted to determine the thermal properties of the composite. Figure 5 shows that the composite brake pads specimen had several temperature variations during thermal decomposition that occurred in the temperature range of 30–550 °C. Weight loss on initial heating (30–200 °C) was caused by the evaporation of water on the rice husk. This happened because water was not chemically bound to the fiber. The reduction of fiber mass was further related to the degradation of hemicellulose. In the second range (200–400 °C), the weight loss that occurred was mostly due to the decomposition of hemicellulose and cellulose fibers and was followed by the decomposition of epoxy resin. In the last range (400–550 °C), the weight loss that occurred was caused by fiber degradation and decomposition of epoxy resin and filler.



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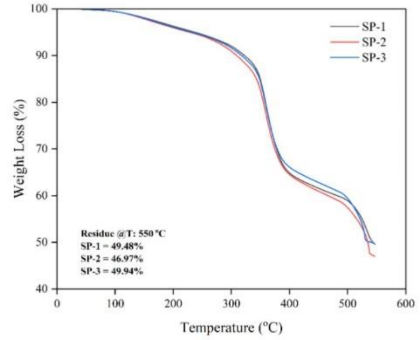

Figure 5. Effect of compression molding temperature on composite weight loss.

This is in accordance with research conducted by ~~Chen et al., (2020)~~ Chen et al. (2020), rice husk experiences weight loss which is divided into three stages. At a temperature of 35–150 °C, rice husk undergoes evaporation of water in the fiber. At temperatures of 150–380 °C decomposition of hemicellulose, cellulose and lignin occurs and at temperature stages of 380–600 °C fiber degradation occurs [49]. The TGA test with a temperature scale of 30–550 °C resulted in a residue of 49.48% in the SP-1 specimen, 46.97% in the SP-2 specimen and 49.94% in the SP-3 specimen. The residual results in this study are in line with research by Li et al., (2020), the residual weight of the composite showed a tendency to increase with increasing molding temperature. The results of the TGA curve analysis show that the right molding temperature will help the curing reaction and cross-linking of resin and fiber. The increased crosslink density of the composite will strengthen the thermal stability of the composite [50].

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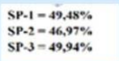
SP-1 = 49,48%
SP-2 = 46,97%
SP-3 = 49,94%

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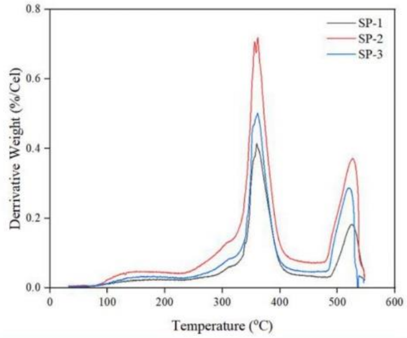

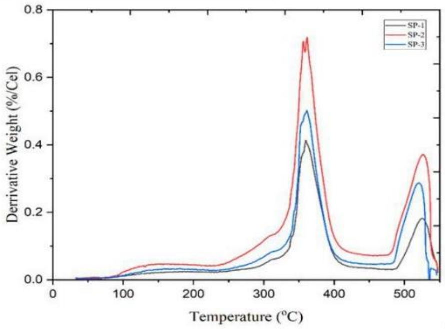

Figure 6. The effect of compression molding temperature on the derivative weight of the composite.

Figure 6 shows the derivative thermal gravimetry (DTG) curve for each composite brake pads specimen that experienced a maximum decomposition phase (Tmax) concerning temperature, which was shown at the main peak. SP-1 specimen occurred at 359.98 °C, SP-2 specimen at 356.13 °C and SP-3 specimen at 360.94 °C. Maximum decomposition is the maximum weight loss on the specimen that occurs at a certain temperature (Tmax), which can be used as the most important indicator in determining the thermal stability of a material [51]. The results showed that there was an effect of the use of specimen molding temperature. The specimen with a molding temperature of 120 °C had a larger Tmax and residue than other specimens. According to research by ~~Li et al., (2020)~~ Li et al. (2020), by increasing the molding temperature, the durability of the epoxy resin and fiber activity tends to increase, which benefits the chemical bond between the fiber and the resin which results in increased composite

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cross-linking so that the degradation temperature will be higher [50].

Based on Figure 7, the exothermic phase occurred when heated to a temperature of 30–550 °C. On the DSC curve, glass transition temperature (Tg) ~~Tg~~ can be known from each specimen. The glass temperature is the transition temperature where the behavior of the composite transitions from hard glass to soft rubber [52]. The SP-1 specimen had a Tg of 311.25 °C. The SP-2 specimen had a Tg of 309.18 °C. The SP-3 specimen had a Tg of 310.06 °C. After passing through the Tg material, each specimen began to form crystals. Specimens will experience cold crystallization; cold crystallization is a unique phenomenon in which crystallization that accompanies an exothermic anomaly occurs when a material is heated to a temperature below its melting point but above the temperature of its glass. On the curve, the peak point of the curve after going through the Tg phase is crystallization temperature (Tc)~~Tc~~. Temperature crystallization is a transition temperature where the formation of a crystal structure occurs due to heating [53].

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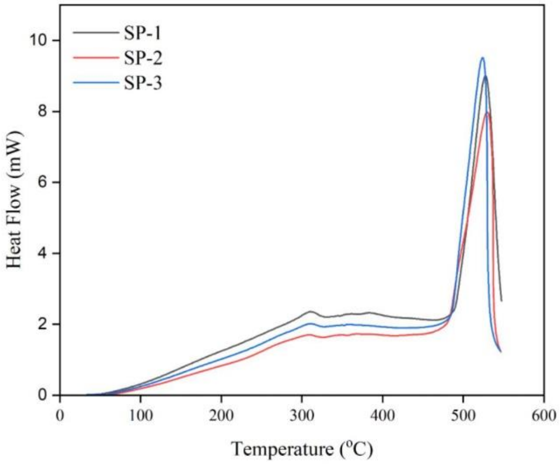

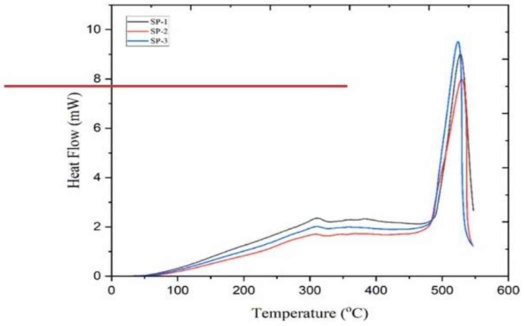

Figure 7. Effect of compression molding temperature on composite heat flow.

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The SP-1 specimen has the highest Tc of 526.17 °C~~°C~~. The SP-2 specimen has the highest Tc of 529.83 °C. The SP-3 specimen has the highest Tc of 523.17 °C. The three specimens did not experience an endothermic phase, in which the specimens did not melt up to 550 °C. On the DSC curve, temperature melting (Tm) can be observed at the turning point of the curve. This is in line with Li et al. (2020), which stated that the first exothermic peak is closely related to the glass transition. Increased Tg and decreased peaks can be attributed to increased crosslinking and amorphism. The decreasing exothermic peak value of the composite indicates that the crosslink density of the mixture is increasing. It can be concluded that a reasonable increase in molding temperature will be conducive to increasing the crosslink density [50].

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4. Conclusions

The effect of compression molding temperature on the characterization of asbestos-free composite friction materials for railway applications has been discussed in this study. Density, hardness, tensile, wear, TGA and DSC tests were performed to evaluate the properties of the obtained specimens. According to our findings, increasing the temperature during the compression molding process reduces the viscosity of the resin, allowing the resin to flow more easily and wet the reinforcement, filler and abrasive materials. This leads to better bonding between the constituent materials, so the density of the composite specimen increases. The mechanical and thermal properties of the composite specimens increase as density increases. The results of this study show that ~~Specimen~~ specimen SP-3 has better characteristics compared to other specimens. The density, Vickers hardness, tensile strength and tensile modulus of SP-3 specimens are 1.70 g/cm^3, 108.2 HVN, 26.25 MPa and 1.16 GPa, respectively. The highest thermal properties were generated by SP-3 specimen, with total residues, Tmax, Tg and Tc values of 49.94%, 360.94 °C~~ºC~~, 310.06 °C~~ºC~~ and 517.17 °C~~ºC~~, respectively. Furthermore, the molding temperature has a significant effect on the specific wear on the composite specimen. Specific wear (× ~~x~~ 10^{-7} mm^2/kg) on SP-1, SP-2 and SP-3 specimens were 9.64, 3.49 and 15.08, respectively.

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Use of AI tools declaration

The authors declare that they have not used Artificial Intelligence (AI) tools in the creation of this article.

Acknowledgments

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Conflict of interest

The authors declare no conflicts of interest.

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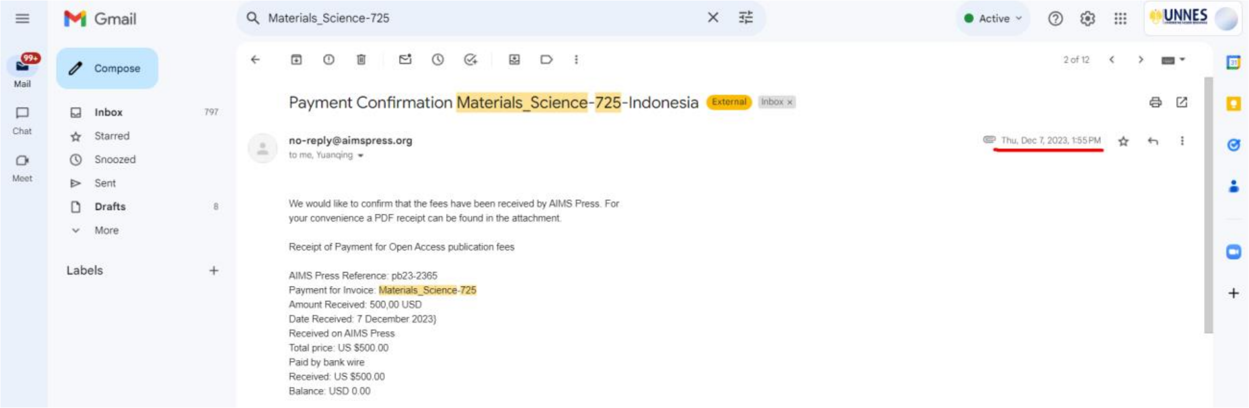

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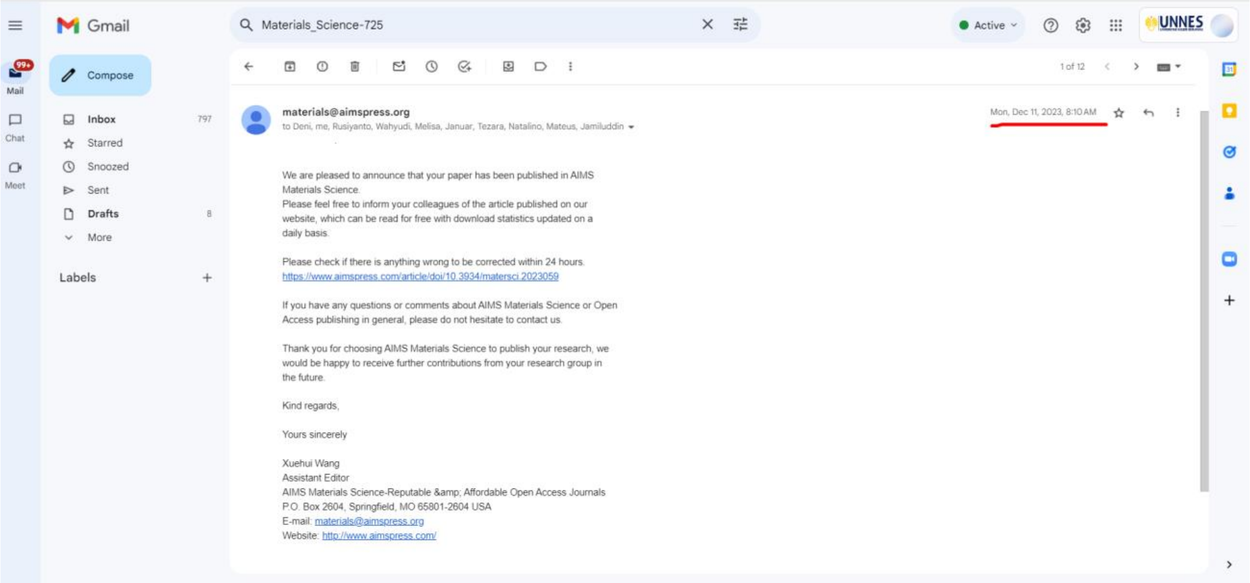

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Research article

Effect of compression molding temperature on the characterization of asbestos-free composite friction materials for railway applications

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Abstract: Brake pads significantly affect the braking performance of railways under both normal and emergency operating conditions. In previous studies, brake pads were made using the hand lay-up method and produced the best properties on specimens with epoxy, rice husk, Al_2O_3 and Fe_2O_3 compositions of 50%, 20%, 15% and 15%. However, the resulting density does not meet the density standard set by PT Industri Kereta Api Indonesia (PT INKA), which is 1.7–2.4 g/cm^3. To date, there has been limited research into the utilization of the compression hot molding method for the production of asbestos-free composite friction materials composed of epoxy, rice husk, Al_2O_3 and Fe_2O_3 for railway applications. In this study, we aimed to determine the effect of compression molding temperature on the characterization of composite brake pads for railway applications. The brake pad specimens were made of epoxy resin, rice husk, Al_2O_3 and Fe_2O_3 with a composition of 50%, 20%, 15% and 15%, respectively. The manufacture of composites in this study used the compression molding method with a pressure of 20 MPa for 15 min holding time. The mold temperature used were 80, 100, 120 °C. Density, hardness, tensile, wear, thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC) tests were performed to evaluate the properties of the specimens obtained. The results demonstrated that an increase in molding temperature improved the characterization of the brake pads, with the best results achieved at a molding temperature of 120 °C (SP-3 specimen). SP-3 specimens had the best density, hardness, tensile properties and thermal properties compared to other specimens.

Keywords: brake pads; railways; composites; compression molding; friction materials

1. Introduction

The effectiveness of brake pads has a significant impact on railways' capacity for stopping in an emergency as well as during normal operations [1]. Brake pads are used as components to ensure the safety of the railways during the braking process. Generally, brake pads on trains can be divided into organic and metal. Organic brake pads, which are composed of organic polymers, have been implemented in cars, trains and other modes of transportation. Metal-based brake pads have emerged as the preferred material for high-speed trains due to their outstanding friction capability, commendable resistance to wear, outstanding thermal conductivity and ability to withstand high operational temperatures [1,2].

Composite brake pads have been used in Indonesia since the last decade to replace cast iron brake pads for trains. Cast iron brake pads wear out faster when compared to composite brake pads [3]. In addition, metallic brake pads have a very high density, which can reduce the energy efficiency of the train system [4]. In recent years, the development of composite technology has made rapid progress with various innovations in the manufacture of brake pads using natural materials that have become waste and are no longer used. Composite itself is a material composed of a mixture of two or more materials with different mechanical properties to produce a new material that has different mechanical properties from its constituent materials. The properties of composite materials are a combination of the properties of its constituents, the matrix and reinforcement or filler. The matrix serves to transfer stress to the fiber, form coherent bonds, protect the fiber and remain stable after the manufacturing process. Reinforcement or filler materials must be able to support or improve the properties of the matrix in fabricating composite materials [5].

In previous studies, brake pads for applications on trains have been successfully made using the hand lay-up method. Brake pads with the best properties were found in specimens with a composition of epoxy, rice husk, Al_2O_3 and Fe_2O_3 of 50%, 20%, 15% and 15% respectively and a mesh size of 200. This composition produced density, hardness, tensile strength, specific wear value and degradation temperature of 1.23 g/cm^3, 81.2 HV, 23.34 MPa, 8.67×10^{-7} N/mm^2 and 379 °C [6]. Moreover, when using 100 mesh rice husk, the density, hardness, specific wear value, and degradation temperature of the resulting composite brake friction material were 1.33 g/cm^3, 83.4 HV, 10.8×10^{-7} N/mm^2 and 363.99 °C [7]. However, the resulting density does not meet the standard density set by PT. Industri Kereta Api (INKA), which is 1.7–2.4 g/cm^3 [8].

The hand layup method is a commonly employed technique in the fabrication of composite materials. Typically, the initial cost associated with fabricating composites using the hand layup technique is quite economical. Furthermore, this methodology enables the production of items with diverse geometries, structures and designs.

Nevertheless, the hand layup technique employed in composite manufacturing exhibits several limitations, including a reduced production speed and a decreased volume proportion of reinforcement. Furthermore, the use of this technique results in an uneven dispersion of reinforcing and matrix substances as a consequence of the inherent imprecision associated with handling by hand. As a result, producing high-quality composites in large quantities using the hand layup method is not possible [9–11].

Based on the description above, another method is needed to meet the characteristic requirements of brake pads for applications on trains. One method of fabricating composites that can produce better characteristics is compression molding. According to the findings of a study by Nyior et al. (2018), compression molding produced composites with better mechanical properties than the manual lay-up (hand lay-up) method. Their study found that the tensile strength and Young's modulus of samples made with compression molding were 77% and 47% higher than samples made by hand lay-up, respectively. The results also showed that the impact strength of the materials made by compression molding (11.5 kJ/m^2) was significantly higher than that of the samples made by hand lay-up, which had an impact strength of 7 kJ/m^2 [12].

In general, the components used in brake pads to create friction materials are the matrix or adhesive, reinforcements, fillers and abrasives. The proper combination of these components is critical to ensuring the efficient and reliable functioning of brake pads in various applications, such as brake pads for railways [13]. The high-speed trains usually use special brake block materials consisting of steel wool fiber, resin, aramid fiber, graphite, barium sulfate, magnesium, friction powder, mineral wool fiber, calcium carbonate powder, butyronitrile rubber powder, antimony sulfide and argil [14]. On the other hand, brake pads designed for high-speed and heavy trains are produced using phenolic resin, composite fibers, graphite, barium sulfate, iron powder, butyronitrile, zirconite, rubber powder, feldspar powder and alumina [15]. Moreover, the composite brake block for trains made by Green Power Runde Industry Co. Limited. consists of phenolic resin, nitrile rubber, steel fiber, reduced iron powder, graphite, etc. [16]. In this study, the fabrication of asbestos-free composite friction material specimens for railway applications utilizes a more straightforward combination of materials that include epoxy, rice husk, Al_2O_3 and Fe_2O_3. The roles of epoxy resin, rice husk, Al_2O_3 and Fe_2O_3 are as matrix [17], reinforcement [18], filler [19] and abrasive [20,21], respectively. The composite material as friction materials will be pressed and heated at a certain pressure and temperature. Furthermore, the fabrication of asbestos-free composite friction materials for railway applications made from epoxy, rice husk, Al_2O_3 and Fe_2O_3 by compression hot molding has not been widely studied. The pressure applied can make the composite tighter and denser. While the heating process makes the resin move to flow to fill the empty composite parts. Our purpose of this study was to determine the effect of compression molding temperature on the characterization of composite brake pads for applications on railways.

2. Materials and methods

2.1. Materials

The materials used in this study were epoxy, hardener, rice husk, aluminum oxide (Al_2O_3) and iron oxide (Fe_2O_3). The matrix used in this study was Bisphenol A-Epichlorohydrin epoxy resin as a binder, and Cycloaliphatic Amine type epoxy hardener obtained from the Justus store in Semarang, Indonesia.

In general, epoxy has a density, tensile strength and flexural strength of 1.18 g/cm^3, 63.7 and 8.3 MPa [22]. The rice husk used in this study was obtained from a rice mill near the campus. The composition of rice husk consists of cellulose (50%), lignin (25%–30%), silica (15%–20%) and the remainder consists of hemicellulose and water content [23]. In this study, Al_2O_3 and Fe_2O_3 were obtained from PT Merck Tbk, Indonesia.

2.2. Specimen fabrications

In this study, the fabrication of brake pad specimens used the compression molding method. The preparation of raw materials for the fabrication of brake pad specimens refers to previous research [6,7]. The rice husk was crushed using the FOMAC FCT-Z300 miller machine and sieved using a 200 mesh. Furthermore, the specimens were prepared by mixing the materials according to a predetermined concentration using a hand mixer in a plastic cup in stages. First, mixing epoxy and hardener with a ratio of 1:3 was carried out for 7 min. Then, a mixture of rice husk, aluminum oxide, iron oxide was added after being stirred for 5 min. Mixing was done again for 10 min. After all the ingredients were mixed, they were poured into the mold and left to harden at room temperature for up to 10 h. Furthermore, the specimen was compressed with a pressure of 20 MPa for 15 min and a temperature of 80, 100 and 120 °C (Table 1). In this study, the specimen produced at a molding temperature of 80 °C is called specimen number 1 and coded SP-1. The specimen produced at a molding temperature of 100 °C is called specimen number 2 and coded SP-2. While SP-3 is specimen number 3 produced at a molding temperature of 120 °C. After that, the composite specimens were cut and characterized.

Table 1. Composite brake pads specimen code and compression molding setting parameters.

Specimen code	Pressure (MPa)	Holding time (min)	Temperature (°C)
SP-1	20	15	80
SP-2	20	15	100
SP-3	20	15	120

2.3. Testing and characterizations

In this study, the tests carried out were density, hardness, tensile and wear using the Ogoshi method, thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC). Density testing was based on ASTM D792 standard. Density testing was performed using an electronic density meter DME 220 series from Vibra Canada Inc. (Mississauga, ON, USA). This test is carried out by weighing the dry mass and the mass of the test object in water (wet mass). Vickers hardness testing in this study refers to the testing method carried out by [6,24]. Hardness testing was carried out using a Microhardness Tester F-800 machine (Future-Tech Corp., Kanagawa, Japan) with a test load of 25 gf and a dwell time of 10 s. Tensile testing was done according to the ASTM D638 standard using a HT-2402 Computer Servo Control Material Testing Machines from Hung Ta Instrument Co., Ltd., Samutprakarn, Thailand. The tensile test results consist of max force and elongation, which will be used to calculate the tensile strength and tensile modulus. Wear testing was performed using the Oghosi High Speed Universal Wear Testing Machine (Type OAT-U). The Ogoshi wear test was carried out with the width of the wear plate (B) = 3 mm, the radius of the wear plate (r) = 13.06 mm, the distance traveled during the wear process (10) = 66.6 m and the test load (F) = 2.12 kg. The TGA test was carried out based on the ASTM D6370 standard using the NEXTA STA test kit (Hitachi STA200RV with Real View Sample Observation). The temperature used in this test was 30–550 °C. The testing process was conducted at a heating rate of 10 °C/min with a 100 mL/min nitrogen gas flow.

The test results obtain the temperature and weight loss values. The DSC test was carried out based on the ASTM D3418 standard using the NEXTA STA test tool (Hitachi STA200RV with Real View Sample Observation). The temperature used in this test was 30–550 °C. The testing process was carried out at a heating rate of 10 °C/min with a nitrogen gas flow of 100 mL/min. The test results obtain the temperature and heat flow values of the composite specimens.

3. Results and discussion

3.1. Effect of compression molding temperature on the density of brake pad composite specimens

Figure 1 shows the density of composite brake pad specimens with variations in molding temperature. The results of this study indicated that the increase in temperature affected the results of the composite density. The lowest density was shown in the SP-1 specimen, which was 1.695 g/cm^3. While the highest density was shown in the SP-3 specimen, which was 1.701 g/cm^3. The increase in density value occurs due to an increase in the temperature of composite fabrications with the compression molding method which causes a decrease in the viscosity of the resin, making it easier for the polymer to fill voids [25,26]. The fewer voids produced, the greater the density of the brake pad samples [6]. Void content is the percentage by volume of empty spaces or cavities inside composite materials [26]. When there are voids in a composite, they occupy space that would otherwise be occupied by the composite material. As a consequence, the composite's overall density decreases as the total mass is distributed over a larger volume [6,26].

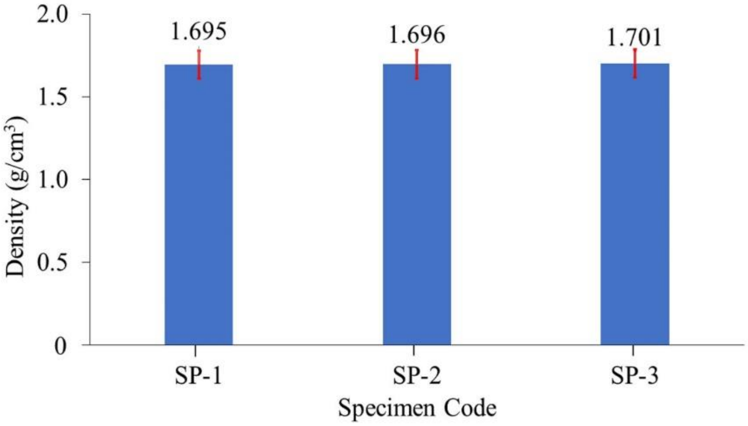

Figure 1. Effect of compression molding temperature on the density of brake pad composite specimens.

In the manufacture of brake pad specimens using the compression molding method, an increase in molding temperature causes the viscosity of the resin to decrease so that the resin flows more easily and wets the reinforcing material. This results in a better bond between the resin, filler and reinforcement, resulting in an increase in the density of the composite specimen [6]. This is what causes SP-3 to have a higher density than other composite specimens. Incomplete resin flow into the desiccated area of reinforcement and filler materials can give rise to porosity in composite specimens. This is typically the result of increased resin viscosity due to exposure to ambient conditions and low-temperature curing cycles, resulting in inadequate flow. The lowered molding temperature reduces the driving force of gas-induced cavity growth, so that more porosity is produced, resulting in a decrease in the specimen's mechanical properties [26–29].

The results of this investigation are consistent with Ochi et al.'s (2015) research. Their findings demonstrated a correlation between the density of long bamboo fiber/PLA Composites and the temperature of the mold. In general, the density of composites increases from 140 to 160 °C as the molding temperature. However, when the molding temperature exceeds 160 °C, the density of the composite decreases. This occurs because mold temperatures above 160 °C reduce the matrix's viscosity and make it simpler for air to become trapped inside the material during the molding process, resulting in the formation of numerous voids [30]. In another study, Ochi et al. (2022) demonstrated the relationship between the density of bamboo fiber bundle-reinforced bamboo powder composite materials and the molding temperature. As the molding temperature increased, the density of the composite specimens remained relatively constant. The density of composites was between 1.41 and 1.42 g/cm^3 [31].

Based on these research results, only the SP-3 specimen met the minimum density determined by PT INKA, which was 1.7–2.4 g/cm^3 [8]. Furthermore, the density of the SP-2 and SP-1 specimens almost met the specified density requirements. In addition, the density obtained in this study was higher than the results of previous studies. In previous research, brake pad composite fabrication used the hand layup method with various compositions. The highest densities produced in previous studies were 1.23 [6] and 1.33 g/cm^3 [7]; whereas in this study, the densities of the specimens SP-1, SP-2 and SP-3 were 1.695, 1.696 and 1.701 g/cm^3, respectively.

3.2. Effect of compression molding temperature on the hardness of brake pad composite specimens

The effect of temperature on the hardness of the composite is shown in Figure 2. The lowest hardness value was shown in the SP-1 specimen, which was 96.9 gf/mm^2 (HV), while the highest density was shown in the SP-3 specimen, which was 108.2 gf/mm^2 (HV). SP-3 and SP-1 specimens were the specimens with the highest and lowest densities produced in the study, respectively. In general, the hardness of composite specimens increases in proportion to their density. This is due to the fact that dense materials have strong interfacial bonding's between their matrix and reinforcement, making them more resistant to indentation and plastic deformation [6,32].

Previous research reached the same conclusion, which is that the specimen's higher density increased its hardness [33–36]. Research conducted by Fouly et al. (2021) showed that increasing the density of the composite causes an increase in the hardness of the specimen. Therefore, the average hardness also increases with increasing density. The poly(methyl methacrylate) (PMMA) nanocomposites specimen reinforced with 8 wt.% hydroxyapatite (PMHA8) specimen obtained a maximum hardness of 87.7 D-index. This happened because the PMHA8 specimen had the highest density compared to the other specimens [32]. Furthermore, an increase in the composite is hardness indicates a good interfacial bond between the matrix and the reinforcing fiber. The stronger the interfacial bond between the matrix and the reinforcing fiber, the higher the hardness of the resulting composite specimen [37,38]. Yawas et al. obtained the maximum hardness and density of non-asbestos brake pad samples using a compaction load and sintering temperature of 15 t and 150 °C, respectively. This happens because using these variations results in a more uniform distribution of frictional filler material particles in the matrix. Furthermore, this variation increases the surface area, which improves the matrix's bonding ability with frictional filler material [39].

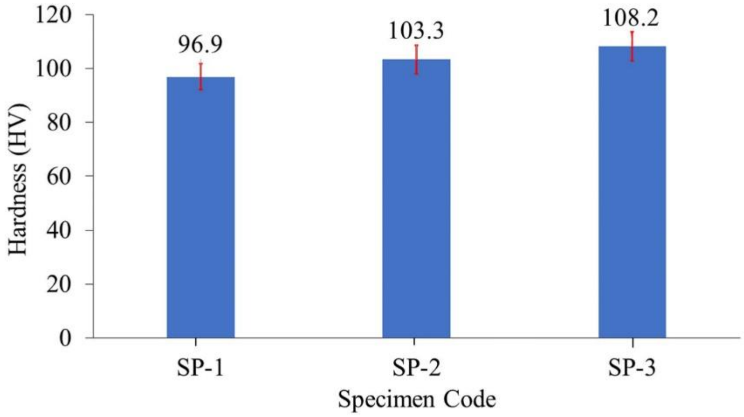

Figure 2. Effect of compression molding temperature on the hardness of brake pad composite specimens.

3.3. *Effect of compression molding temperature on the tensile strength of brake pad composite specimens*

The effect of temperature on the tensile strength of the composite is shown in Figure 3. The lowest tensile strength value was shown in the SP-1 specimen, which was 7.26 MPa, while the highest tensile strength was shown in the SP-3 specimen, which was 26.22 MPa.

The increase in the value of tensile strength is affected by the behavior of the bond between the resin and fiber interfaces at each increasing compression molding temperature variation [40]. This is in line with research conducted by Sumesh et al. (2020). Composites with an epoxy resin matrix formed by the compression molding method produce the best mechanical properties of tensile strength at higher temperatures [41].

Heating at high temperatures facilitates the mobilization of the resin in fiber impregnation. The increase in tensile strength is also due to an increase in mold temperature, which reduces the viscosity of the matrix which has an impact on reducing voids in the composite [42].

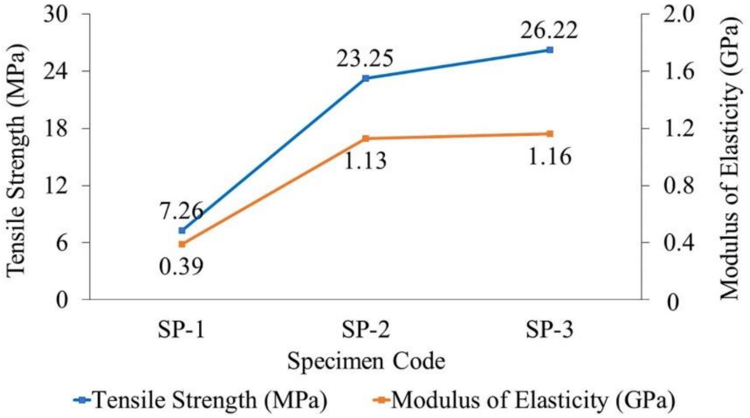

Figure 3. Effect of temperature on tensile strength and tensile modulus of brake pad composite specimens.

The increase in tensile strength may also be caused by the main process that occurs at a molding temperature of 80–120 °C for the evaporation. The higher the molding temperature, the higher the moisture content in the evaporating fiber, therefore the higher the tensile strength. Mvondo et al. (2017) stated that there was a negative relationship between the moisture content of tropical wood fiber and its tensile strength. Differences in the percentage of water content in fiber were studied. In fibers that have a lower water content produces high tensile strength, while high water content produces low tensile strength [43]. Figure 3 shows that the modulus of elasticity had increased with increasing molding temperature. The modulus of elasticity represents the inflexibility of a material [44,45]. The higher the value of the modulus of elasticity is, the less flexible the material. At higher molding temperatures, the specimen becomes stiffer so that the modulus value is higher [18,19]. In accordance with research conducted by Ochi et al. (2022) [31], the value of the elastic modulus increased with increasing temperature used in the specimen molding process using the compression molding method.

3.4. Effect of compression molding temperature on the specific wear of brake pad composite specimens

The wear test results showed that the use of higher mold temperatures can cause a decrease in the wear resistance of the composite (Figure 4). A higher specific wear value indicated a lower wear resistance property. In the study, the SP-3 specimen had the highest specific wear value of 15.08×10^{-7} mm^2/kg while the SP-2 specimen had the lowest specific wear value. Thus, it can be said that the highest wear resistance was found in the SP-2 specimen. According Günay et al. (2020), a negative correlation exists between a material's hardness value and the specific wear value it exhibits. Materials possessing high hardness exhibit enhanced resistance to wear and tear, hence endowing the test specimens with excellent wear resistance.

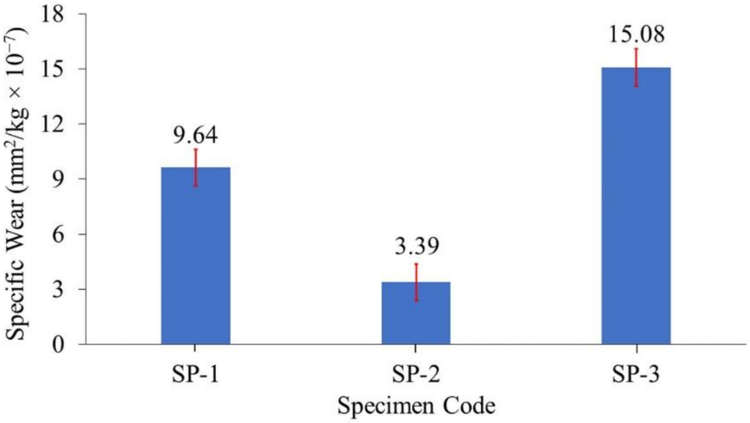

Figure 4. Effect of compression molding temperature on the specific wear of brake pad composite specimens.

The reason for this phenomenon is that the wear value acquired during Ogoshi wear testing exhibits an inverse relationship with the wear resistance qualities of the material being tested. A material's wear resistance qualities are improved when its specific wear value decreases [46].

Nevertheless, this investigation revealed that SP-3 had the highest specific wear value. It can be asserted that SP-3 exhibits the lowest level of wear resistance. This phenomenon can be attributed to the positive correlation between the temperature applied and the surface roughness observed in the composite specimen. The findings of a study conducted by Jan et al. (2020) indicate that elevating the temperature of the mold leads to a corresponding increase in the roughness of the composite material. Their findings indicated that elevating the mold temperature led to a corresponding increase in the surface roughness of the brake pad [47]. The occurrence of increased surface roughness in the specimen can be attributed to the roughness of the composite surface when it comes into contact with the surface of the hard steel disc (used as test equipment). This contact leads to the formation of cracks on both the surface and subsurface of the specimen, which results in matrix delamination or wear [48]. This results in the removal of material in large flakes and creates various irregular edge shapes resulting in higher friction and wear [48].

3.5. Effect of compression molding temperature on the thermal properties of brake pad composite specimens

TGA and DSC testing in this study was conducted to determine the thermal properties of the composite. Figure 5 shows that the composite brake pads specimen had several temperature variations during thermal decomposition that occurred in the temperature range of 30–550 °C. Weight loss on initial heating (30–200 °C) was caused by the evaporation of water on the rice husk. This happened because water was not chemically bound to the fiber. The reduction of fiber mass was further related to the degradation of hemicellulose. In the second range (200–400 °C), the weight loss that occurred was mostly due to the decomposition of hemicellulose and cellulose fibers and was followed by the decomposition of epoxy resin. In the last range (400–550 °C), the weight loss that occurred was caused by fiber degradation and decomposition of epoxy resin and filler.

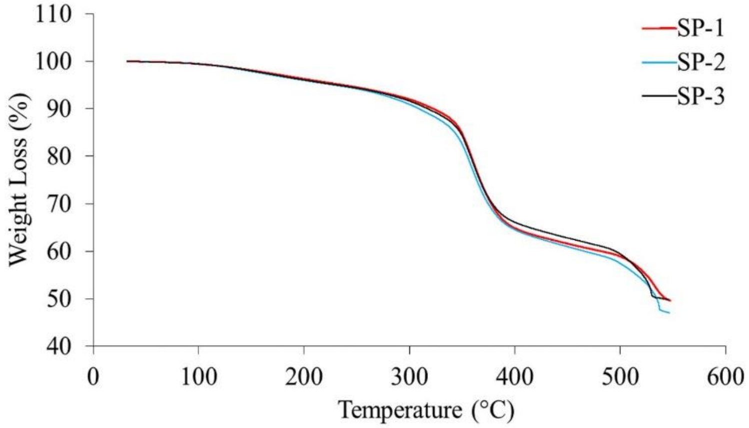

Figure 5. Effect of compression molding temperature on composite weight loss.

This is in accordance with research conducted by Chen et al. (2020), rice husk experiences weight loss which is divided into three stages. At a temperature of 35–150 °C, rice husk undergoes evaporation of water in the fiber. At temperatures of 150–380 °C decomposition of hemicellulose, cellulose and lignin occurs and at temperature stages of 380–600 °C fiber degradation occurs [49]. The TGA test with a temperature scale of 30–550 °C resulted in a residue of 49.48% in the SP-1 specimen, 46.97% in the SP-2 specimen and 49.94% in the SP-3 specimen. The residual results in this study are in line with research by Li et al. (2020), the residual weight of the composite showed a tendency to increase with increasing molding temperature. The results of the TGA curve analysis show that the right molding temperature will help the curing reaction and cross-linking of resin and fiber. The increased crosslink density of the composite will strengthen the thermal stability of the composite [50].

Figure 6 shows the derivative thermal gravimetry (DTG) curve for each composite brake pads specimen that experienced a maximum decomposition phase (Tmax) concerning temperature, which was shown at the main peak. SP-1 specimen occurred at 359.98 °C, SP-2 specimen at 356.13 °C and SP-3 specimen at 360.94 °C. Maximum decomposition is the maximum weight loss on the specimen that occurs at a certain temperature (Tmax), which can be used as the most important indicator in determining the thermal stability of a material [51]. The results showed that there was an effect of the use of specimen molding temperature. The specimen with a molding temperature of 120 °C had a larger Tmax and residue than other specimens. According to research by Li et al. (2020), by increasing the molding temperature, the durability of the epoxy resin and fiber activity tends to increase, which benefits the chemical bond between the fiber and the resin which results in increased composite cross-linking so that the degradation temperature will be higher [50].

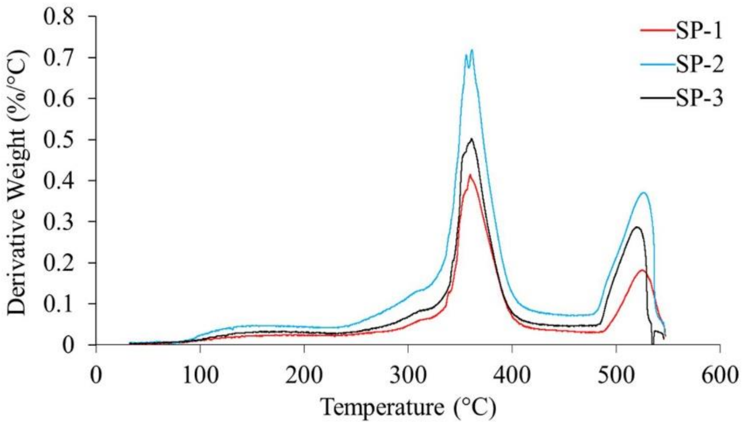

Figure 6. The effect of compression molding temperature on the derivative weight of the composite.

Based on Figure 7, the exothermic phase occurred when heated to a temperature of 30–550 °C. On the DSC curve, glass transition temperature (Tg) can be known from each specimen. The glass temperature is the transition temperature where the behavior of the composite transitions from hard glass to soft rubber [52]. The SP-1 specimen had a Tg of 311.25 °C. The SP-2 specimen had a Tg of 309.18 °C. The SP-3 specimen had a Tg of 310.06 °C. After passing through the Tg material, each specimen began to form crystals. Specimens will experience cold crystallization; cold crystallization is a unique phenomenon in which crystallization that accompanies an exothermic anomaly occurs when a material is heated to a temperature below its melting point but above the temperature of its glass. On the curve, the peak point of the curve after going through the Tg phase is crystallization temperature (Tc). Temperature crystallization is a transition temperature where the formation of a crystal structure occurs due to heating [53].

The SP-1 specimen has the highest Tc of 526.17 °C. The SP-2 specimen has the highest Tc of 529.83 °C. The SP-3 specimen has the highest Tc of 523.17 °C. The three specimens did not experience an endothermic phase, in which the specimens did not melt up to 550 °C. On the DSC curve, temperature melting (Tm) can be observed at the turning point of the curve. This is in line with Li et al. (2020), which stated that the first exothermic peak is closely related to the glass transition. Increased Tg and decreased peaks can be attributed to increased crosslinking and amorphism. The decreasing exothermic peak value of the composite indicates that the crosslink density of the mixture is increasing. It can be concluded that a reasonable increase in molding temperature will be conducive to increasing the crosslink density [50].

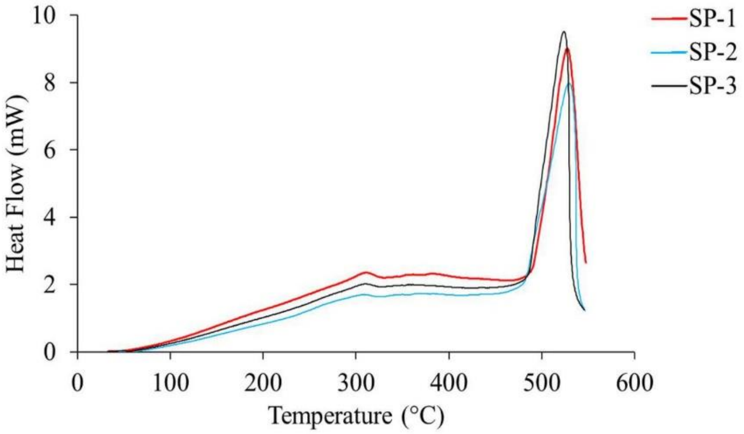

Figure 7. Effect of compression molding temperature on composite heat flow.

4. Conclusions

The effect of compression molding temperature on the characterization of asbestos-free composite friction materials for railway applications has been discussed in this study. Density, hardness, tensile, wear, TGA and DSC tests were performed to evaluate the properties of the obtained specimens. According to our findings, increasing the temperature during the compression molding process reduces the viscosity of the resin, allowing the resin to flow more easily and wet the reinforcement, filler and abrasive materials. This leads to better bonding between the constituent materials, so the density of the composite specimen increases. The mechanical and thermal properties of the composite specimens increase as density increases. The results of this study show that specimen SP-3 has better characteristics compared to other specimens. The density, Vickers hardness, tensile strength and tensile modulus of SP-3 specimens are 1.70 g/cm^3, 108.2 HV, 26.25 MPa and 1.16 GPa, respectively. The highest thermal properties were generated by SP-3 specimen, with total residues, Tmax, Tg and Tc values of 49.94%, 360.94, 310.06 and 517.17 °C, respectively. Furthermore, the molding temperature has a significant effect on the specific wear on the composite specimen. Specific wear ($\times 10^{-7}$ mm^2/kg) on SP-1, SP-2 and SP-3 specimens were 9.64, 3.49 and 15.08, respectively.

Use of AI tools declaration

The authors declare that they have not used Artificial Intelligence (AI) tools in the creation of this article.

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Conflict of interest

The authors declare no conflicts of interest.

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