WEAR RESISTANT OF CACO₃/FLY-ASH CERAMIC MATRIX COMPOSITE

QOMARUL HADI*, DWIKI HARDIYANTO

Department of Mechanical Engineering, Faculty of Engineering, Universitas Sriwijaya, Sumatera Selatan, Indonesia

*Corresponding author: Qoma2007@unsri.ac.id

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ABSTRACT: This study aimed to investigate the potential of utilising fly ash, a harmful byproduct of coal waste, in the manufacturing of composite materials to reduce its environmental impact. Calcium carbonate (CaCO3) and fly ash were mixed with zinc stearate using the powder metallurgy method to create wear-resistant samples. The compaction load was adjusted to achieve optimal low-pressure compaction, with pressures of 22.75 MPa, 23.45 MPa, 24.14 MPa, and 24.83 MPa used for single punch pressing. After 10 minutes of holding time, sintering was conducted at 900 OC for 1 hour. The raw materials were tested using X-ray diffraction (XRD) and sieved based on the mesh size. The composite was characterised through various tests, including density testing, wear testing, and scanning electron microscopy (SEM) to observe the microstructure. The density value increased with the compaction load, with a maximum apparent density of 1.794 g/cm3 and the highest porosity percentage at a pressure of 22.75 Mpa of 31.155%. Furthermore, the specific wear value decreased as the compaction load increased, with the lowest specific abrasion value of 1.17 x 10-6 mm2/kg. Therefore, using fly ash to produce composite materials is a promising solution to reduce the negative impact of coal waste while producing wear-resistant materials.

KEY WORDS: Composite, Fly Ash, CaCO3, Wear, Compaction

1. INTRODUCTION

Power plants that burn coal to produce electricity produce fly ash as a waste. The main components of these particles are silica, alumina, and ferric oxide. Fly ash has been studied as a potential concrete additive for decades due to its pozzolanic properties. On the other hand, calcium carbonate (CaCO₃) is a naturally occurring mineral that can be found in the earth's crust. It has applications in construction, agriculture, and even medicine [1].

Fly ash has been a popular supplementary cementitious material (SCM) since the 1930s due to its affordable, eco-friendly, and widely available nature. However, its supply and quality may make it as power plants migrate from coal to renewable energy sources or natural gas [2]. Ammonia injection in flue gas can lower SOx and NOx emissions but increase ammonia concentrations in fly ash, leading to unpleasant odours during construction. Fly ash is a byproduct of coal combustion in thermal power plants and is one of the most abundant industrial wastes generated worldwide. It is a fine powder consisting of spherical particles with a diameter ranging from 0.5 to 100 μ m [1]. Fly ash has been extensively studied as a filler in composites due to its low cost, high availability, and unique properties such as high surface area, pozzolanic activity, and high silica content [3].

A study by Jian et al. (2007) investigated the effect of fly ash content on the mechanical properties of polypropylene (PP) composites [4]. The results showed that the addition of fly

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ash improved the tensile strength and modulus of the composites. Calcium carbonate (CaCO₃) is another industrial waste extensively studied as a filler in polymer composites. It is a naturally occurring mineral that is abundant in nature and has unique properties such as high surface area, low cost, and high thermal stability[5].

The composite can be made through a variety of processes, including carbonation, sintering, and mechano-chemical synthesis. Calcite is formed when fly ash and calcium carbonate react with a carbon dioxide-rich environment [6]. Sintering involves heating a mixture of fly ash and calcium carbonate, which results in the formation of calcium silicates and calcium aluminates. During the mechano-chemical process that yields the composite, fly ash and calcium carbonate are milled together to form a powder.

In the building industry, the CaCO₃/Fly-Ash composite is used for precast concrete, concrete pavements, and concrete blocks, and, lastly, it is a workable alternative to traditional concrete. [7, 8]. It can also be implemented in soil stabilisation initiatives to enhance the soil's load-bearing capacity [9]. There are many advantages to using this composite, including its longevity, reusability, and low price. It is a high-value item that facilitates the reuse and recycling of waste products from manufacturing facilities.

Studies have been conducted to evaluate the mechanical properties and potential applications of CaCO₃/fly ash composites. For example, a study by Nuaklong et al. (2018) investigated the effect of adding CaCO₃ to fly ash-based geopolymer concrete and found that it improved the compressive strength and reduced the porosity of the resulting composite [10]. Another study by Ju et al. (2020) examined using CaCO₃/Fly-Ash composites as a potential construction material for railway sleeper applications. It exhibited superior strength and durability compared to traditional concrete mixes [11].

Sun et al. (2020) evaluated the flexural behaviour and fracture toughness of CaCO₃/ash composites with varying amounts of CaCO₃ and fly ash content [12]. The study found that increasing the CaCO₃ content resulted in a higher flexural modulus and fracture toughness while increasing the fly ash content improved the compressive strength. Furthermore, a report investigating the effect of incorporating nano silica particles into CaCO₃/Fly-Ash composites found that it further improved the mechanical properties of the composite, resulting in increased compressive and flexural strength [13].

While most of the research has looked into the impacts of CaCO₃ and fly ash on the hydration, compressive strength, and microstructure of concrete, only a few have looked into the specific wear performance of CaCO₃ and fly ash. Consequently, this study aimed to examine the synergistic mechanism for the influence of CaCO₃ and fly ash on strength development and wear properties.

2. MATERIALS AND METHOD

The materials used for this research include fly ash obtained from the residual combustion of power plants and CaCO₃ and zinc stearate obtained from e-commerce. This research was started by pre-treating the powders by heating the powders to remove the dirt at a temperature of 500 °C and a holding time of 30 minutes, then continued by sieving the coal fly ash powder and calcium carbonate with a sieve size of 200 mesh. The composition of the materials used is CaCO₃ 40%, as a matrix with fly ash 55% as a reinforcement and zinc stearate 5% as a binder. In the composite manufacturing process, the powders were mixed together using a Jar Test Flocculator with a rotation speed of 80 rpm and a holding time of 60 minutes. In the green body manufacturing process, compaction load was carried out by trial and error to get an excellent low-pressure compaction, after which the pressure variation used was 22.75 MPa, 23.45 MPa,

24.14 MPa and 24.83 MPa using single punch pressing with a holding time of about 10 minutes. The sintering process was carried out using a muffle furnace. The sintering process that will be carried out in this study uses a sintering temperature of 900 °C with a sintering holding time of 1 hour.

Material characterisation, including the mechanical strength, analyse the compounds and observe the microscopic structure of the CaCO₃/Fly-Ash ceramic composite. The researchers conducted several tests, namely wear testing (Ogoshi et al. type OAT-U) based on ASTM G99 standardisation, density testing using the Archimedes' method, XRD testing using the Rigaku Mini Flex Series 600 tool and SEM testing on the inspect S50 machine made by FEI.

3. RESULTS AND DISCUSSION

The XRD test results show that the graph has a rising peak line, meaning that the content is most dominant in the powder. After obtaining the phase graph formed, further analysis is carried out using Crystallography Open Database software to obtain the content of the peak graph formed. The XRD result of fly ash powder can be seen in Figure 1. Quantitative analysis of the XRD result of fly ash shows the presence of several mineral phases, with the following weight percentages: Quartz low (SiO₂) 28%, Hematite (Fe₂O₃) 22%, Aluminium oxide (Al₂O₃) 19%, Lime (CaO) 17%, Periclase (MgO) 14%.

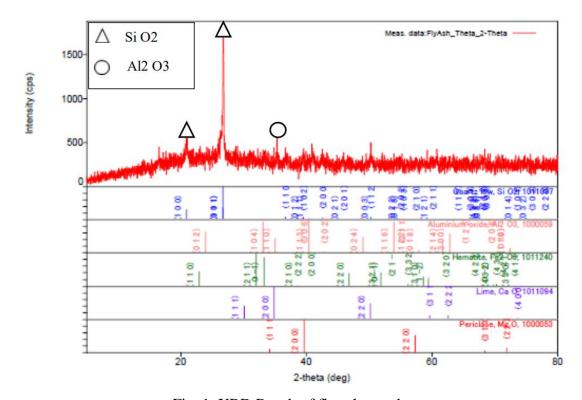


Fig. 1. XRD Result of fly ash powder

Fly ash is a byproduct of coal combustion, and its composition can vary depending on the type of coal and combustion conditions. The XRD result provides a quantitative analysis of the mineral phases present in the fly ash, indicating its chemical and physical properties. Based on the XRD result, the fly ash primarily comprises silica (SiO₂), present as quartz low; this is a



common component of fly ash, as it is a significant component of coal. Alumina (Al_2O_3) is also common in fly ash, as it is a significant component of clay minerals present in coal. The presence of hematite (Fe_2O_3) suggests that the fly ash may contain iron, which could have been present in the coal or introduced during combustion. Lime (CaO) and periclase (MgO) are likely to be present as impurities or additives, as they are not commonly found in coal.

The XRD result provides some information on the potential uses of the fly ash, as the high silica content suggests that it may be suitable for use as a pozzolan in concrete. Silica reacts with calcium hydroxide in the presence of water to form calcium silicate hydrate (C-S-H), a key component of concrete. The presence of alumina can also be beneficial, as it can improve the strength and durability of the material. The iron content may be a disadvantage, as it can cause discolouration of the concrete, but this can be mitigated by using appropriate admixtures.

Figure 2 shows the XRD result of as-received CaCO₃ powder, primarily composed of calcite (CaCO₃), making up 97% of the material. Anhydrite (CaSO₄) is also present, but only in trace amounts (2.9%).

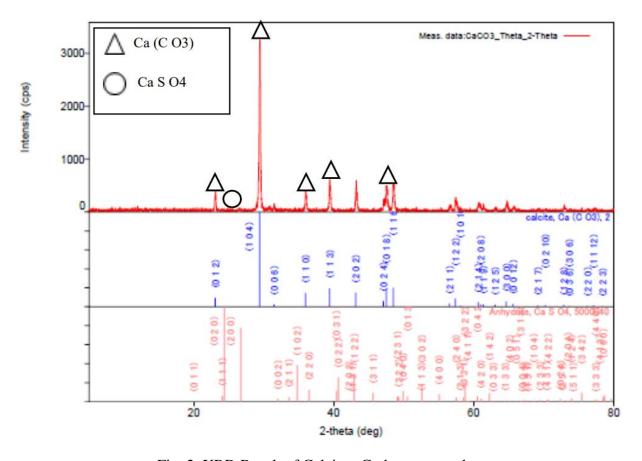


Fig. 2. XRD Result of Calcium Carbonate powder

Calcite is a common mineral found in many natural and synthetic materials. It is the main component of limestone, marble, and chalk. It is also used in a variety of industrial applications, such as in the production of cement, lime, and calcium carbonate products. The high percentage of calcite in the calcium carbonate powder suggests that it is a high-quality material that can be used for various purposes. Anhydrite is a mineral similar in composition to gypsum



(CaSO₄•2H₂O) but contains no water molecules. It is less common than gypsum but can be found in sedimentary rocks and evaporite deposits. The presence of anhydrite in the calcium carbonate powder is relatively low, and it is unlikely to impact the material's properties or performance significantly. The XRD results also confirm the crystal structure of the calcium carbonate powder. Calcite has a rhombohedral crystal structure [14], which gives it distinctive XRD peaks that can be used to identify the mineral phase. Anhydrite has a different crystal structure, so its XRD peaks will be distinct from calcite ones.

The results of the density testing of the green body calcium carbonate-fly ash composite indicate a direct relationship between the compaction load applied during the manufacturing process and the density of the composite. As shown in Figure 3, the highest density value of 2.265 gr/cm³ was obtained at the highest compaction load of 24.83 MPa, while the lowest density value of 1.987 gr/cm³ was obtained at the lowest compaction load of 22.75 MPa; this suggests that the higher the compaction load applied during the manufacturing process, the denser the composite will be. This finding is consistent with the conclusions drawn from the earlier analysis of the research results. It is important to note that the density testing was carried out on the green body composite before the sintering process, which means that the final density of the composite may be further increased after sintering.

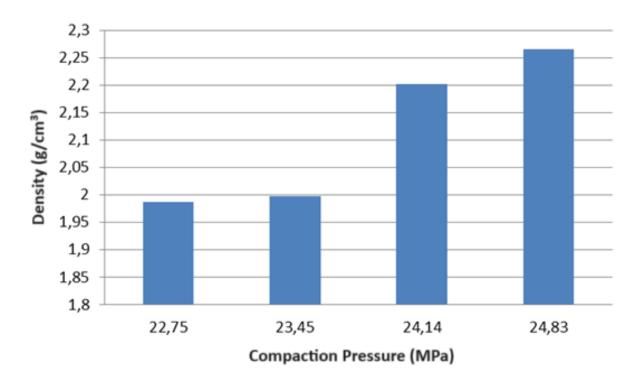


Fig. 3. Density of green body

The increase in density with increasing compaction load can be attributed to a higher compaction load leading to a higher packing density of the particles in the composite; this results in a denser and more compact composite with fewer voids and a higher overall density. Overall, the density testing results support the conclusion that the compaction load significantly impacts the physical properties of the fly ash-calcium-carbonate-composite and that optimising the compaction load during the manufacturing process can lead to a denser and more durable composite material.



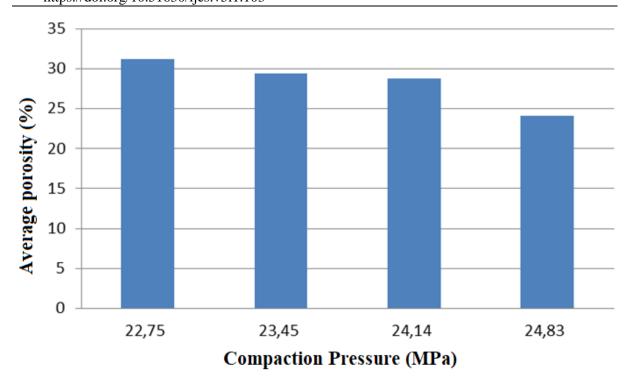


Fig. 4. Porosity of sintered CaCO3/Fly-Ash composite

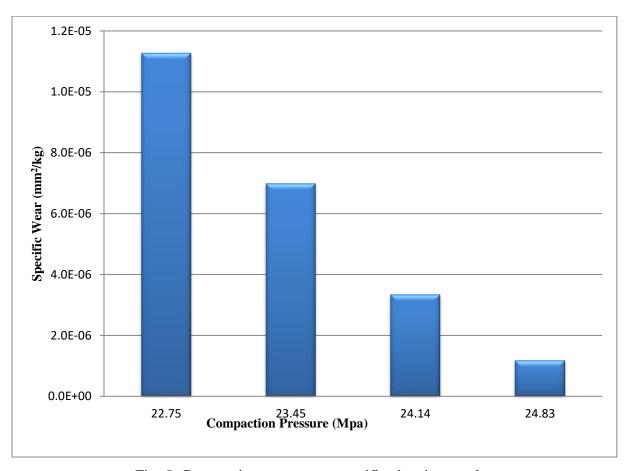
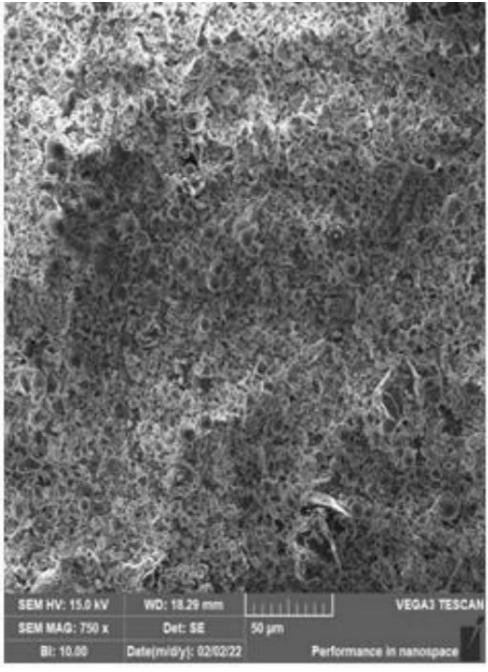


Fig. 5. Compaction pressure to specific abrasion results



The porosity graph of the CaCO₃/Fly-Ash composite in Figure 4 shows an inverse relationship between the compaction load and the porosity of the composite. Specifically, the higher the compaction load, the lower the porosity of the composite. The highest average porosity of 31.155% was found at the lowest compaction load of 22.75 MPa, while the lowest porosity of 24.085% was found at the highest compaction load of 24.83 MPa. These results support the earlier conclusion that increasing the compaction load during manufacturing leads to a denser and more compact composite with fewer voids. As the compaction load increases, the particles in the composite are more tightly packed, resulting in a smaller amount of empty space or porosity between the particles.





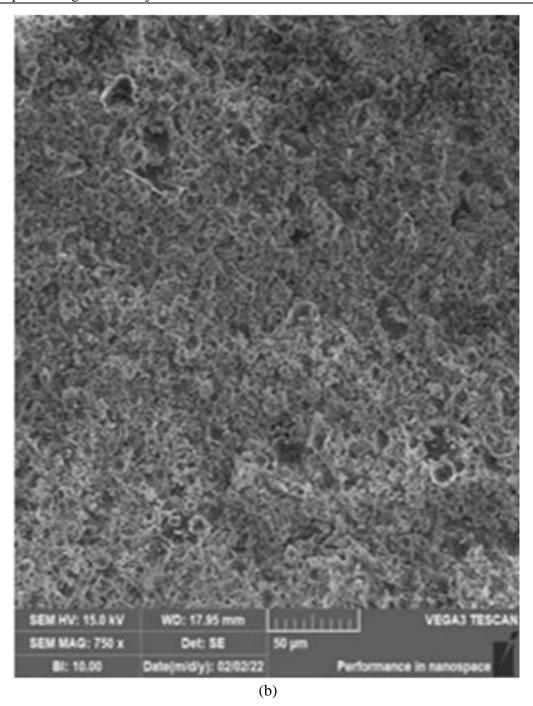


Fig. 5. SEM images (750x) samples with compaction load of a). 22.75 MPa and b). 24.83 MPa

The provision of higher compaction pressure results in a higher contact area between particles, leading to a more efficient packing of the particles and a lower overall porosity. The result also confirms the importance of optimising the compaction load during the manufacturing process of the CaCO₃/Fly-Ash composite to minimise porosity and maximise density. A lower porosity can increase mechanical strength, durability, and resistance to wear and tear, making the composite more suitable for various applications [15].

The wear test graph in Figure 4 shows that the specific wear value of the CaCO₃/Fly-Ash h composite decreases as the compaction load increases. The average specific wear value was found to be 1.12E10⁻⁵ mm²/kg at a compaction load of 22.75 MPa, while the most optimal specific wear value was 1.17E10⁻⁶ mm²/kg at a compaction load of 24.83 MPa.

This result indicates that increasing the compaction load during manufacturing can result in a composite with improved wear resistance. As the compaction load increases, the density between the matrix particles and the reinforcement also increases, resulting in a decrease in the number of cavities within the composite. This reduction in cavities, in turn, leads to a decrease in the number of scratches on the surface of the specimen, making the composite more resistant to wear and tear. The finding that increasing compaction pressure results in improved wear resistance is consistent with the results of previous studies [16]. These studies have shown that increasing compaction pressure improves mechanical properties, including wear resistance. The results of the wear testing indicate that the CaCO₃/Fly-Ash composite has potential as a material with good wear resistance, especially when prepared using higher compaction loads. These findings may be relevant to various applications, including manufacturing construction materials and automotive parts.

Figure 5 shows the SEM images of CaCO₃/Fly-Ash composite specimens with a compaction load of 22.75 MPa and 24.83 MPa at magnifications of 500x and 750x. The images reveal that the pore structures of the composite samples have irregular and random shapes, which is typical for composites prepared via powder metallurgy techniques. However, the images also show that the number of pores and the pore size decreases with increasing compaction load. This observation is consistent with the results of the porosity test, which also showed that the porosity of the composite decreases with increasing compaction load.

Additionally, the images reveal the presence of some powder clumps, likely due to incomplete mixing of the CaCO₃ and Fly-Ash powders before compaction. These powder clumps may cause a non-uniform distribution of the reinforcement in the matrix, which can affect the mechanical properties of the composite. The SEM images provide visual evidence for the effect of compaction load on the microstructure of the CaCO₃/Fly-Ash composite and highlight the importance of proper mixing of the powders before compaction.

4. CONCLUSION

The results of the research show that the preparation of CaCO₃/Fly-Ash composites was successfully carried out at various compaction loads. The study found that the higher the compaction load applied during the manufacturing process, the higher the composite density; this suggests that the compaction load significantly impacts the physical properties of the composite and that the higher the load, the denser the composite will be. The study also found that the porosity level of the composite was fairly high.

The research also found that the best compaction load variation from the manufacture of this composite is the 24.83 MPa compaction load; this suggests that the optimal compaction load for this type of composite ranges from 24.14 MPa to 24.83 MPa.

Finally, the study found that the specific abrasion of the composite decreased as the compaction load increased; this suggests that the denser composite is more resistant to abrasion, which could have implications for the practical applications of this material. Overall, the research provides valuable insights into the manufacturing process and physical properties

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of fly ash-calcium-carbonate-composites, which could be useful for future research and development.

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