

**PRODUCTION AND CHARACTERIZATION OF
COMPOSITE-BASED FRICTION MATERIALS
FOR SAFETY CLUTCHES IN AVIATION
APPLICATIONS**

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ABSTRACT

PRODUCTION AND CHARACTERIZATION OF COMPOSITE-BASED FRICTION MATERIALS FOR SAFETY CLUTCHES IN AVIATION APPLICATIONS

Clutch systems are one of the most important equipment in terms of controlling the speed of the moving vehicle. This study investigates aircraft clutch systems, focusing on the chemical and mechanical properties, production methods, and usage conditions of facing materials. Material characterization of a commercial composite-based aircraft brake pad (Trimat MN2221) was performed to understand pad compositions, using microstructural, phase (XRD), chemical (XRF), bond structure (FTIR), and thermal (TGA) analyses. New pad formulations were developed, and safety clutch pads were produced under constant conditions by varying raw materials using composite pad production methods, including raw material mixing, hot pressing, and baking. Experiments with different additives and mixture ratios were conducted, and the differences in pad properties were analyzed. This research aims to investigate the effects of varying the amounts of phenolic resin, quartz, alumina, glass fiber, and graphite in a predetermined clutch friction material composite matrix on the friction force and wear resistance of the samples. Three different clutch pad samples were produced with varying proportions of alumina and quartz, and their friction properties were tested. Similar experiments were conducted with different amounts of phenolic resin, glass fiber, and graphite. Physical properties were evaluated by measuring density, surface roughness, and hardness. Density was calculated based on mass-volume, and hardness was measured using a SHORE D device. Results showed alumina enhances friction materials and clutch performance, while quartz improves the friction coefficient. The study identified the most suitable formulation and optimal production parameters for clutch performance.

Keywords: Clutch, Composite, Friction, Microstructure, Material,

ÖZET

HAVACILIK UYGULAMALARINA YÖNELİK EMNİYET KAVRAMALARI İÇİN KOMPOZİT ESASLI SÜRTÜNME MALZEMELERİNİN ÜRETİMİ VE KARAKTERİZASYONU

Debriyaj sistemleri, araç hızını kontrol etmede kritik öneme sahiptir. Bu çalışmada, uçak debriyaj sistemleri, yüzey malzemelerinin kimyasal ve mekanik özellikleri, üretim yöntemleri ve kullanım koşulları incelenmiştir. Ticari kompozit esaslı bir uçak fren balatası (Trimat MN2221) malzeme karakterizasyonu, mikro yapısal (OM, SEM-EDS), faz (XRD), kimyasal (XRF), bağ yapısı (FTIR) ve termal (TGA) analizleri ile gerçekleştirilmiştir. Yeni balata formülasyonları geliştirilmiş ve kompozit balata üretim yöntemleri kullanılarak sabit koşullar altında güvenlik debriyaj balataları üretilmiştir. Farklı katkı maddeleri ve karışım oranları ile deneyler yapılarak balata özelliklerindeki farklılıklar analiz edilmiştir. Bu araştırma, fenolik reçine, kuvars, alümina, cam elyafı ve grafit miktarlarının sürtünme kuvveti ve aşınma direnci üzerindeki etkilerini incelemeyi amaçlamaktadır. Farklı oranlarda alümina (Al_2O_3) ve kuvars (SiO_2) içeren üç farklı debriyaj balatası örneği üretilmiş ve sürtünme özellikleri test edilmiştir. Benzer deneyler, fenolik reçine, cam elyafı ve grafit miktarları değiştirilerek tekrar edilmiştir. SAE J661 standardına göre sürtünme-aşınma testleri yapılmış, yoğunluk, yüzey pürüzlülüğü ve sertlik gibi fiziksel özellikler değerlendirilmiştir. Yoğunluk kütle-hacim ilişkisine göre hesaplanmış, sertlik SHORE D cihazı ile ölçülmüştür. TSE 555 standardına göre spesifik aşınma oranları belirlenmiştir. Sonuçlar, alüminanın sürtünme malzemelerini ve debriyaj performansını artırdığını, kuvarsın ise sürtünme katsayısını iyileştirdiğini göstermiştir. Çalışma, debriyaj performansı için en uygun formülasyon ve optimum üretim parametrelerini belirlemiştir.

Anahtar Kelimeler: Debriyaj, Kompozit, Sürtünme, Mikroyapı, Malzeme,

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SYMBOL LIST

A	Contact Area of the Sample
D	Ball diameter
D_n	Ball-on Disc Ball track diameter
F	Load
F_k	Kinetic Friction Force
F_s	Static Friction Force
L	Distance
m	Mass
m_k	Kinetic Friction Coefficient
m_s	Static Friction Coefficient
N	Normal Force
P	Applied Pressure Force
T	Time
V	Volume
W	Weight
ρ	Density

LIST OF ABBREVIATIONS

ABOSM	Asbestos Based Organic Friction Materials
NAO	Asbestos-Free Organic Friction Materials
SMF	Synthetic Mineral Fiber
BaSO ₄	Barium Sulfate
CaCO ₃	Calcium Carbonate
SEM	Scanning Electron Microscope
EDS	Energy Dispersion Spectroscopy
XRD	X-Ray Diffraction
TGA	Thermogravimetric Analysis
DSC	Differential Scanning Calorimetry
UV-VIS	Ultraviolet and Visible Light
XPS	X-Ray Photoelectron Spectroscopy
FTIR	Fourier Transform Infrared Spectroscopy
NMR	Elemental Analysis and Nuclear Magnetic Resonance
OM	Optical Microscope
XRF	X-Ray Fluorescence Spectroscopy

CHAPTER 1

INTRODUCTION

In this study, the experimental analysis of the structural components of safety clutch friction materials for aviation applications has been conducted. Firstly, material characterization studies of commercial composite-based aircraft brake pad material were carried out to acquire information about the brake pad compositions. Commercial Trimat friction material is used in aircraft clutch systems in aviation, and detailed information about the material properties (microstructure-composition-properties) of the product is not available in the literature. Different analysis methods were employed to determine the structure, morphology, and chemical content of the components constituting this friction material. Then, microstructural optical microscope (OM) and scanning electron microscope (SEM-EDS) examination, phase analysis (XRD), chemical analysis (XRF), analysis of chemical bond structures (FTIR) and thermal analysis of the composites to examine the properties of these materials (TGA) studies have been carried out.

The study investigates the effect of different additives and fiber types (such as glass fiber, metal-based fibers) and their singular and hybrid usage on the properties of the friction material produced, employing an experimental design approach. While the impact of fiber usage on the performance of organic-based composites in friction materials has been researched in the literature, the statistical interpretation of the effects of experimental design approach and experimental parameters on the properties of friction materials has not been previously explored in scientific studies.

In the light of the studies carried out, new formulations were created and in the continuation of the research, safety clutch facings to be used in aviation applications were produced under constant production conditions by adding different amounts of structural materials with the powder metallurgy method, which includes powder mixing, pressing and baking processes respectively.

In this study, the effects of unique compositions and compositions prepared in single or hybrid form using different fibers on the physical and mechanical properties of friction material samples were examined. In order to determine the physical and mechanical properties of the produced facing materials, friction wear tests were carried out according to the SAE J661 test standard. Friction and wear characteristics of the samples were determined using a ball-on-disk type wear tester. Comparison of physical properties was made by measuring the density, surface roughness and hardness values of pad samples containing different structural ratios. Thus, the effect of optimum usage rates on material properties was interpreted statistically.

CHAPTER 2

CLUTCH AND BRAKE PADS INSPECTION

2.1. Brake Pad

The brake allows a moving vehicle or machine part to stop by converting its kinetic energy into heat. At this time, the brake absorbs heat and gradually radiates into the atmosphere. A brake usually consists of two parts, a rotor and a stator on which friction material is located. The friction material is considered the replaceable part of the brake that wears out into gases and wear debris over the long period of use of the brake. ¹

2.2. Clutch Facing

The function of the clutch is to transfer the kinetic energy of a rotating crankshaft from a power source to the transmission and wheels. During engagement, slippage occurs, resulting in heat generation. This generated heat is absorbed by the clutch and distributed to the atmosphere. Thus, a clutch is a static friction pair that temporarily slips during gear shifting or other engagements. The clutch friction material facing is considered replaceable.

Brakes and clutches can operate in both dry and wet conditions. In dry friction pairs, the generated heat is generally removed by being transmitted to the surrounding air and structural elements. On the other hand, wet friction pairs, generally operate in an oil that absorbs heat and keeps the pair at low temperatures (usually below 200°C). It can also retain liquid wear debris. ²

Friction materials are used in a variety of ways to control the acceleration and deceleration of vehicles and machines. These materials can be found everywhere, from a small work machine clutch to jumbo airplane brakes.

For example, bicycle brakes can range from systems that rub against iron to rubber-coated pads that rub against a steel or aluminum rim on hand-operated brakes. Passenger vehicles may have disc brakes or drum brakes, or a combination of front disc brakes and rear drum brakes.³ Friction materials can be resin or rubber bonded composites based on asbestos, metallic fibers, or a combination of other fibers.

Trucks and off-road vehicles usually have large drum brakes, while some are equipped with front disc brakes only. These types of brakes generally operate at higher friction levels and temperatures compared to passenger vehicles. Large aircraft are equipped with disc brakes made of sintered friction material. New generation aircraft brakes are made of carbon composites and serve as rotors and stators.²

2.3. Types of Friction Materials

Before the mid-1970s, organic materials were the most common friction material for brakes and clutches in factory installations and the aftermarket. These materials generally contained 30-40% organic components and were asbestos-based.⁴ However, after the mid-1970s, vehicle downsizing and the introduction of front-wheel drive vehicles in North America led to the proliferation of a new class of friction materials, also known as semi-metals and semi-metals.⁵ Considering the health risks associated with asbestos fibers, a second class of friction material called asbestos-free organic materials emerged.⁶

2.3.1. Asbestos Based Organic Materials

Asbestos-based organic friction materials (ABOSM) are materials used to increase friction and wear resistance in industrial applications. However, due to the health risks of asbestos, the use of asbestos-containing materials has gradually decreased and is banned in many countries. Asbestos is a long-fiber mineral and has naturally durable and fire-resistant properties. Therefore, they have been used as friction materials in the past, especially in the automotive industry and industrial equipment. However, inhaling asbestos has been proven to be a carcinogen that can lead to serious health problems, for this reason, many countries have restricted or completely banned the use of asbestos. It has been replaced with asbestos-free alternatives.

Synthetic fibers, organic resins, metal powders and other materials have replaced asbestos and provide similar friction and wear resistance. These new materials are safer and environmentally friendly options. ⁷⁻¹⁰

2.3.2. Asbestos-Free Organics Materials

Due to the undesirability of using asbestos, organic friction materials (NAOs) that do not contain asbestos have been developed. NAOs (Non-Asbestos Organic) are available in a variety of formulations and other supplements are often used to replace asbestos. These reinforcements can be other fibers or materials with different properties. Generally, large amounts of asbestos-free fibers are used to replace asbestos. This is not only due to processing differences, but also to the fact that no single alternative fiber can fully replace asbestos. ⁶ Fiber combinations can be glassy: Glass or synthetic mineral fibers (SMF) blown from glass or slag; ceramic, metallic: steel, copper, or brass; wollastonite; or organic: cotton, acrylic, polynuclear, or cellulose-based. ¹¹

2.3.3. Semi-Metallic Materials

Carbon-metallic materials, also called semi-metallic, were introduced in the late 1960s but became more widely used in the mid-1970s, eventually becoming preferred in more than 90% of the front axles of passenger cars and light trucks in the United States by the 1980s. These materials generally contain more than 50% iron and/or steel components by weight. Originally, the main component of almost all semi-metals was a small amount of steel fibers together with iron powder. ¹² Later, large quantities of steel fibers began to be used along with small amounts of iron powder. Various property modifiers such as ceramic powders, organic or rubber particles, and graphite powders are added to increase the performance of the material to desired levels. Additionally, the resin binder necessary to hold the materials together is added to the mixture. ¹² Semi-metallics offered more stable friction, better fade resistance and longer life, rotor compatibility and quieter operation compared to the asbestos-based Class B organic materials they originally replaced.

2.3.4. Carbon Composite Materials

Carbon (or graphite) is a material with low density and highly specific heat, thermally stable. The combination of these properties makes carbon an attractive choice as a friction material. Therefore, many companies produce carbon fiber-reinforced carbon matrix composites, which are used specifically for aircraft brakes and racing cars.¹³ Carbon composites generally consist of three different types of carbon: carbon fibers, carbon resulting from the controlled pyrolysis of a phenolic-based resin, and carbon obtained by chemical vapor deposition that fills the pores.¹³

2.4. Clutch Systems in Aircraft (Overload Clutch)

Clutch systems in aircraft are vital mechanical systems that constantly evolve with technological developments in the aviation industry. These systems play a fundamental role in ensuring the controllability and maneuverability of aircraft. Clutch systems convert the forces applied by the pilot to the control surfaces into the movement of the aircraft, allowing basic movements such as roll, pitch and yaw to be performed. These systems are also critical to ensuring the aircraft can land and take off safely.

Clutch systems used in aircraft generally operate hydraulically or electrically. Hydraulic clutch systems move control surfaces using fluid pressure and are generally preferred on large commercial aircraft. On the other hand, electrical clutch systems, move control surfaces via electric motors or actuators and are generally widely used in modern aircraft and light aircraft. Both systems are rigorously examined and tested during the design and manufacturing processes because they are critical to the controllability and safety of the aircraft.

CHAPTER 3

FRICION MATERIALS: THEIR PROPERTIES, MANUFACTURING PROCESSES AND TESTS

3.1. Friction Materials; Characteristics of Ideal Friction Materials

3.1.1. Tribological Properties of the Material

Various studies conducted worldwide show that approximately one-third of the energy produced is lost due to friction. In addition, it has been determined that wear on the working surfaces of machines causes great economic losses. For example, it is estimated that annual maintenance, repair and renovation works to compensate for various wear and tear for a large integrated steel plant require an additional expenditure of 10 to 15% of the establishment cost of the plant.¹⁴

Increasing speeds and loads make the reliability and life conditions of machine systems more stringent, which requires controlling and limiting friction. Friction, wear and lubrication play an important role, especially in factories using flexible and rigid automation, aircraft and rocket systems, nuclear reactors and other advanced technologies. In line with these needs, a new branch of science, "Tribology", has emerged, which includes friction, wear and lubrication. The main goal of this discipline is to increase the durability of materials by minimizing the negative effects related to friction, wear and lubrication.

The aviation industry has an important application area of tribology. Friction and wear reduction technology allows aircraft to travel longer distances and consume less fuel, making aviation environmentally friendly and economical. Tribology research focuses on the development of new materials and coatings. In addition, the correct design of tribological systems is also important because correctly designed systems can reduce energy consumption, extend their life and ensure environmental friendliness.

As a result, the field of tribology is constantly evolving and will play an even more important role in the future. Increased tribology research will lead to more widespread use of tribology in industry and many other fields.¹⁵

3.1.2. Frictional Force

Friction is a force that occurs at the points where two surfaces touch each other. This force prevents the surfaces from contacting and moving with each other. The friction force varies depending on many factors such as the material properties of the surfaces, their roughness, shape and environmental conditions. Although usually very small, the friction force is practically always present. Absolute dry friction theoretically occurs between surfaces that are free of any foreign matter and are rubbing in a complete vacuum. However, under atmospheric conditions, thin absorption layers and tribo-mechanical reaction layers form between the surfaces. Even in this case, dry friction is accepted in practice. When special lubricants are applied to surfaces, the surfaces are no longer considered dry, and friction occurs between lubricated surfaces. This lubricated friction state can occur in boundary or hydrodynamic conditions.

Wear occurs as a result of the relative motion occurring between two objects in contact with each other and the surfaces of the objects affecting each other. ¹⁶

Total friction resistance is most generally affected by the following factors ¹⁵:

- Presence of wear products or particles coming from outside the system on the sliding surface,
- Relative hardness of the materials in contact with each other,
- Applied external load and/or displacement,
- External conditions such as temperature or lubricant,
- Surface topography,
- Microstructure or morphology of materials,
- Visible contact area,
- Kinematics of the surfaces in contact (direction of movement, magnitude of speed, etc.).

The coefficient resulting from the friction force is divided into two: static friction coefficient (μ_s) and kinetic friction coefficient (μ_k).

The load (F) is increased until slipping begins, and the applied load at the moment of slipping is determined as the friction force (F_s). Normal force is considered equal to the weight (W=N). In this case, the static friction coefficient (μ_s) is as follows;

$$\mu_s = \frac{F_s}{N} \quad (3.1)$$

For kinetic friction coefficient (μ_k) measurement, if sliding stops, more weight is applied for a new attempt until a constant sliding speed is achieved. In this case, the final load is the kinetic friction force (F_k).¹⁷ Kinetic friction coefficient (μ_k) is as follows;

$$\mu_k = \frac{F_k}{N} \quad (3.2)$$

3.1.3. Wear Resistance

Wear is the occurrence of permanent changes on the surfaces of materials. These changes can cause damage and wear to the surfaces and shorten their lifespan.¹⁵

In order for wear and tear on engineering materials to be considered wear, the following conditions must be present:

- Being a mechanical factor,
- Presence of friction (relative motion),
- It should be slow and continuous,
- Causes changes on the material surface,
- It occurs against our will.

Wear is the loss of material that breaks off unintentionally due to mechanical factors on surfaces that are in friction. In this way, the initial shapes of the surfaces are distorted, the gaps between the parts become larger and the intended function cannot be fulfilled normally.¹⁶

The biggest reason for machine malfunctions and structural elements becoming dysfunctional is wear. While friction creates energy loss, wear is an irreversible loss of material and requires additional energy to supply new material. It is difficult to establish a direct relationship between friction and wear because the frictional resistance between different pairs of materials may be the same, but the amount of wear can be very different.

The wear problem is often more complex than friction problems. Various types of wear patterns occur, and different wear patterns can coexist under the same conditions. ¹⁵

3.1.4. Lubricants

Lubrication is a method used to reduce friction and prevent wear. In this method, a liquid called a lubricant moves between the surfaces. Lubrication is effective depending on the material properties, roughness, geometry and environmental conditions of the surfaces. Graphite is often preferred as a lubricant because it is a cheap and effective lubricant. ¹⁸ As shown in Fig.3.1., lubricants are available in solid, liquid, gas and paste forms.

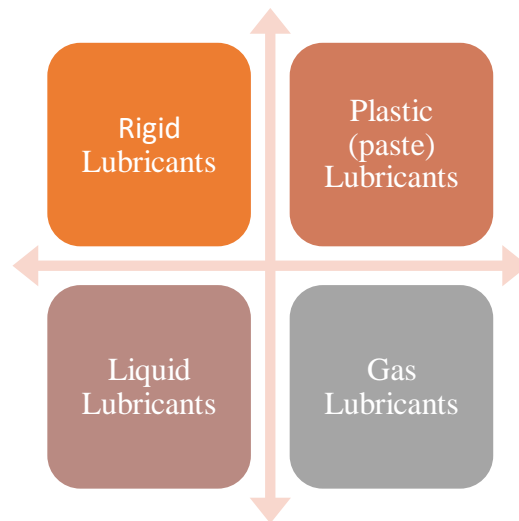


Figure 3.1. Lubricant Types

Advantages of lubricants,

- Reducing friction and wear on elements such as,
- Entry of foreign matter onto functional surfaces can also be prevented with adequate lubrication,
- Lubricants function as coolants in many cases and remove the generated heat from the bearing area,
- It is also a protector against corrosion and rust,
- It is also used to transfer mechanical energy in hydrostatic power transfer,
- In some cases, it can also be used as an insulator.

3.1.5. Characteristics of Ideal Friction Materials

Friction materials are used as brake pads and clutch discs in automotive, railway and aviation applications, which generally consists of metallic, ceramic, organic and composite-based materials. Friction materials must possess the ability to quickly and safely stop a moving vehicle under various conditions. During this process, kinetic energy is converted into thermal energy by the friction systems, thus material properties are very important.¹⁹⁻²⁰ Such as,

- Stable friction level (over a range of operating speeds, pressures, and temperatures),
- High resistance (to wear, cracking, thermal fatigue, fade),
- Minimum sensitivity (to water, moisture, oils, corrosive, salty, and muddy environments),
- Thermal stability (ability to withstand frictional heat),
- Should operate smoothly (without noise, vibrations),
- Easy in manufacturing.

Figure 3.2. shows a braking system and friction materials. As shown in this picture, the braking function is achieved by compressing the friction materials onto the disc with the help of a piston.

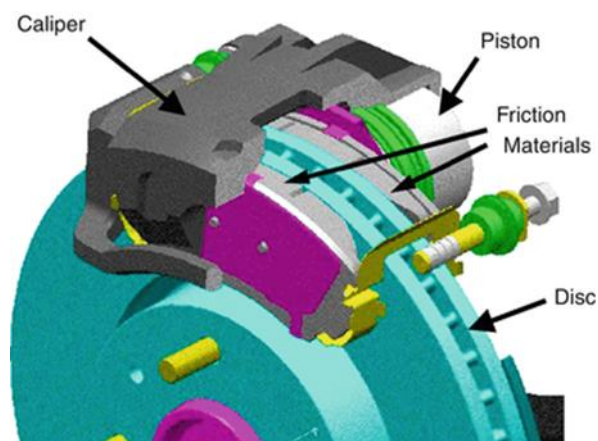


Figure 3.2. Friction Materials²¹

3.2. Components and Properties of Friction Materials

3.2.1. Materials Used in Composite Clutch Facing

Composite clutch pads are an important component often used in automotive and industrial applications. The materials used in the production of these pads usually consist of a combination of different components bonded together (Fig.3.3). Typically, these components include a matrix material with a high coefficient of friction, usually a resin-based matrix. This matrix material is usually reinforced with a fiber reinforcement, these fibers usually consist of carbon fiber, glass fiber or sometimes metallic fibers. These reinforcing fibers increase the durability, hardness and heat resistance of the pad. Additionally, fillers and additives can also be used in composite clutch pads, helping to optimize friction performance and increase wear resistance.

This complex combination of materials is carefully selected and optimized to provide the desired friction properties, durability and performance, often through a variety of tests.

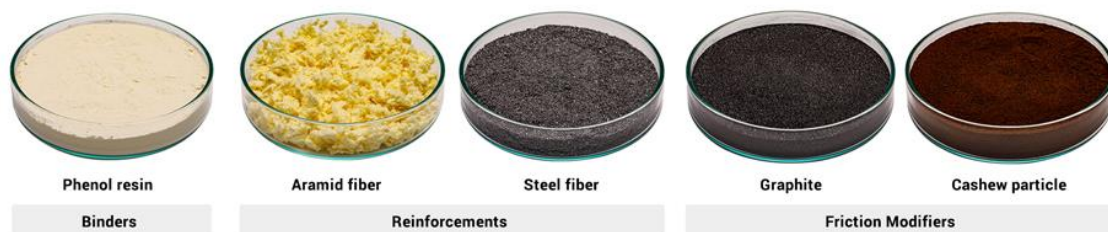


Figure 3.3. Materials Used in Composite Clutch Facing ²²

3.2.1.1. Binders

Fasteners are materials that hold together and strengthen the structure of composite clutch pads. Generally, polymers such as organic resins, phenolic resins, epoxy resins or polyester resins are used. The selected binder must have excellent heat resistance. ²³ It also increases the durability of the facing by providing mechanical strength and wear resistance.

While low resin concentration may lead to poor physical properties of composite clutch pads, high resin concentration generally results in low high temperature resistance. Therefore, when the physical properties of the pad such as porosity and hardness need to be changed, the resin ratio should be taken into account. This is an important factor for the pad to have the desired performance and durability. ²⁴

3.2.1.2. Reinforcements

Reinforcers are materials that increase the mechanical durability of the pad. The load applied during friction is carried by the reinforcement elements. ²⁵ Hardness, strength, thermal stability to the main body; They provide wear resistance and friction coefficient stability. ¹⁸ High-strength materials such as carbon fiber, glass fiber and aramid fibers are often used as reinforcement. These fibers increase the strength of the pad, providing higher performance and durability. Today, materials such as glass fibers, metal particles, aramid fibers and potassium titanate are preferred instead of asbestos. These materials have replaced asbestos by providing high friction properties, durability and safer use. ²⁵

3.2.1.2.1. Non-Metal Fibers

Non-metallic fibers are often used to strengthen and increase the resistance of composite clutch pads. Fiber materials, especially glass fiber and aramid fibers, provide high strength and durability. These fibers optimize the wear and temperature resistance by increasing the mechanical properties of the facing.

3.2.1.2.2. Metallic Fibers

Metal fibers are used in composite clutch pads to increase friction performance. Particularly thin fibers of metals such as copper and steel can be used as friction enhancers on the surface of the pad. These metal fibers help harden the surface of the pad and increase friction resistance. Generally, metals such as steel, copper and brass are used in composite clutch pads.

The main reason why these metals are preferred is that they provide effective absorption of the heat generated on the wear surface. This helps the pads last longer and maintain their performance. Additionally, by using oxidized or phosphorized fibers in some pads, fracture toughness and durability are increased. This increases the durability and longevity of the brake pad, ensuring safe use. ²⁶

3.2.1.3. Fillers

Fillers are used to improve or stabilize the properties of composite clutch pads. It is also used to increase manufacturability and reduce cost. ²³ Generally, materials such as mineral fillers, ceramic powders or glass spheres can be used. These fillers can increase the temperature resistance of the pad, improve wear resistance or provide sound and vibration absorption.

3.2.1.3.1. Organic Fillers

Organic filling materials are substances of natural or synthetic origin that are generally used in the production of composite materials. These materials are generally in fiber or powder form and are used to improve the mechanical properties of composite structures or to provide special functions. Organic fillers are generally used for various purposes such as increasing the strength of the composite material, increasing wear resistance, improving sound absorption or controlling thermal conductivity. Organic filling materials are widely preferred to adjust the properties of composite materials as desired. Other materials with similar properties are used as sound attenuators, especially due to their superior viscoelastic behavior. ²⁷

3.2.1.3.2. Inorganic Fillers

Inorganic filling materials are substances generally of mineral or metallic origin and used to improve the properties of composite materials. These materials can generally be in the form of solid particles, fibers or powders.

There are many different types of inorganic filler materials, but common ones include mica, talc, silica, calcite, barite, titanium dioxide, and alumina.

These materials are used to improve the mechanical properties, thermal conductivity, wear resistance, non-flammability or chemical resistance of composite materials. For example, silica powder can increase the hardness of composites, while mica plates can increase thermal resistance and also provide electrical insulation. Therefore, inorganic fillers play an important role in the formulation of composite materials to meet various industrial and application requirements. Typical inorganic fillers often used include barium sulfate (BaSO₄), mica, vermiculite and calcium carbonate (CaCO₃). These materials are preferred in many industrial applications because they have high melting temperatures.²⁸

3.2.1.4. Friction Modifiers

Friction modifiers are additives used to optimize the friction performance of the pad and consist of a mixture of abrasives and lubricants. Lubricant is generally used to reduce the wear of the friction material, while abrasive is used to improve the coefficient of friction.²³

Substances such as graphitized materials, metal powders or mineral additives can increase the coefficient of friction or reduce undesirable friction effects. These modifiers are used to adjust the friction characteristics of the pad to specific application requirements.

3.2.1.4.1. Lubricants

Lubricants are used to improve the friction performance of composite clutch pads. Generally, solid lubricants or oil-based fluids such as molybdenum disulfide or graphite can be used. Graphite is often preferred as a lubricant because it is a cheap and effective lubricant.¹⁸ By reducing the friction on the surface of the pad, these lubricants can reduce the amount of wear and heating, reduce the friction force and extend the life of the pad.²⁶

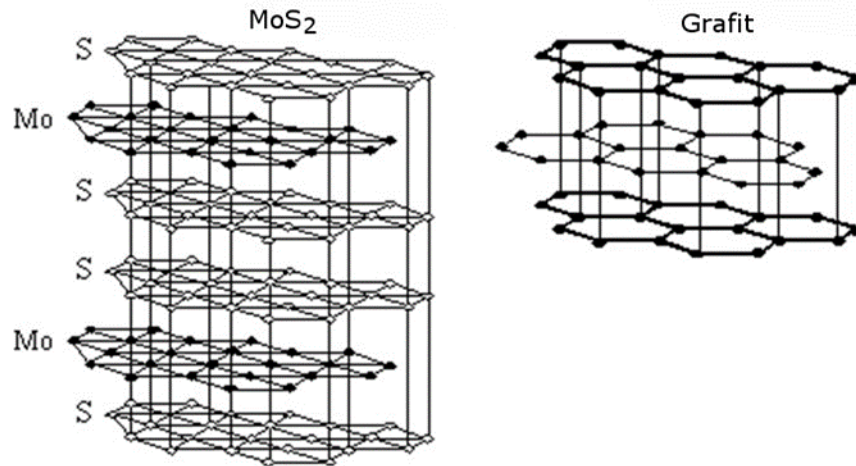


Figure 3.4. Structure of MoS₂ and Graphite²⁹

Thanks to the layered cage structure of the lubricants (Fig. 3.4.), their sliding properties are greatly improved. This shift is observed as individual solid lubricant particles coalesce to form a continuous, crystallographically oriented thin film that adheres to surfaces.²⁹

3.2.1.4.2. Abrasives

Abrasives used in composite clutch pads are used to increase friction performance and optimize surface roughness.

These materials are generally of natural or synthetic origin and may include minerals such as silica, sand, quartz, alumina, boron carbide, aluminum oxide and silicon carbide. Abrasives increase the contact surface between the pads and brake discs, providing more effective friction and providing better heat dissipation during braking. However, excessive use can lead to undesirable effects, so they require careful selection and use.³⁶

Different abrasives are used for various purposes in laboratory experiments. Properties of abrasives such as size, hardness and shape are important factors affecting wear rate. Research has shown that increasing temperature does not affect wear up to a certain point, but it does have an effect at higher temperatures. Studies have also found that materials with high melting temperatures generally have high wear resistance.³⁷

According to Rabinowicz, it has been stated that high humidity increases the amount of abrasive wear by approximately 15% and water vapor increases the abrasion effect similar to other lubricants.³⁸

3.3. Production Stages of Composite Clutch Facing

Composite materials are materials that are created by combining different components and generally have complementary properties. Composite clutch pads are one of these types of materials and are used in critical applications such as braking systems, especially in the automotive industry. The methods used in the production of these pads are of great importance in terms of both increasing durability and providing the desired performance properties.

Commonly used methods in the production of composite clutch pads include techniques such as hot pressing, mold injection and coil winding. The hot-pressing method allows the previously prepared composite material to be shaped by placing it in molds under a certain temperature and pressure. Mold injection is a method that allows molten material to be shaped by injecting it into a mold. Winding is a method that allows pre-prepared fibers to be combined with resin by wrapping them around a mold.

Each production method offers different advantages and disadvantages. For example, the hot-pressing method provides high strength and hardness, while the mold injection method may be more suitable for the production of more complex geometries. When choosing the method to be used, manufacturers must take into account factors such as material properties, production quantity, cost and quality. Therefore, the production methods of composite clutch pads must be carefully selected to ensure successful performance in industrial applications.

The stages of the hot-pressing method used in the production of pads generally consist of mixing the raw materials that make up the pad components, hot pressing and curing of the mixture, respectively (Fig.3.4.).

3.3.1. Mixing

At this stage, the components that make up the composite material are brought together. The components generally used in composite clutch pads are fibers (e.g. carbon fiber, glass fiber), fillers, friction modifier additives and phenolic resin. Composite pad material contains different components, and these components must be distributed homogeneously. With an effective mixing process, materials with different properties can be produced while preserving the desired properties of the facing material. However, the biggest challenge in composite production is distributing the reinforcement element homogeneously within the matrix and strengthening the bond between the reinforcement material and the matrix. The method and duration of mixing play an important role in determining the distribution of the reinforcement element in the matrix and the properties of the powders.³⁰

3.3.2. Pressing

The powder mixture, which obtains a homogeneous distribution after mixing, is concentrated by compressing it in the mold using a hydraulic or mechanical press. This process usually involves one-way compression. The molding process directly affects the properties of the friction material, so the pressure and temperature used must be adjusted to suit the material. The process generally takes place in the temperature range of 140-250 °C and forms the stage of giving density to the pad material. At this stage, lines close to the final dimensions are formed. Additionally, it increases the hardening of the resin contained and the durability of the material.³¹

3.3.3. Curing

After the pressing process, the composite material moves to the curing step. In this step, the pressed material is kept at a certain temperature and humidity for a certain period of time. During this process, the resin contained inside hardens completely and the material acquires the desired mechanical properties. The curing process is usually carried out according to a specific time-thermal program and ensures that the material is ready for end use.

The combination of these three steps constitutes the manufacturing process of composite clutch pads. Meticulous execution of each step ensures that the final product has the desired performance characteristics.



Figure 3.5. Production Flow Chart

3.4. Tests and Analyzes Applied to Friction Materials

Material characterization techniques are important research and analysis methods that help determine the physical, chemical and mechanical properties of the material. These techniques provide detailed information about the material's structural properties, components, surface structure, thermal behavior, mechanical strength and other properties. Material characterization plays a critical role in the design, production, processing and performance evaluation of the material.

One of the main techniques used in material characterization is the scanning electron microscope (SEM). SEM allows imaging and analyzing the surface structure of the material at high resolution. This technique is used to determine the morphology of the material, the amount of porosity, surface roughness and interactions between components.

It can also provide information about material composition through additional component analysis techniques such as energy dispersive spectroscopy (EDS). Besides this, X-ray diffraction (XRD) is another important technique used to study the crystal structure and phase composition of the material. XRD helps determine the crystallographic properties of the material and identify crystalline phases. This technique is widely used in solid-state physics research, material synthesis processes, and structural analysis of polycrystalline materials.

Other techniques used in material characterization include methods such as thermogravimetric analysis (TGA), mechanical tests, spectroscopic methods (FTIR) and analysis of magnetic properties. These techniques are important tools to understand and optimize the properties of the material from various aspects.³²

Physical properties include attributes such as the material's external appearance, shape, size, and structural properties. Chemical properties include properties of the material such as its chemical constituents, reactivity, chemical bonds, and chemical stability. Mechanical properties include properties of the material such as strength, flexibility, hardness, toughness and elasticity. These properties are critical in determining the material's performance in engineering applications.

3.4.1. Physical Tests

Density testing, one of the physical tests, measures the mass of a unit volume of a material and is usually expressed in grams/cm³ or kg/m³. This test is used to determine how light or dense the material is. Density may vary depending on factors such as the composition, porosity and structural properties of the material. Testing is usually performed by measuring the volume of the material sample and weighing its mass.

Another physical test, the surface roughness test, measures irregularities, protrusions and recesses on the surface of a material. This test is used to evaluate the surface quality of the material and ensure the level of smoothness required in certain applications. Surface roughness is often measured using devices such as profilometers.

3.4.2. Chemical Tests

Composition analysis is a set of techniques used to determine the chemical components and elemental content of a material. Fourier transform infrared spectroscopy (FTIR) method is one of the analyses.

3.4.3. Mechanical Tests

Hardness test, one of the mechanical tests, measures how much resistance the surface of a material offers under various loads. This test provides information about the material's hardness, toughness and wear resistance. Hardness may vary depending on factors such as the material's microstructure, composition and processing methods. Commonly used hardness tests include methods such as Brinell, Rockwell, Vickers and Shore D.

3.4.4. Performance Tests

Wear test, one of the performance tests, is used to determine the wear resistance and durability of a material. The material is subjected to friction or wear forces for a certain period of time, and the amount of wear and changes in the surface are measured during this time. This test is used to evaluate the material's lifespan, coefficient of friction and surface quality.

CHAPTER 4

EXPERIMENTAL STUDIES

4.1. Raw materials and Compositions for Friction Facing

During the experimental studies, a recipe list with different compositions was prepared depending on the pad compositions obtained from the material characterization of the brake pads produced by different companies. The recipe contents and percentage rates of the friction materials obtained as a result of the characterization are as seen in Table 4.1.

The research aimed to investigate the effects of varying the phenolic resin, quartz, alumina, glass fiber and graphite content of a clutch disc composite matrix determined by material characterization on the friction force and wear resistance. Based on the determined recipe list, 13 different recipe lists with ingredients in different chemical compositions were created. Three clutch disc samples were created using various combinations of abrasives and fillers, including alumina (Al_2O_3) and quartz (SiO_2), in amounts ranging from 0% to 8.5% by mass (2nd, 3rd and 4th recipes). The investigation focused on their friction properties. This process was similarly applied to phenolic resin (17% to 19% by mass) (5th, 6th and 7th recipes), glass fiber (3.5% to 5.5% by mass) (8th, 9th and 10th recipes), and graphite (8.5% to 10.5% by mass) (11th, 12th and 13th recipes), with different quantities being tested in each case.

Table 4.1. Raw Materials Used in Facing Sample Production

Friction Clutch Facing Materials		Raw Materials
Binders (% 10-20)		Phenolic Resin
		Rubber Dust
		NBR
Reinforcements (<% 25)	Non-Metallic Fibers	Glass Fiber
	Metallic Fibers	Steel Fiber
Fillers (<% 50)		Barite (BaSO ₄) or SrSO ₄
		Calcite (CaCO ₃)
		Zinc Oxide (ZnO)
		Quartz (SiO ₂)
		Iron Oxide (Fe ₂ O ₃)
		Talc:MagnesiumSilicateHydr.
		Expanded Vermiculite
Friction Modifiers (<% 10)	Lubricants (% 15)	Zinc Sulphide (ZnS)
		Graphite
	Abrasives (% 15)	Alumina (Al ₂ O ₃)

4.2. Process: Preparation of Mixtures for Friction Materials

In the continuation of the research, respectively; Experiments were carried out by adding different amounts of structural materials using the powder metallurgy method, which includes powder mixing, hot pressing and baking processes.

Sample preparation and testing steps of the composite friction materials performed Fig.4.1. It is schematized in 6.

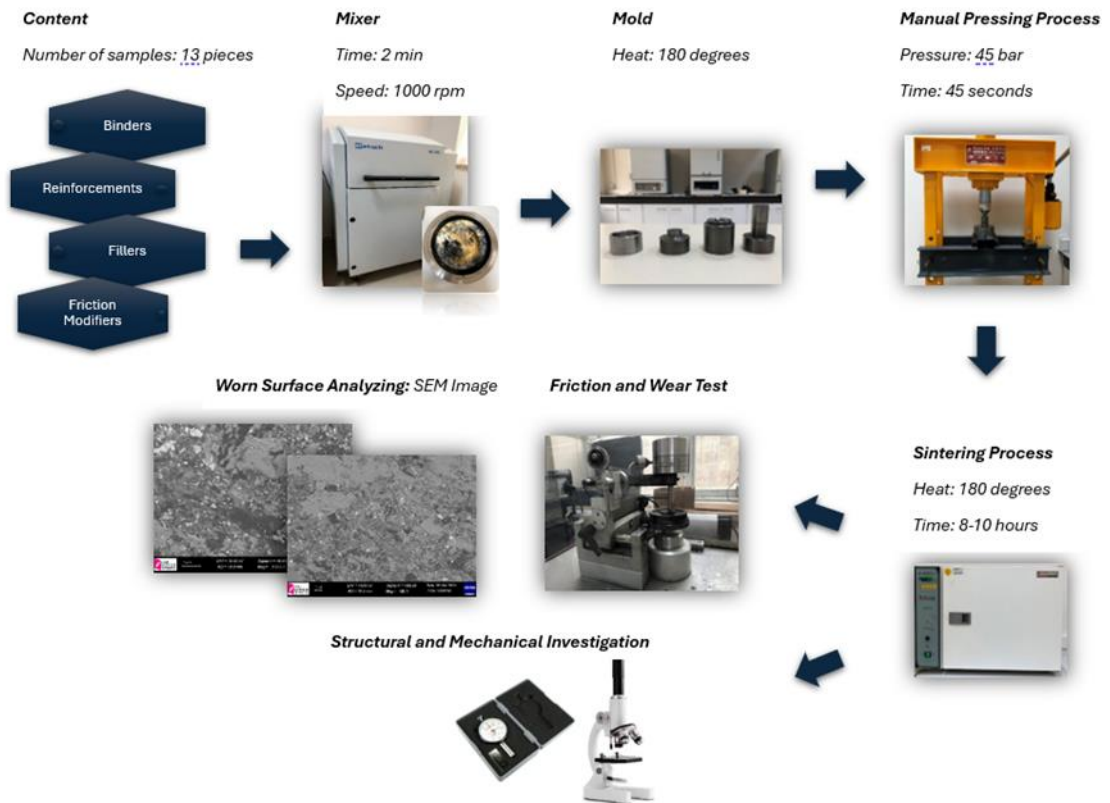


Figure 4.1. A Schematic Representation Showing the Sample Preparation and Testing Steps of Clutch Facing Composite Friction Materials

First, the raw materials in the recipe list are mixed in a Vibratory Disc Mill (Fig. 4.2.) at 700 rpm for 1 minute and the coarse grains are reduced.



Figure 4.2. Retsch Vibratory Disc Mill RS 200

Materials that provide sufficient homogeneity are taken from the mixer, 10 g of material is filled into a mold (Fig. 4.3.) with a diameter of approximately 25 mm and a thickness of 10 mm and subjected to the pressing process.



Figure 4.3. Steel Pellet Mold

Pressure holding and mold temperatures are as shown in the Table 4.2. From time to time, the chamber is lifted up to allow air release (Fig 4.4.).

Table 4.2. Data of Hydraulic Press Machine

The Operation Performed	Numerical Data
Mold temperature	160 °C
Time to wait above	1 sec
Waiting time at pressure	15 sec
Operating pressure	6-7 tones
Number of air intakes	6 pieces



Figure 4.4. Manual Press Machine

Finally, the test samples are subjected to the sintering process in a laboratory oven (Fig. 4.5.) at a temperature between 180 °C, on a fixed band for 8-10 hours to obtain their final shape.



Figure 4.5. M420 Electro-mag Branded Laboratory Oven

During the production of the brake pads used in the experiments, support was received from Eren Balata Sanayi company, Izmir Institute of Technology and Manisa Celal Bayar University. Clutch pads were produced under constant production conditions by adding different amounts of structural materials using the powder metallurgy method, which includes powder mixing, hot pressing and baking processes, respectively.

During the production of the pads used in the experiments, the raw materials in the recipe list were first mixed in a Retsch (RS200) branded ring mill at 700 rpm for 1 minute and coarse grains were reduced.

Materials that provide sufficient homogeneity were taken from the mixer, approximately 10 g of material was filled into a 10mm thick mold and completed samples were shown as in Fig. 4.6.

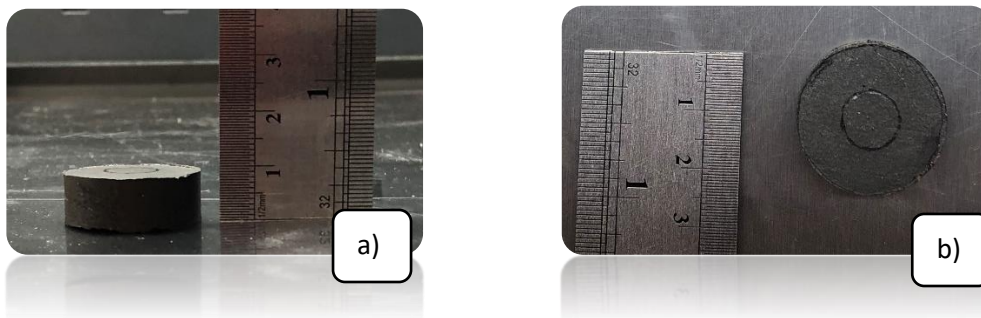


Figure 4.6. Sample and Physical Dimensions a) Depth of Sample b) Diameter of the Sample

The raw material analyzes performed are as follows; mineral phase analysis (X-Ray Diffraction (XRD) Analysis), chemical composition analysis (XRF), thermal analysis (Thermogravimetric Analysis-TGA-DTA), PSD, microstructure analyzes (SEM-EDS analysis-optical microscope), particle density.

4.3. Testing and Characterization of the Produced Samples

4.3.1. Wear and Friction Tests

A mechanism called ball-on disc was used in the wear and friction tests carried out to determine the tribological properties of the prepared brake pad materials.

During the experiment, first of all, care was taken to ensure that the disk part was in full contact with the sample. Different simulations were tried with 2000 m rotation by applying the desired amount of pressing force on the disc with a 10 N weight placed on the test device.

The ball-on-disc tester is a laboratory device used to evaluate the wear and friction properties of material surfaces. Basically, this device contains a disk and a ball. The disc is usually made of metal and has a smooth surface. The ball, on the other hand, is usually made of a hard material and creates friction and wear as it rotates on the disc. During the test, the disc is rotated, and the ball follows a specific path on the disc. The amount of friction and wear is measured by the amount of marks or wear particles formed on the disc surface. This testing machine is widely used to compare the friction and wear behavior of different materials, to guide material selection and to conduct research on tribology (friction, wear and lubrication).

In the test carried out according to the ASTM G99 standard, the amount of wear can be determined by measuring the initial dimensions of the sample when it is inserted into the device and the final dimensions after the wear test, and similarly, the amount of wear can be determined by weighing the first and last weight amounts and determining the weight loss.

In addition to the material, dimensions and surface quality of the test sample used during the test, other parameters that affect the test are the loading force used, the rotation speed of the disk, the distance of the contact point of the sample to the disk from the center, temperature and environmental conditions.

In this experiment, which was carried out to determine the wear and friction behavior of the test samples, a Ball-on disc device (Fig. 4.7.) and a 1050 steel disc were used as abrasive. The samples were weighed with a scale with a precision of 0.0001 g before the experiment.



Figure 4.7. Ball-on Disc Test Device Used in Experiments

During the experiment, a load of 10 N was applied to the samples connected to the device. The friction force generated by operating the device with a maximum speed of 25 cm/h was read on the screen (Fig. 4.8.). Based on the data recorded by the device, friction force and friction coefficient figures were created depending on the path taken.

During the experiment, the samples were measured at a distance of 2000 meters, and at the end of the wear, their weights were measured and the mass loss due to wear was calculated. The mass changes of the samples are given in the table below.

In addition, in the light of the data obtained, wear loss, wear rate and friction coefficient depending on the mass and volume of the materials were calculated and this information was supported with graphics.

During the experiment, basic parameters such as load, distance and speed were first determined. Considering that a plane landing on the runway travels an average of 1500-2000 meters, this distance was taken as the distance. The maximum speed of the device was 25 cm/h, and a weight of 10 g was used as the load. ³³

$$\text{Weight} \quad W=mg \quad [\text{N}] \quad (4.1)$$

$$\text{Area} \quad A= \pi r^2 \quad [\text{mm}^2] \quad (4.2)$$

$$\text{Pressure} \quad P=W/A \quad [\text{N}/\text{mm}^2] \quad (4.3)$$

$$\text{Frictional Force} \quad F_s=W \times F \quad (4.4)$$

With the help of formulas 4.1, 4.2 and 4.3, the applied pressure force was found depending on the load used and the contact area of the sample.

Time or distance can be calculated by drawing the desired unknown value with the formula 4.5. In the experiment, the change was examined over a total distance of 2000 meters.

$$L = \pi D n N T \quad (4.5)$$

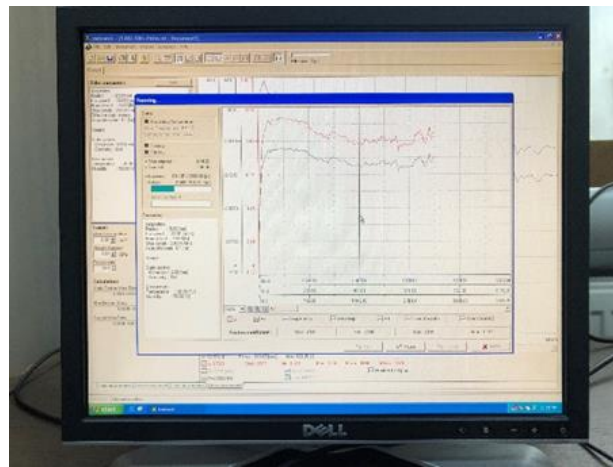


Figure 4.8. Ball-on Disc Tester Control Screen

During the calculation of the friction coefficient, the friction force read from the device was divided by the applied load and calculations were made with formula 4.4.

Following the basic values, the samples were connected to the device and subjected to abrasion at distances of 2000 meters, and mass and volume losses were determined with formulas 4.6 and 4.7.

$$\text{Mass Loss} \quad m_1 - m_2 \quad [\text{gr}] \quad (4.6)$$

$$\text{Volume Loss} \quad V = (m_1 - m_2) / \rho \quad [\text{gr/mm}^3] \quad (4.7)$$

4.3.2. Hardness Tests

SHORE D Calibration Set hardness measurement method was used in the experiments carried out to determine the hardness of the brake facing samples. Since it is a composite-based material, it has become difficult to make measurements with other measuring devices.

4.3.3. Density Calculation Experiments

Following the microstructure examination of the samples used in the experiments, the weights of the materials were first determined on a precision scale before the abrasion test. Then, the weights of the samples after wear were measured and the change in their weights due to wear was examined. The densities of the samples were weighed before and after wear and were calculated using the formula 4.8. ²⁵

m_1 : weight of the material before wear

m_2 : weight of the material after wear

ρ : density;

$$V = (m_2 - m_1) / \rho \quad (4.8)$$

Friction and wear characteristics of the samples were determined using a Ball-on-disk type wear tester. I will talk about the first four trials, because anomaly was observed in other prescription trials and the tests will be repeated.

4.3.4. Microstructural Investigations

Microstructural studies involve examining and characterizing a material or a system under a microscope.

These examinations are used to determine the material's internal structure, crystal structures, size and shape of grains, amount and distribution of porosity, chemical composition and other microscopic properties.

Common techniques used for microstructural investigations and crystal phase contents of materials include scanning electron microscopy (SEM) and X-ray diffraction (XRD), respectively.

These techniques help material scientists, engineers, and scientists understand and improve the properties of materials.³⁵

These are important for understanding their effects on the mechanical, thermal and optical properties of the material. Scanning Electron Microscope (SEM) device and its associated Energy Dispersive spectroscopy (EDS) elemental analyzer were used to determine the microstructures of the prepared samples. X-ray diffraction (XRD) analyzes were performed to detect the phases and compounds that may occur in the samples as a result of sintering of samples produced with different compositions.

CHAPTER 5

RESULTS AND DISCUSSION

5.1. Characterization of Commercial Friction Materials (Trimat MN2221)

In the thesis study, a commercial friction material whose shape is shown in Fig. 5.1. was examined. Material characterization studies of composite-based brake pad material were conducted to obtain information about commercial aircraft clutch pad compositions. These materials are from Trimat Ltd. that it is an aircraft brake pad material produced by the company. Microstructural examination of the composites was carried out using different analysis methods optical microscope (OM) and scanning electron microscope (SEM-EDS) in order to determine the structure, morphology and chemical content of the components that make up these friction materials (Trimat MN2221). In order to examine the properties of these friction materials and the components that make up the material, phase analysis (XRD), chemical analysis (XRF), analysis of chemical bond structures (FTIR) and thermal analysis (TGA) studies were carried out.

In this part of the research, composite-based brake pad samples will be produced with compositions prepared depending on the determined brake pad compositions, and these materials will be examined through performance tests.

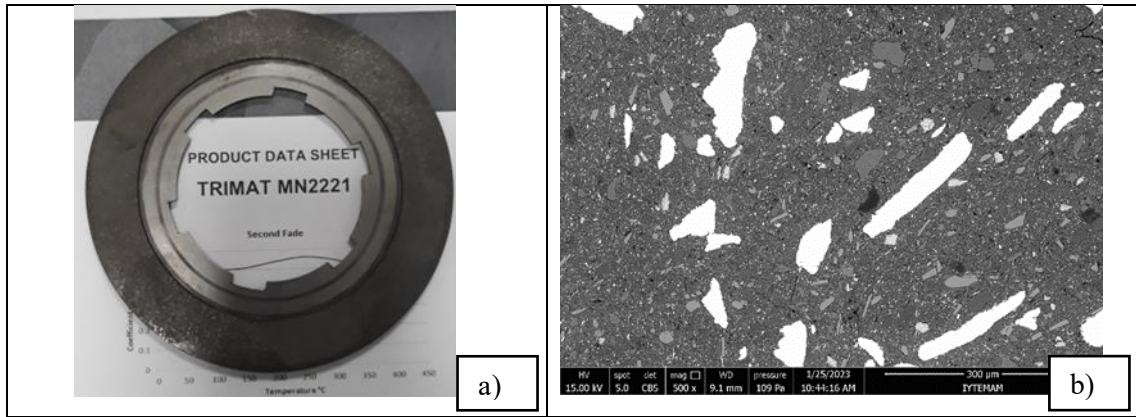


Figure 5.1. a) TRIMAT MN2221 Aircraft Clutch Facing Sample, b) SEM Image of the Commercial Sample

Trimat manufactures a variety of organic and semi-metallic press-molded friction products. The product range is asbestos-free and covers a wide range of performance properties, from low modulus products for tensioning devices to high-friction, heavy-duty materials used in aircraft braking systems. The friction coefficient and maximum continuous and intermittent operating temperature values of the Trimat MN2221 sample examined in the thesis study are 0.53, 250 °C and 350 °C, respectively, as shown in Fig. 5.2. In addition, when we look at the technical properties of the commercial friction material given in Fig. 5.2., it is stated that the wear rate is 63 mm³/MJ, the specific gravity is 2.5, and the tensile and compressive strengths are 23 and 88 N/mm², respectively.

Technical Details:		
Property	Typical Values	
Coefficient of Friction (SAE J661)	0.53	
Wear Rate (SAE J661)	63 mm ³ /MJ	(0.0103 in ³ /hp.hr)
Specific Gravity	2.50	
Ultimate Tensile Strength	23 N/mm ²	(3335 psi)
Ultimate Compressive Strength	88 N/mm ²	(12760 psi)
Recommended Operating Range:		
Maximum Intermittent Temperature	350 °C	(662°F)
Maximum Continuous Temperature	250 °C	(482°F)
Maximum Pressure*	3.0 N/mm ²	(290 psi)
Maximum Rubbing Speed	30 m/s	(5000 ft/min)
Recommended Mating Surfaces:		
Close grained cast iron (180 Brinnell or over); forged or cold rolled steel (200 Brinnell or over).		

Figure 5.2. Technical Detail Data of TRIMAT MN2221 Aircraft Brake Pad Sample ²¹

According to these technical characteristics, the Trimat MN2221 sample has a high friction level and is characterized by high stability, a very stable coefficient of friction at high temperatures and good wear resistance.

The raw materials to be used in composite brake pad material compositions suitable for aviation applications were determined, and powder mixtures were prepared by determining appropriate friction material recipes using experimental design methods (such as mixture design). The samples prepared in accordance with these prescriptions were subjected to the necessary tests and analyses. The findings obtained from the experimental parameters were evaluated statistically. As a result, the aim of this study is to develop a composite clutch facing composition that can be used in friction systems for the aviation industry, to produce clutch discs and to conduct material analysis.

In the studies, first of all, the microstructures of the TRIMAT MN2221 aircraft brake pad sample and the distribution of the pad components within the pad structure were examined under the image analysis microscope. For this purpose, during the sample preparation stage, the samples were first sectioned, bakelite, sanded with sandpaper and then polished. Two samples, one from the cross section and one from the surface, were cut with a precision cutting disc and then molded using bakelite as shown in Fig. 5.3., and the surface of the samples was sanded.

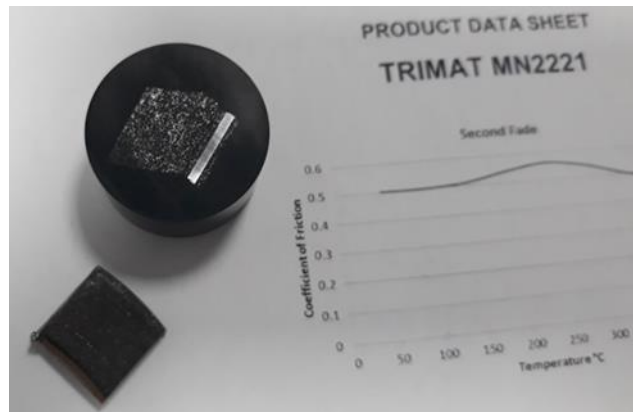


Figure 5.3. TRIMAT MN2221 Aircraft Clutch Facing
Sample a) Surface Section, b) Cross Section

In order to determine the structure, morphology and chemical content of the components that make up this friction material, microstructural examination of the composites was carried out using analysis methods such as OM, SEM-EDS.

Afterwards phase analysis (XRD) and thermal analysis (TGA) studies were carried out to examine the properties of these materials. Formulations were created in the light of the studies carried out.

5.1.1. Microstructure Analysis (Optical Microscope)

Figure 5.4. shows cross-sectional images of the polished section of the Trimat sample taken under an optical microscope at 50 magnifications in two different modes. Structures consisting of shiny metallic particles and shiny/dark inorganic particles with different particle sizes and morphologies embedded in the matrix attract attention.

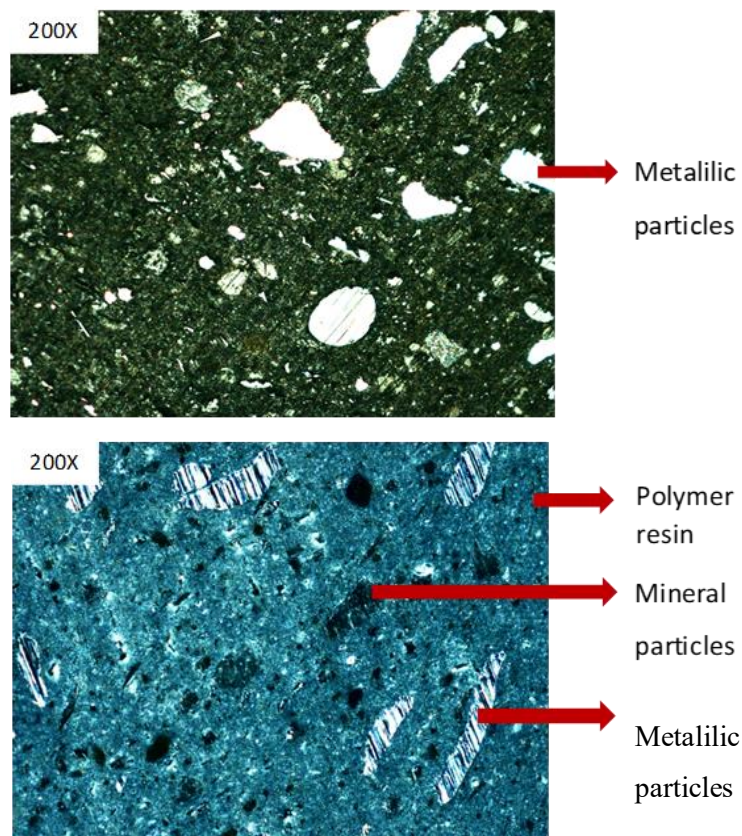


Figure 5.4. Images of the Sample Under an Optical Microscope Taken at 200X Magnification

It is seen that the content of clutch facing consists of different types of particles (metallic, mineral, fiber).

5.1.2. Mineral Phase Analysis (X-Ray Diffraction (XRD) Analysis)

Fig. 5.5. shows the phase analysis result of commercial friction material. As a result of the XRD analysis, we examined the properties of the materials. As you can see, compound names of possible phases that present in the commercial sample are listed according to scale factor.

This analysis provides information about crystal phases. As can be seen from the analysis, there are many different crystals.

According to the data obtained from the XRD analysis, acicular/layered minerals such as Zincite (ZnO), Quartz (SiO₂), Calcite (CaCO₃) and Sulphate mineral (SrSO₄) and also phlogopite (vermiculite), antigorite (talc, serpentine) attract attention as fillers.

Lubricants (graphite, Sulfides: Zinc sulfide (ZnS)) and abrasives (hematite (Fe₂O₃), magnesium oxide (MgO), spinel powders: (Mg, Fe) (Cr, Fe)₂O₄, (NiFe₂O₄) as friction modifiers) and other possible are phases.

Reinforcements are short glass fibers and metallic chips/powders (Iron (Fe) alloy).

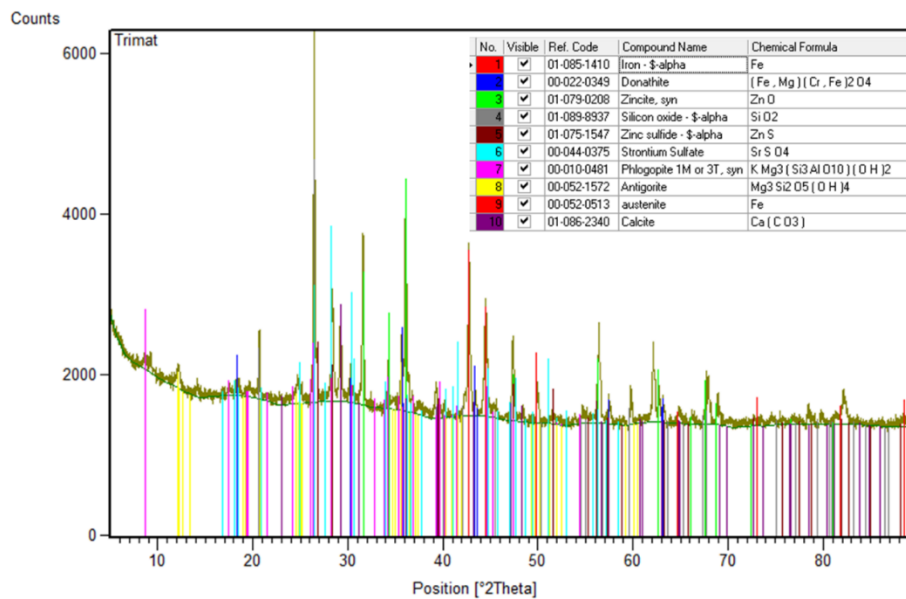


Figure 5.5. XRD Phase Analysis Results

5.1.3. Thermal Analysis (Thermogravimetric Analysis-TGA)

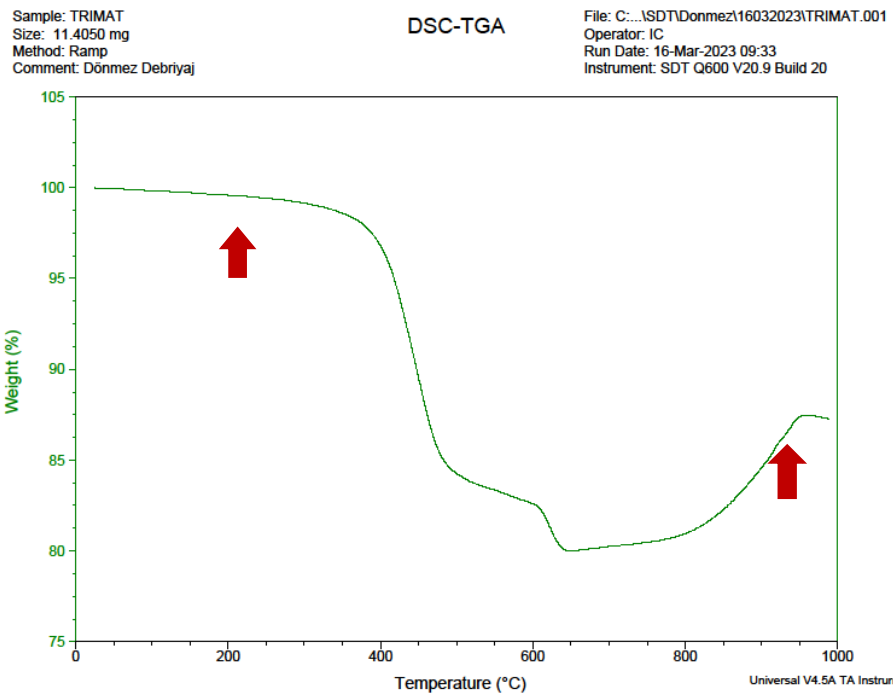


Figure 5.6. TG/DTA Analysis Results

The material we analyzed is an organic composite material, so thermogravimetric Analysis was performed to detect the amount of resin in it and some decomposed components.

As a result of the TGA analysis, when Fig. 5.6. is examined, the commercial brake pad sample heated up to 1000°C showed the reactions mentioned below. It is observed that approximately 15% weight loss occurs in the range of 350-450°C. It is thought that the reason for this reaction may be polymer resin degradation and/or dehydroxylation of minerals. In the literature, the permanent, light, and mid-medium decomposition products released during the heat treatment of a phenol formaldehyde (phenolic) resin have been characterized using TGA-Micro GC/MS. In this study, the degradations occurring in the resin structure support our work.³⁹ In another research conducted in the literature, FTIR test was carried out on phenolic resin heated to 1200°C, which is a sample prepared to ensure complete combustion of the resin down to carbonaceous residues. When this study was examined, it was observed that there was a similarity between the transition temperatures.⁴⁰

It can be interpreted that the reaction occurring at approximately 600-650°C will occur due to calcite decomposition.

The fact that the weight begins to increase as the temperature exceeds 700°C is explained by the oxidation of Fe-based metals.

Total weight loss up to 650°C is approximately 20%.

When commenting on the content that may arise as a result of the TGA transaction, the percentages stated below will be taken into account. These percentages are important to understand the composition of the pad material and evaluate its properties:

Table 5.1. TG/DTA Analysis Results

Material	Percent Content [%]
Polymer resin	~ %10-15
Calcite	~ %5
Hydrated minerals	~ %3-5
Fe metal	~ %8-25

5.1.4. Microstructural Analyzes (SEM-EDS Analysis: Scanning Electron Microscopy-Energy Dispersive X-ray Spectroscopy)

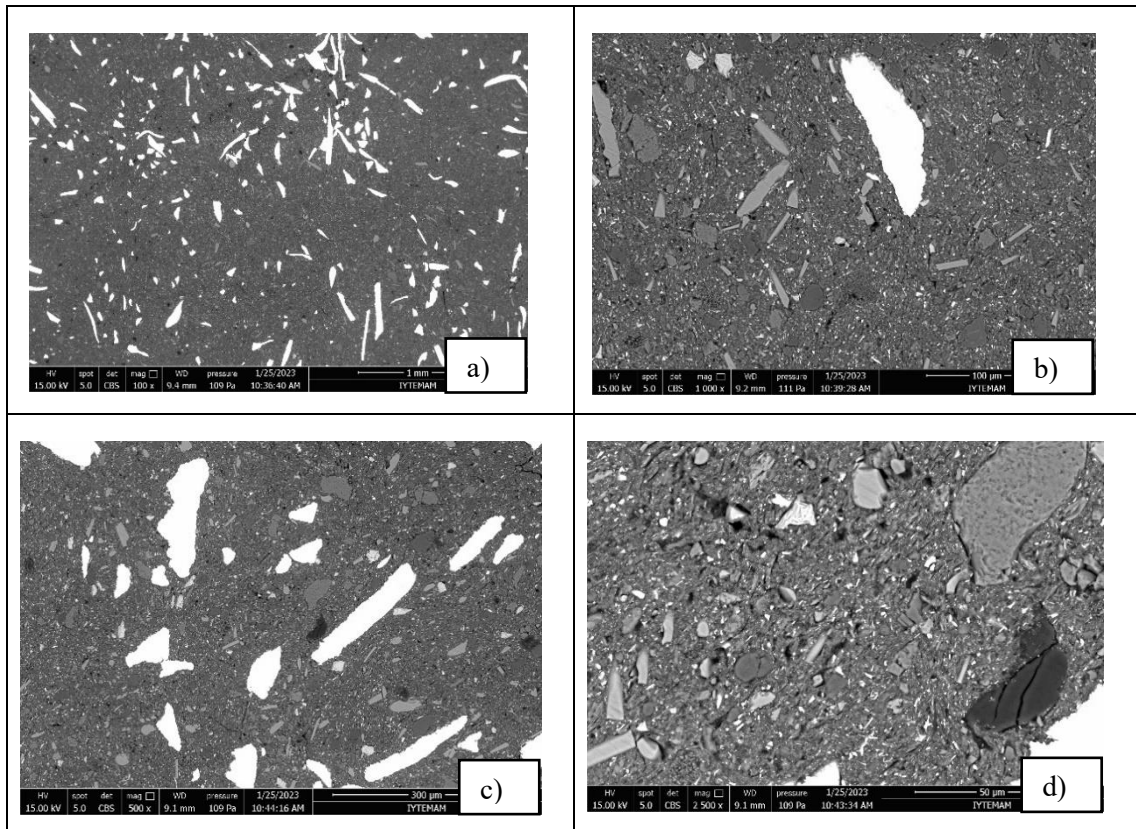
Studies have been conducted to determine the microstructure and chemical content of the components by SEM analysis.

According to the SEM images taken at different magnifications from different parts of the commercial sample, it is seen that the bright white particles are particles in the form of metallic fibers and sawdust. The sizes of these particles vary, some reaching lengths of up to 400 microns, and they generally have dimensions of 100 microns and below. In addition to metal fibers, the presence of a small amount of smooth and chipped glass fibers (<100 microns) is also observed in gray color within the brake pad sample. Apart from these, it is seen that different particles in angular, angular and fibrous forms with different large and small morphologies are densely present in the body and these particles are below 50 microns.

There are also particles of different color tones (bright and dark) with very fine particle sizes. According to the SEM-EDS analysis taken from the entire examined surface of the brake facing sample, it was confirmed that the elements of the crystal phases and compounds detected in the XRD analysis were present in the structure.

It is noteworthy that there is a high concentration of Fe element in the examined brake pad. Apart from this, the presence of C (carbon) indicates the presence of organic resin, carbonaceous and carbonate compounds in the body. Other elements such as Si, Zn, Mg, Ca, S and O indicate oxide, sulfate and sulfide compounds.

Since EDS analysis was not taken on each particle in the SEM images in the analyzes carried out at IYTE-MAM, the analyzes were examined again at MCBÜ-DEFAM for the subsequent SEM-EDS analysis and examined in detail.



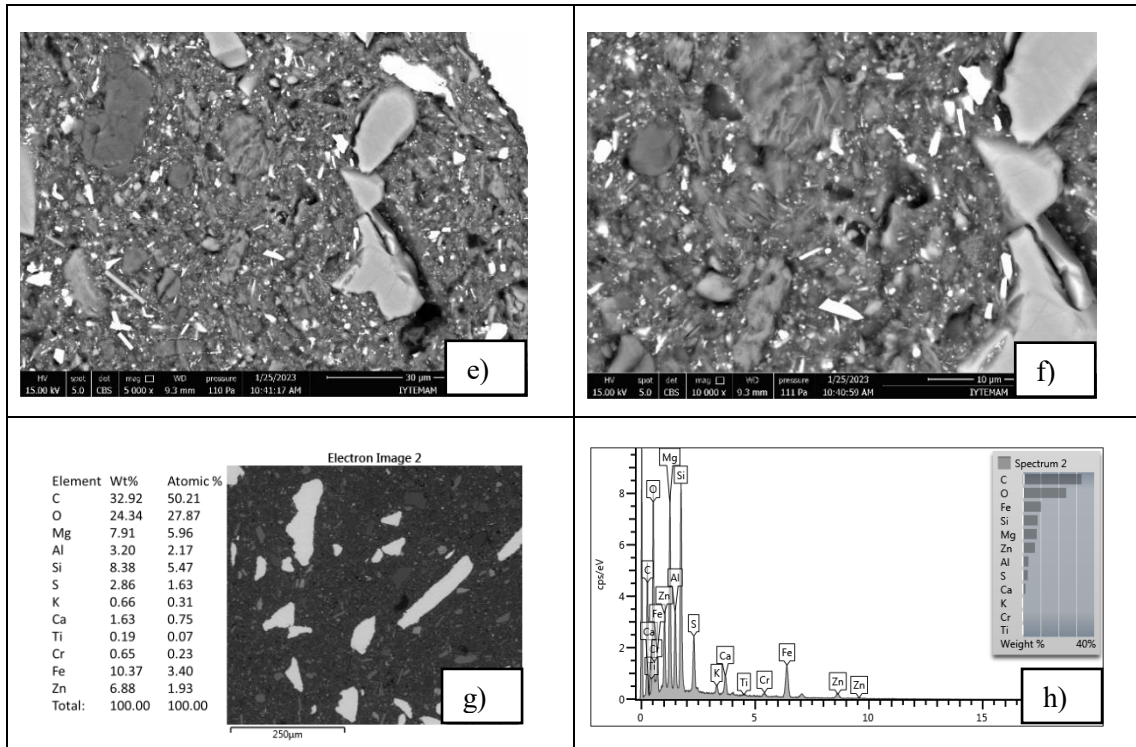


Figure 5.7. Images of the Sample Taken with Different Magnifications in SEM Analysis a) Taken at 100X Magnification, b) Taken at 1000X Magnification, c) Taken at 500X Magnification, d) Taken at 2500X Magnification, e) Taken at 5000X Magnification, f) Taken at 10000X Magnification, g) Weight and Atomic Percentage, h) Percentage Content

As a result of the SEM analysis performed on commercial brake pad samples, the SEM image and EDS results in Fig. 5.7., the signals taken from the region of each picture, are shown below the picture in Fig. 5.8.

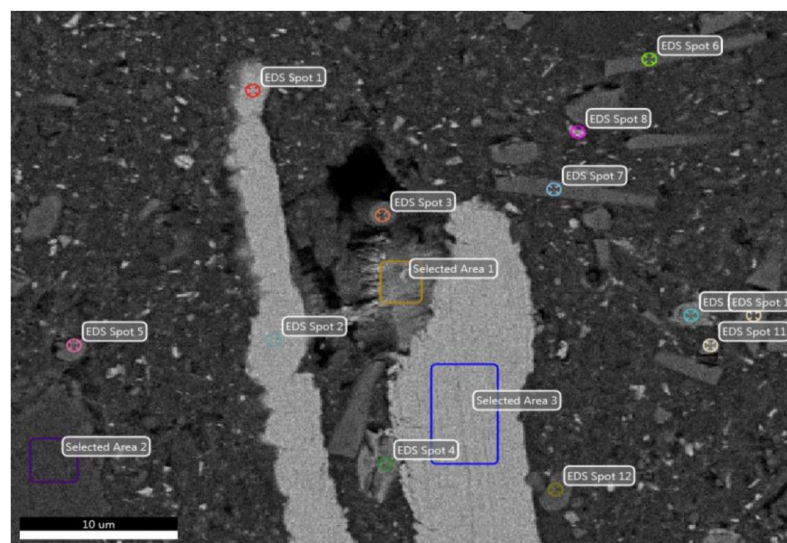


Figure 5.8. The signals taken from the region

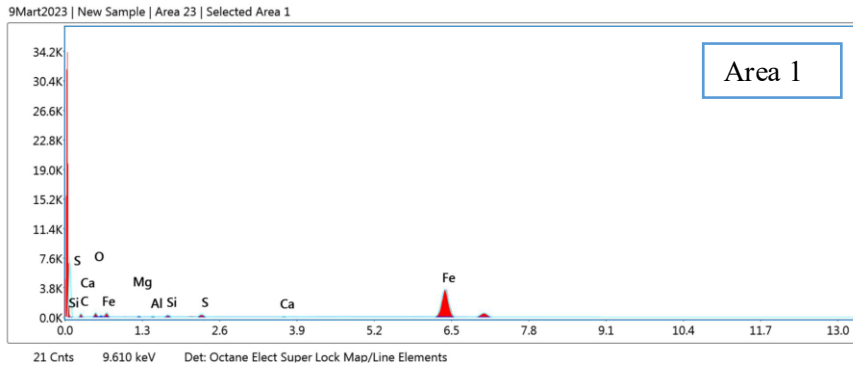


Figure 5.9. SEM Analysis Image of the Sample Taken 1

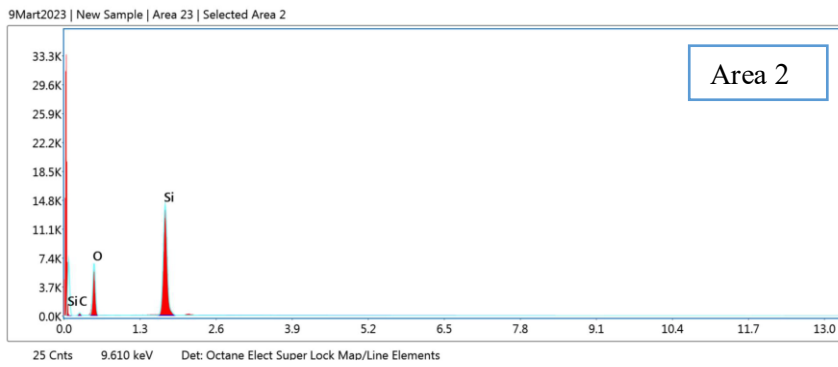


Figure 5.10. SEM Analysis Image of the Sample Taken 2

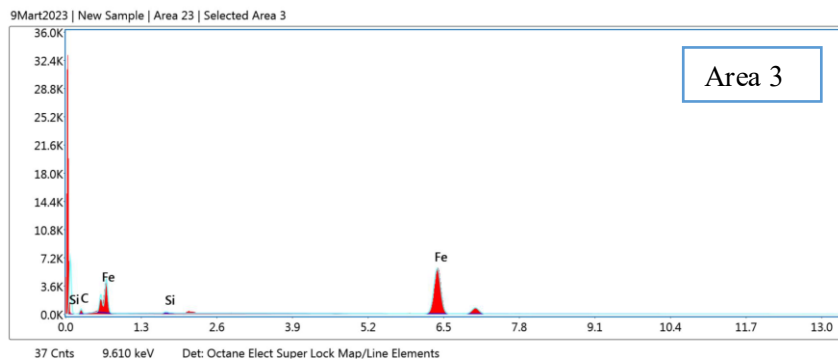


Figure 5.11. SEM Analysis Image of the Sample Taken 3

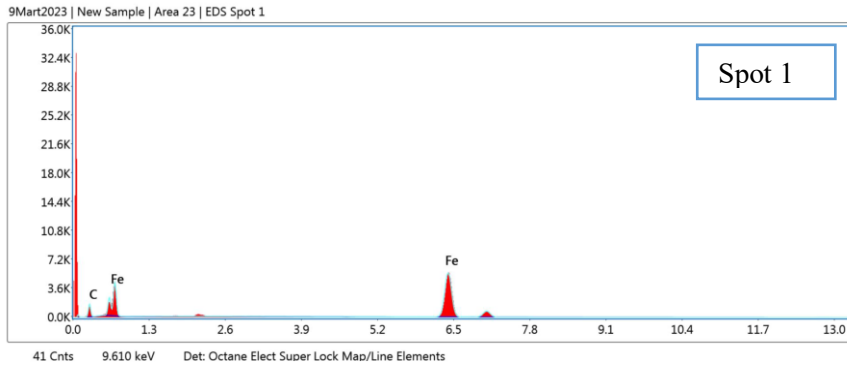


Figure 5.12. SEM Analysis Image of the Sample Taken 4

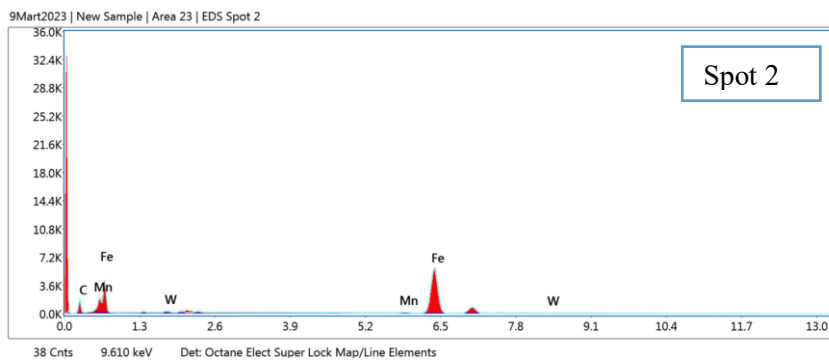


Figure 5.13. SEM Analysis Image of the Sample Taken 5

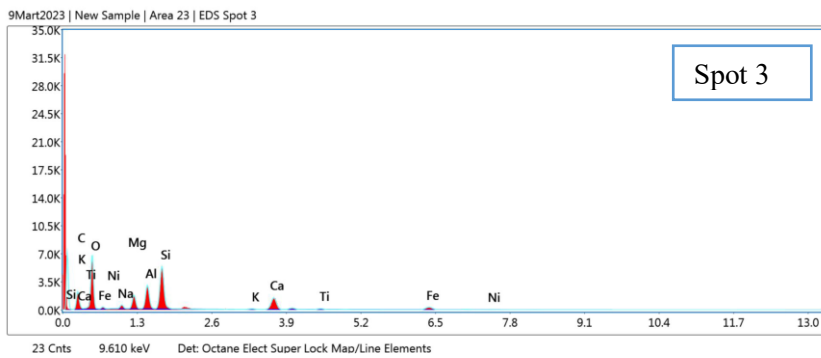


Figure 5.14. SEM Analysis Image of the Sample Taken 6

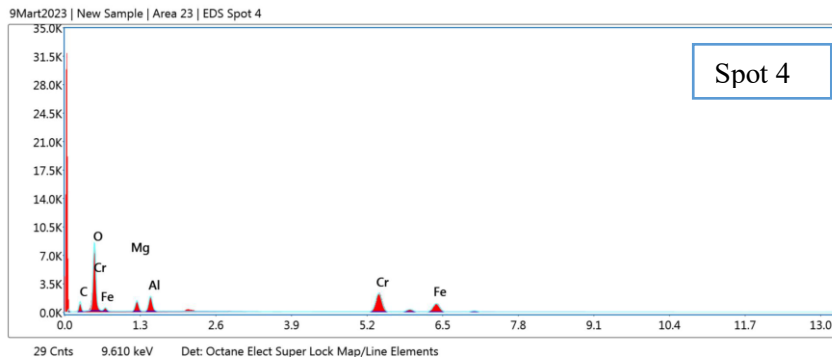


Figure 5.15. SEM Analysis Image of the Sample Taken 7

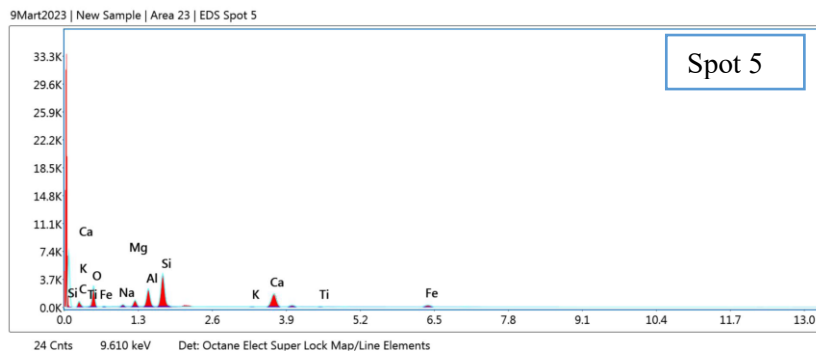


Figure 5.16. SEM Analysis Image of the Sample Taken 8

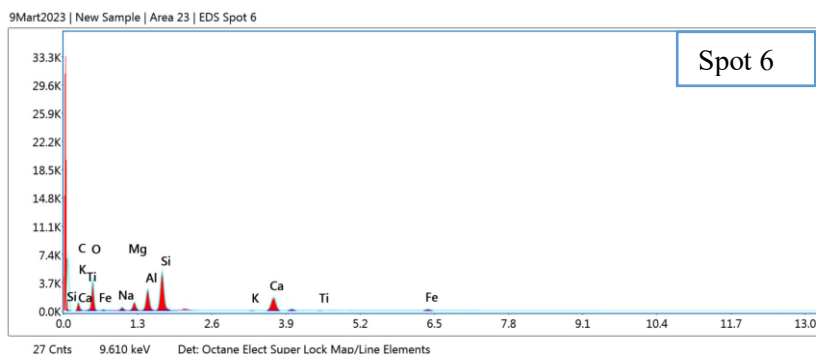


Figure 5.17. SEM Analysis Image of the Sample Taken 9

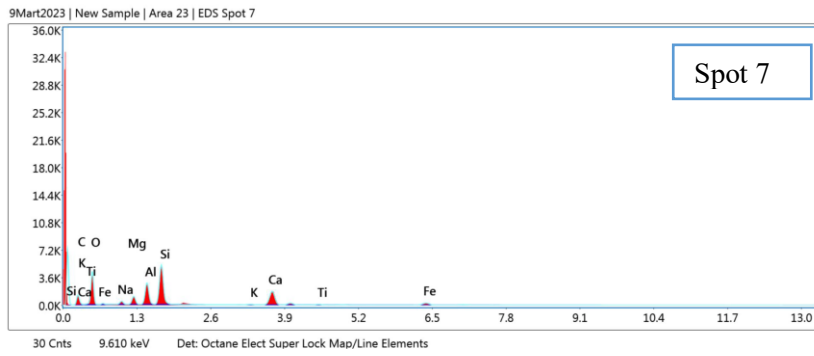


Figure 5.18. SEM Analysis Image of the Sample Taken 10

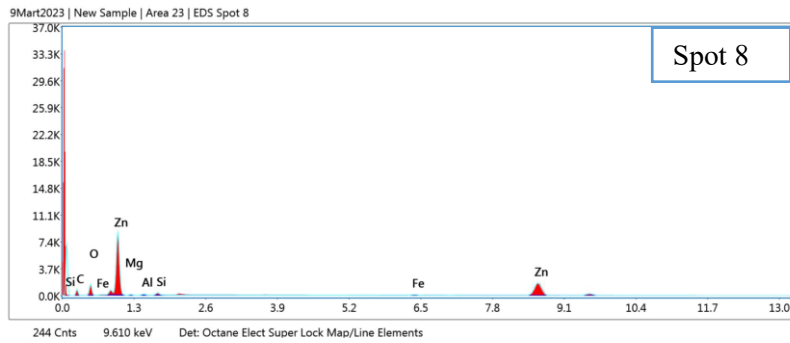


Figure 5.19. SEM Analysis Image of the Sample Taken 11

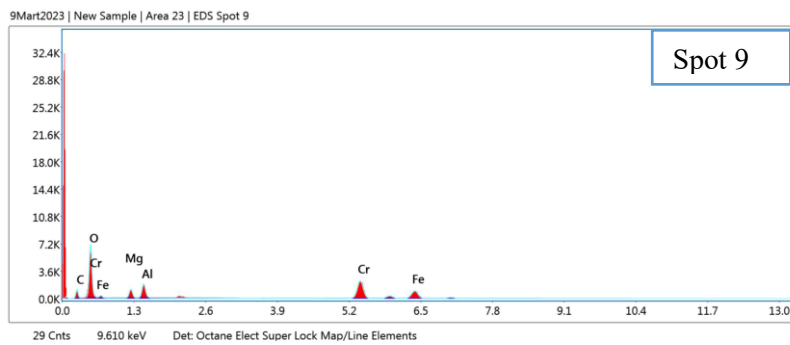


Figure 5.20. SEM Analysis Image of the Sample Taken 12

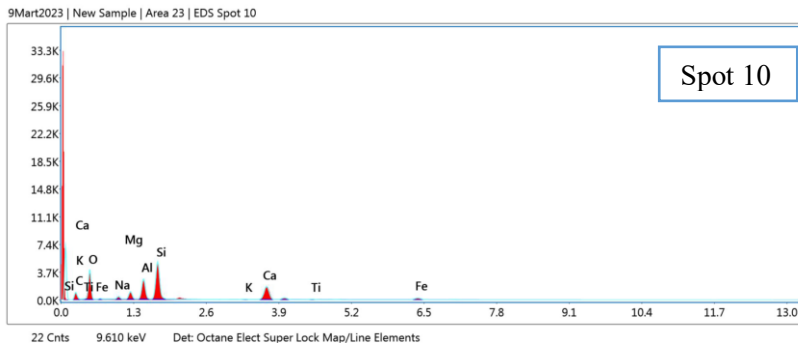


Figure 5.21. SEM Analysis Image of the Sample Taken 13

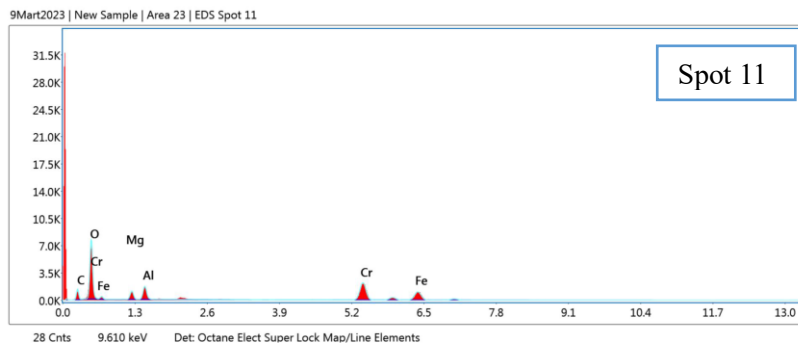


Figure 5.22. SEM Analysis Image of the Sample Taken 14

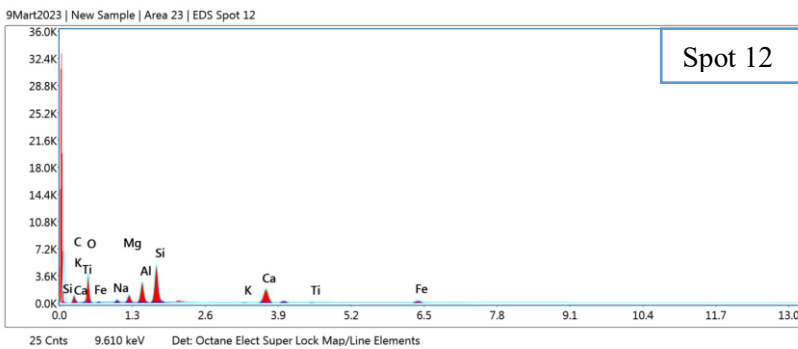


Figure 5.23. SEM Analysis Image of the Sample Taken 15

According to SEM-EDS analysis, it is understood that the particles containing metallic iron are Fe-alloy and contain alloying elements such as Mn, Cr, W. It can be said that particles containing Si and O are quartz particles. They are the ZnS and ZnO components of the brightly colored fine particles within the Zn-containing brake pad. Apart from these, it can be said that particles containing Mg, Al, Si are silicate minerals and fillers.

5.2. Results and Evaluation of Produced Facing Samples

5.2.1. Mechanical Properties of Test Samples

5.2.1.1. Wear and Friction Tests

The tribology test results performed on the facing samples in order to determine the tribological properties of the materials and the pressure force and friction force calculations based on them are given in Table 5.2.

Table 5.2. Friction Coefficients and Force Calculations Based on Wear Test

Sample	Start [μ]	Min [μ]	Max [μ]	Mean [μ]	Mass [gr]	Weight [N]	Pressure Force [N/mm ²]	Frictional Force
1	0.141	0.141	0.195	0.182	5.117	50.193	0.631	9.158
2	0.166	0.166	0.619	0.533	10.143	99.503	1.252	53.052
3	0.140	0.140	0.556	0.437	9.926	97.377	1.225	42.546
4	0.123	0.123	0.243	0.208	10.064	98.729	1.242	20.502
5	0.164	0.164	0.291	0.296	9.899	97.108	1.222	25.732
6	0.255	0.255	0.304	0.278	10.083	98.911	1.244	29.293
7	0.202	0.202	0.291	0.265	9.943	97.538	1.227	27.118
8	0.128	0.128	0.242	0.205	9.941	97.524	1.227	19.989
9	0.186	0.186	0.302	0.209	9.893	97.050	1.221	20.324
10	0.142	0.142	0.287	0.219	9.881	96.928	1.219	21.255
11	0.208	0.208	0.287	0.275	9.883	96.956	1.220	26.658
12	0.164	0.164	0.224	0.218	9.930	97.412	1.226	21.260
13	0.226	0.226	0.282	0.205	9.913	97.250	1.224	19.890

The statistics of the facing samples obtained are visualized with the friction coefficient-sample and friction force-sample figures below in Fig. 5.24. and Fig. 5.25. The relation of friction coefficient and sliding force was revealed in Appendix A.

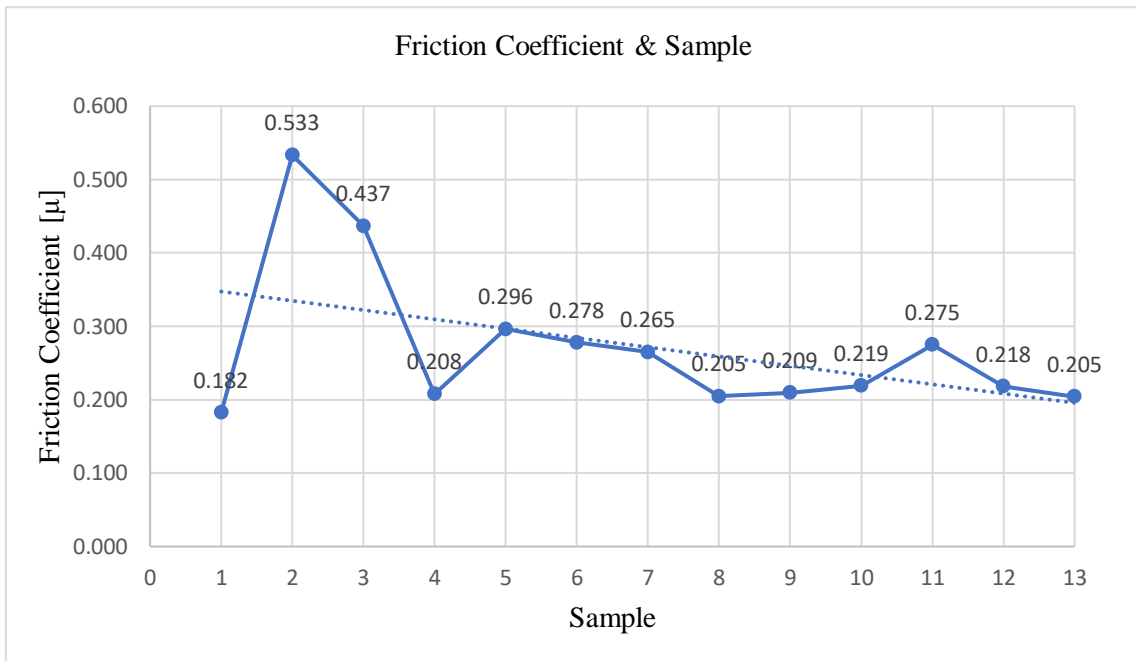


Figure 5.24. Friction Coefficient-Sample

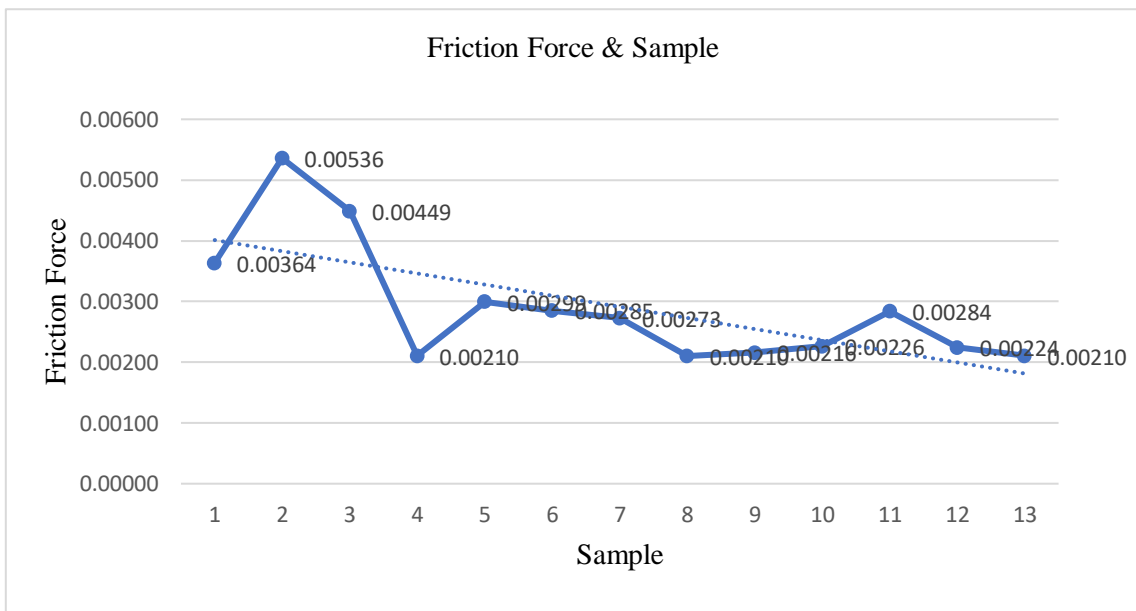


Figure 5.25. Friction Force-Sample

2-3-4: Quartz is a harder material than steel; therefore, increasing quartz is expected to increase the friction coefficient. Alumina is often used as a grinding or polishing agent because of its high hardness and good wear resistance. Considering the literature reviews, it is expected that the friction coefficient will decrease by reducing the alumina ratio.

As seen in the Friction K. & Sample graph in the experiment with the increase of quartz and decrease of alumina; A decrease in the friction coefficient, and the friction force proportional to it was observed. It can be interpreted that when quartz is used instead of alumina the friction coefficient is balanced. Alumina has a higher rate of increasing the friction coefficient value compared to quartz. In this way, it is understood that alumina is more effective on the friction coefficient than quartz. In this case, a result that supports the literature study was obtained.

5-6-7: In this experiment, it is desired to limit the amount of wear by increasing the phenolic resin and bringing it to the maximum level. Actions taken to reduce the material breaking off from the surface will reduce wear. In this case, the filler and resin must adhere well. At the same time, when literature studies are examined, it is known that increasing the amount of resin has a reducing effect on the coefficient of friction. As a result of the tests, it was observed that the coefficient of friction decreased in proportion to the increasing amount of resin, and it can be interpreted that 19% resin amount is ideal for this recipe. ³⁴

8-9-10: Glass fibers, one of the strengthening raw materials, provide extreme convenience during application due to their light weight. Although it has a light structure, its durability provides the opportunity to achieve quality in every field it is used. In this case, it is expected that the amount of wear will decrease by increasing the glass fiber. In addition, considering the literature reviews, it is expected that the friction coefficient will increase with the increase in glass fiber ratio. In the study, it is observed that the friction coefficient increases as the glass fiber ratio increases from the 8th sample to the 10th sample. In this case, a result that supports the literature study was obtained.

11-12-13: It is a compression-resistant material and is used to reduce or prevent wear of working parts as they rub against each other. It is aimed to reduce the friction coefficient by increasing the amount of use. As a result of increasing the amount of graphite in lubricants; It was observed that the sample friction coefficient decreased.

The results recorded by measuring the mass before and after wear and the results obtained by the calculations are given in Table 5.3.

Table 5.3. Sample Mass and Volume Losses Calculation

Sample	Starting Mass [gr]	Mass after wear [gr]	Mass Loss [gr]	Density [mm ³]	Volume Loss [gr/mm ³]
1	5.117	5.006	0.111	0.002	51.946
2	10.143	10.040	0.103	0.002	44.634
3	9.926	9.838	0.089	0.002	38.705
4	10.064	9.990	0.074	0.002	34.246
5	9.899	9.826	0.073	0.002	32.535
6	10.083	10.020	0.063	0.002	28.999
7	9.943	9.873	0.070	0.002	37.444
8	9.941	9.868	0.074	0.002	35.359
9	9.893	9.804	0.089	0.002	40.627
10	9.881	9.795	0.085	0.002	36.530
11	9.883	9.813	0.070	0.002	33.395
12	9.930	9.852	0.078	0.002	38.345
13	9.913	9.826	0.087	0.002	42.020

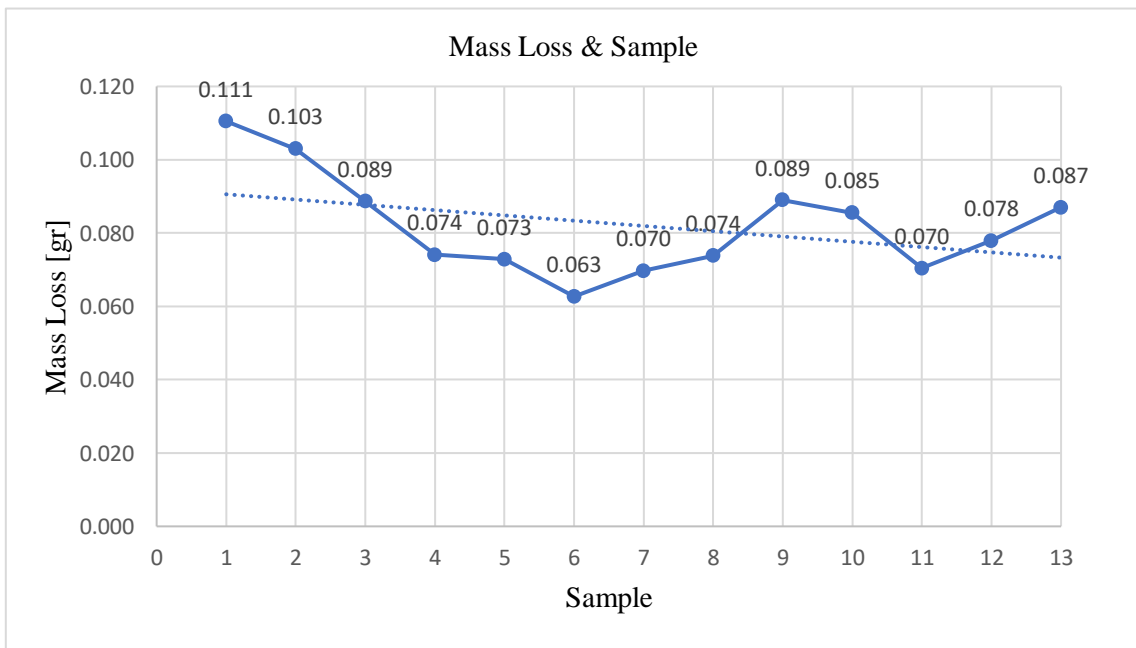


Figure 5.26. Mass Loss-Sample Graph

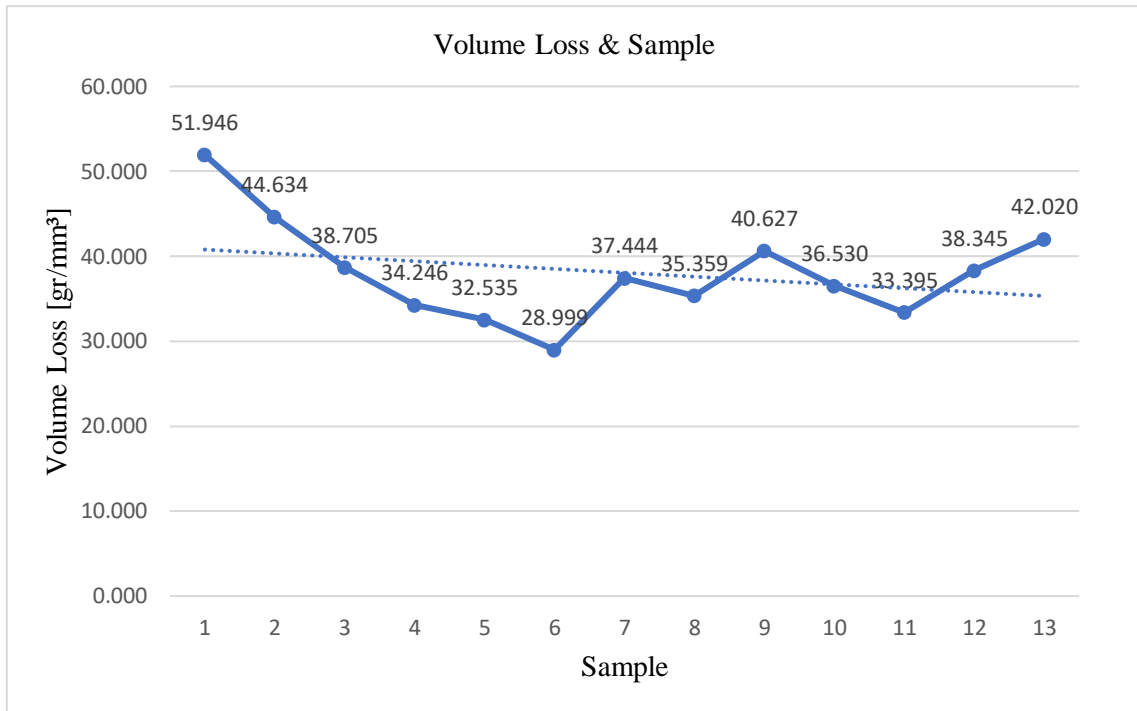


Figure 5.27. Volume Loss-Sample Graph.

5.2.1.2. Hardness Tests

Hardness measurements of the samples were carried out with the SHORE D Calibration Set measurement method. Each sample was measured 3 times. the number of samples being 13. Then, the averages of the measured hardnesses were calculated in order to minimize external errors. The results are in Table 5.4.

Table 5.4. SHORE D Calibration Set Hardness Measurement

Sample	SHORE D			
	Trial1	Trial2	Trial3	Average
Trimat MN2221	81	82	82	81.667
1	82	82	82	82
2	76	78	77	77
3	80	77	79	78.667
4	79	80	79	79.333
5	80	78	80	79.333
6	80	81	80	80.333
7	83	83	82	82.667
8	80	82	83	81.667
9	83	82	85	83.3
10	80	80	84	81.3
11	73	72	74	73
12	85	84	86	85
13	85	84	86	85

The average shore hardness value of the commercial Trimat friction sample was measured as 81.667. While the 8th mixture gave the same value, mixtures coded 1, 7, 9, 12 and 13 showed slightly higher hardness values. The highest hardness was measured as 85 shores in the 12th and 13th samples. The hardness value decreased slightly in the other mixtures, and the 11th sample showed the lowest hardness value.

The first recipe composition, prepared by analogy with the commercial sample recipe. presented a hardness value very close to the hardness value of the Trimat sample.

An increase in the hardness value of quartz mixtures was observed in the recipes prepared according to the alumina-quartz replacement ratio in the 2nd, 3rd and 4th pad compositions. This may be due to the use of alumina, which has submicron properties compared to quartz.

The effect of changing different phenolic phenolic ratios in the 5th, 6th and 7th pad compositions were examined. Accordingly, the increase in resin ratio caused an increase in hardness values. While alumina was not used in these recipes, quartz was used at the highest rate.

The effect of the increase in glass fiber ratio in the 8th, 9th and 10th pad compositions were examined. Increasing the glass fiber ratio at a certain rate caused the hardness value to increase. Increasing it at a higher rate showed the same hardness value again.

The effect of increasing the rate of sulphide component in the 11th, 12th and 13th pad compositions were examined. Accordingly, the change in sulfide ratio caused the hardness to increase.

5.2.2. Sample Physical Properties

5.2.2.1. Density Calculation Experiments

Density calculations were made from the mass-volume relationship by calculating the volume using physical measurements taken before wear. Calculation results are as in Table 5.5.

Table 5.5. Density Calculation Obtained from Mass-Volume Relationship

Sample	Mass [gr]	Diameter [mm]	Height [mm]	Volume [mm ³]	Intensity [gr/cm ³]
1	5.117	25	4.90	2405.282	2.13
2	10.143	24.4	9.40	4395.390	2.31
3	9.926	25.25	8.65	4331.405	2.29
4	10.064	25.1	9.40	4651.202	2.16
5	9.899	25	9.00	4417.865	2.24
6	10.083	25	9.50	4663.302	2.16
7	9.943	25.45	10.50	5341.396	1.86
8	9.941	25.4	9.40	4763.050	2.09
9	9.893	25	9.20	4516.039	2.19
10	9.881	25	8.60	4221.515	2.34
11	9.883	25.2	9.40	4688.337	2.11
12	9.930	25.2	9.80	4887.841	2.03
13	9.913	25.2	9.60	4788.089	2.07

The density value of the first prescription composition prepared by analogy with the commercial sample was measured as 2.13 g/cm³.

In the recipes, prepared according to the alumina-quartz substitution ratio in the 2nd, 3rd and 4th brake pad compositions, the increase in the alumina ratio caused an increase in the density values. This is because the density of alumina powders is higher than that of quartz.

The effect of changing the different phenolic ratios in the 5th, 6th and 7th pad compositions were examined. Accordingly, increasing the resin ratio caused a decrease in density values.

In the 8th, 9th and 10th compositions, the effect of the increase in glass fiber ratio on density was examined. Increasing the glass fiber ratio caused the density value to increase.

The effect of increasing the graphite component ratio in the 11th, 12th and 13th pad compositions were examined. Accordingly, increasing the sulfite ratio caused the density to decrease.

5.2.3. Microstructural (SEM) Analysis of Test Samples

Microstructure images taken from the surface of Sample 2 that was not exposed to wear Fig. 5.28 and surface microstructure images subjected to Ball-on disc type wear are shown in Fig. 5.29. When these images are examined, the wear has a visible effect on the sample and wears the surface, as shown in Fig. It is understood that the smooth surface seen in 5.28 has lost its effect.

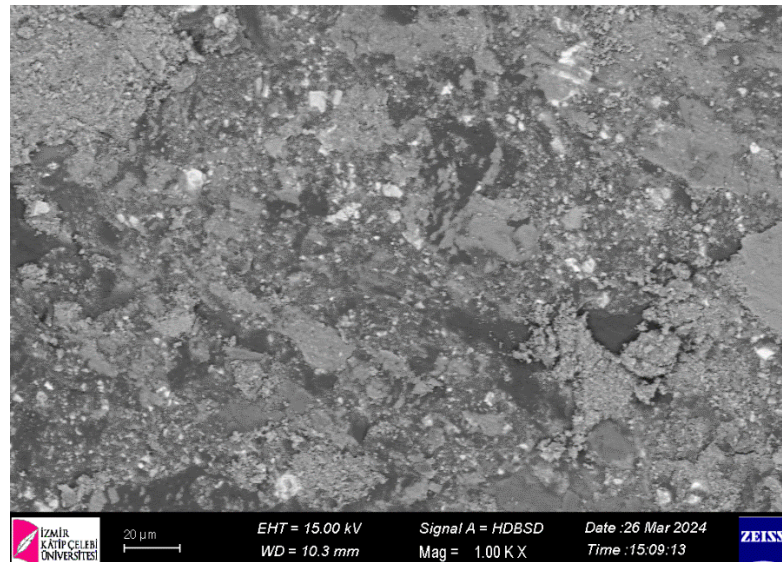


Figure 5.28. Sample 2 SEM Image

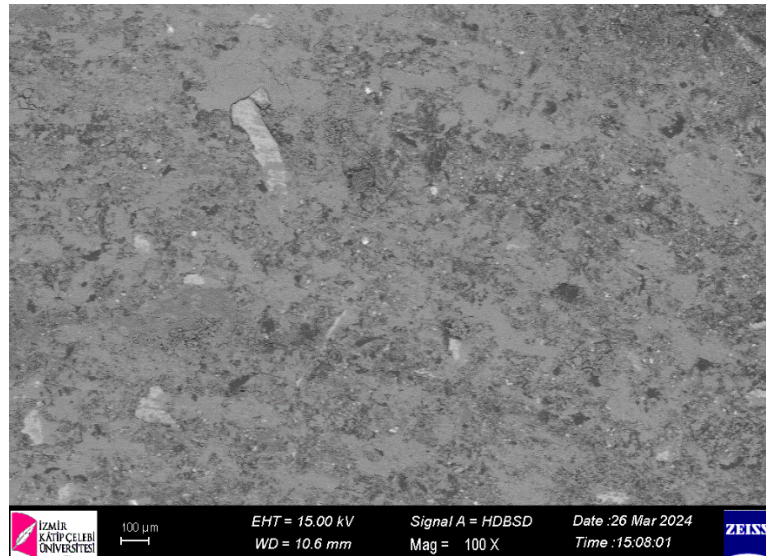


Figure 5.29. Sample 2 Worn Surface SEM Image

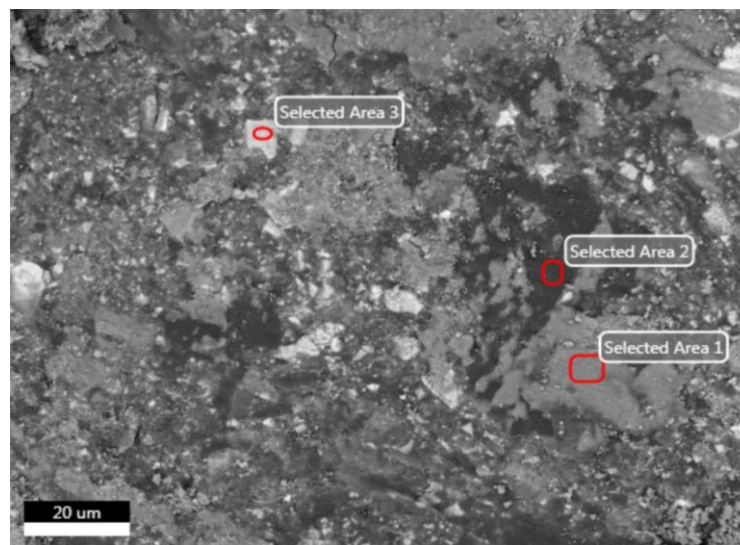


Figure 5.30. Sample 3 Worn Surface SEM Image

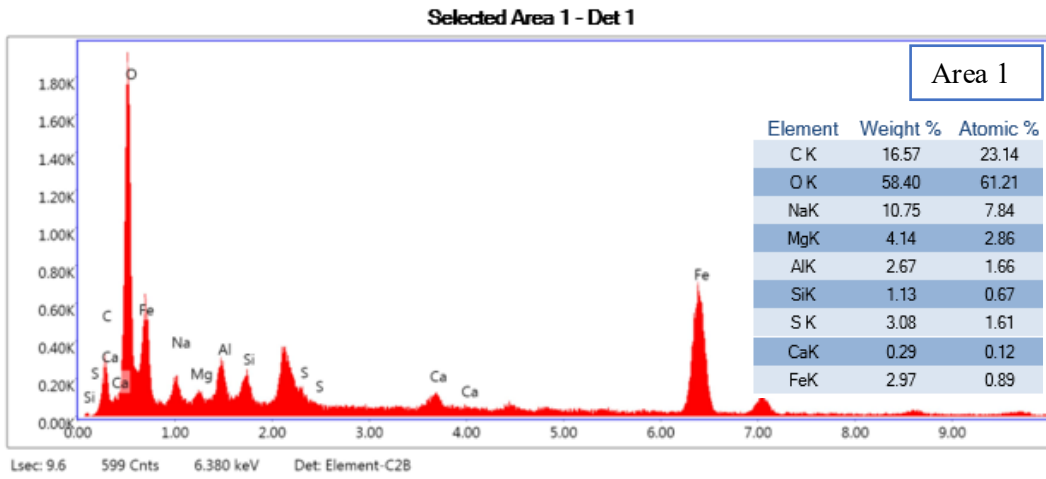


Figure 5.31. SEM Analysis Result 1

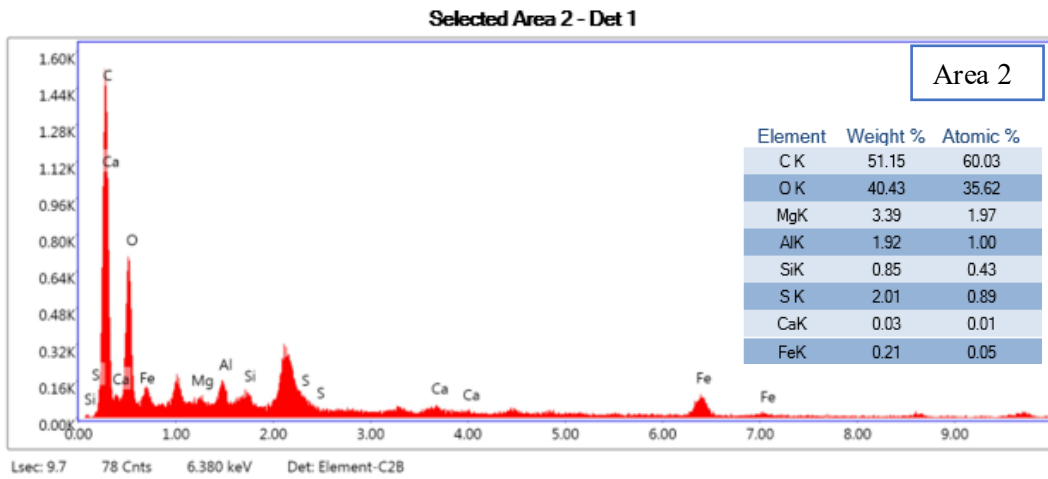


Figure 5.32. SEM Analysis Result 2

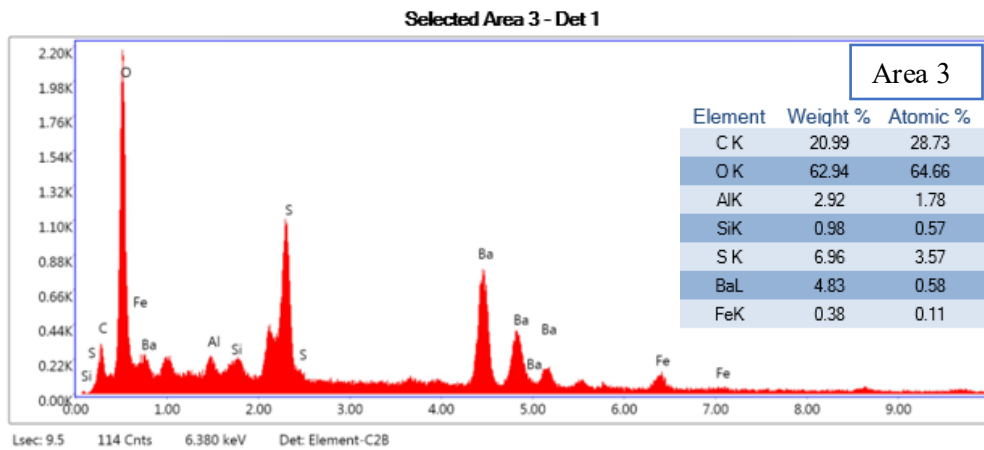


Figure 5.33. SEM Analysis Result 3

CHAPTER 6

CONCLUSIONS

As a result, in the study, experimental analysis of the structural components of safety clutch facings used in aviation applications was carried out. Characterization studies of commercial composite-based aircraft brake pad material were carried out and the microstructure, morphology and chemical content of the pad components were determined. For this purpose, methods such as optical microscope (OM), scanning electron microscope (SEM-EDS), phase analysis (XRD), chemical analysis (XRF), analysis of chemical bond structures (FTIR) and thermal analysis (TGA) have been used.

In the light of the studies carried out. New pad formulations have been created. As a continuation of the research, composite pads were produced under the fixed production conditions specified in Table 4.1. using different proportions of structural raw materials for safe clutch pads to be used in aviation applications.

Many experiments were carried out with varying additive materials and mixture ratios, and the differences in pad properties were interpreted based on the final 13 samples.

The research aimed to investigate the effects of varying the phenolic resin, quartz, alumina, glass fiber and graphite content of a predetermined clutch pad composite matrix on the friction force and wear resistance. In this context, pad compositions with different contents of components have been prepared. Three clutch pad samples were produced using different combinations of abrasive and filler, namely alumina (Al_2O_3) and quartz (SiO_2) (ranging from 0% to 8.5% by mass), and their friction properties were investigated. The same process was repeated by changing the amounts of phenolic resin alone (ranging between 17% and 19% by mass), glass fiber (ranging between 3.5% and 5.5% by mass) and graphite (ranging between 8.5% and 10.5% by mass).

The friction material with 7.5% Alumina (Al_2O_3) added gave the best results in terms of friction coefficient. Friction materials containing 8.5% Quartz (SiO_2) showed improvements in friction coefficient. Thus, the effect of the determined prescriptions on grasping performance was examined. It has been observed that Alumina has an ideal application effect on friction materials and gives positive results in grip performance.

The pad material with the most suitable formulation and optimum production process parameters was determined. From the results obtained, it is predicted that structural materials will have significant effects on the grip performance of the pad, and if used in appropriate proportions, the life of the pad will increase and provide more controlled and safer grip.

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APPENDIX

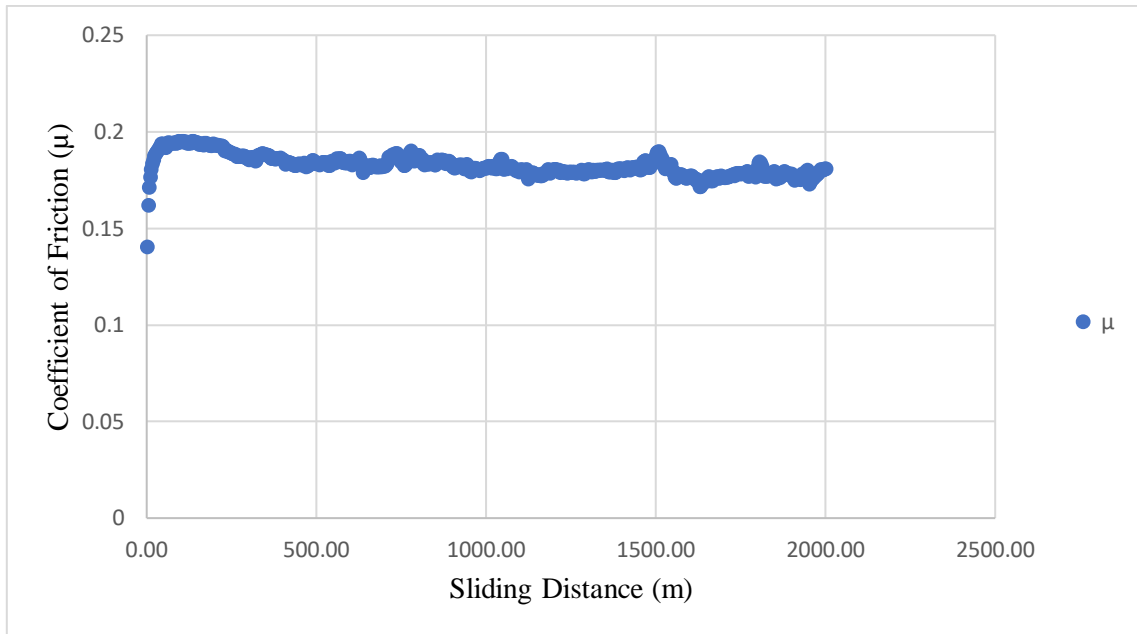


Figure A.1. MX.1. Coefficient of Friction Vs. Sliding Distance of sample 1

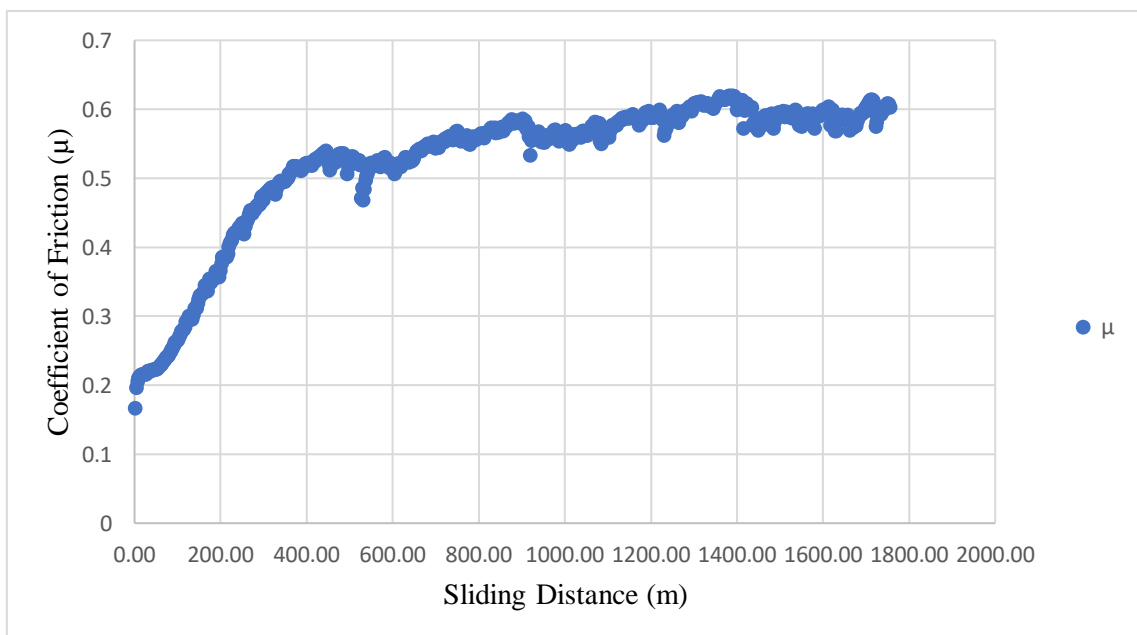


Figure A.2. MX.2. Coefficient of Friction Vs. Sliding Distance of sample 2

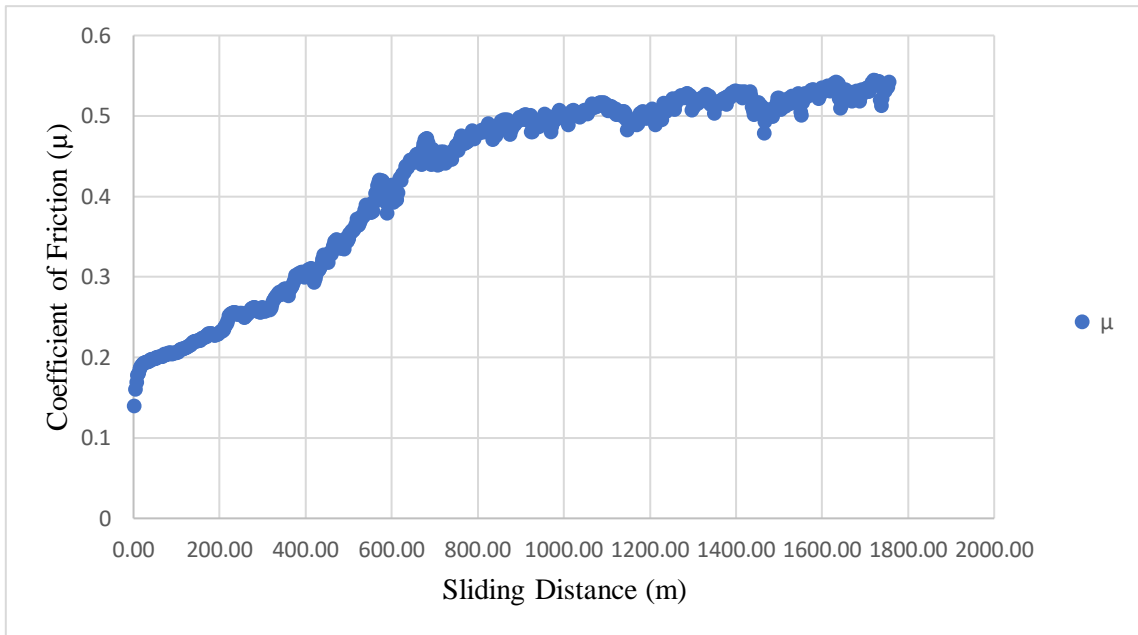


Figure A.3. MX.3. Coefficient of Friction Vs. Sliding Distance of sample 3

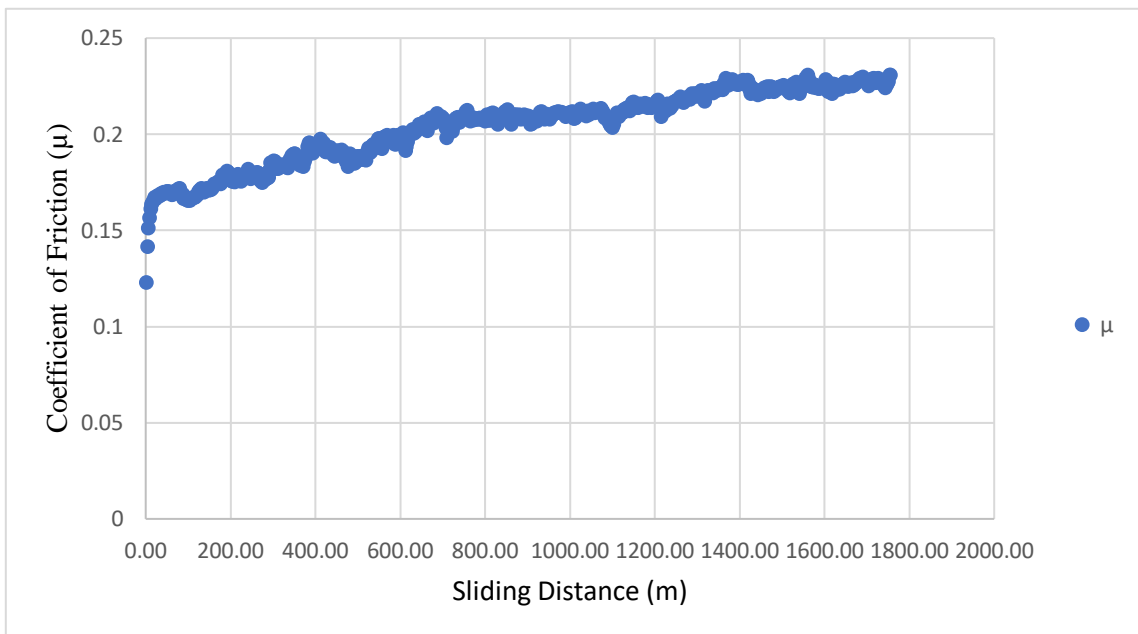


Figure A.4. MX.4. Coefficient of Friction Vs. Sliding Distance of sample 4

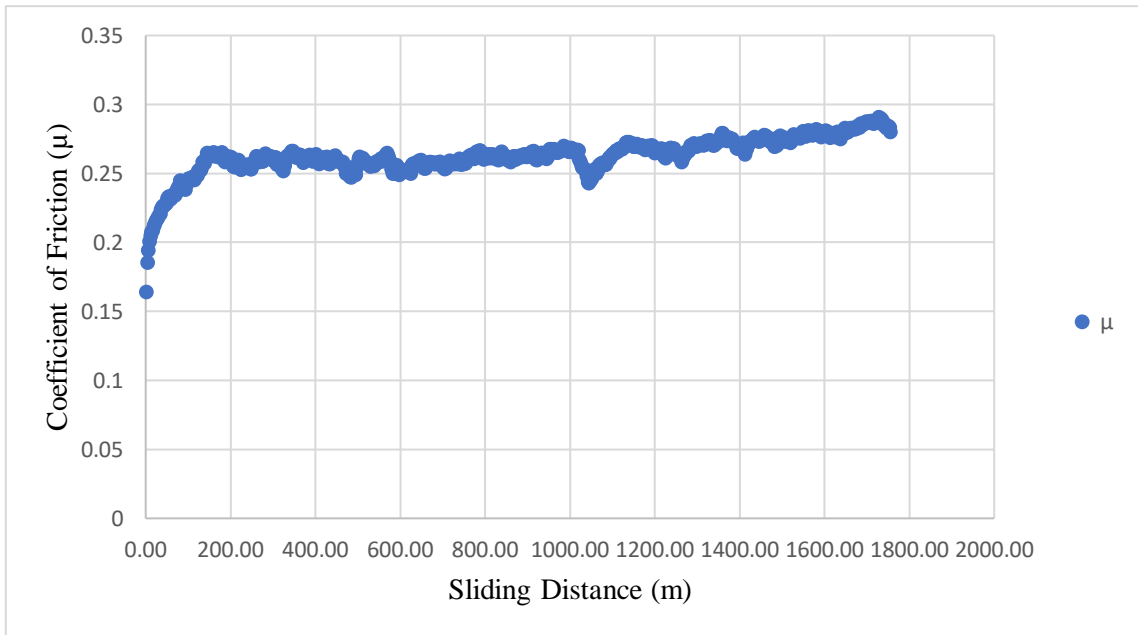


Figure A.5 MX.5. Coefficient of Friction Vs. Sliding Distance of sample Graph MX.5.

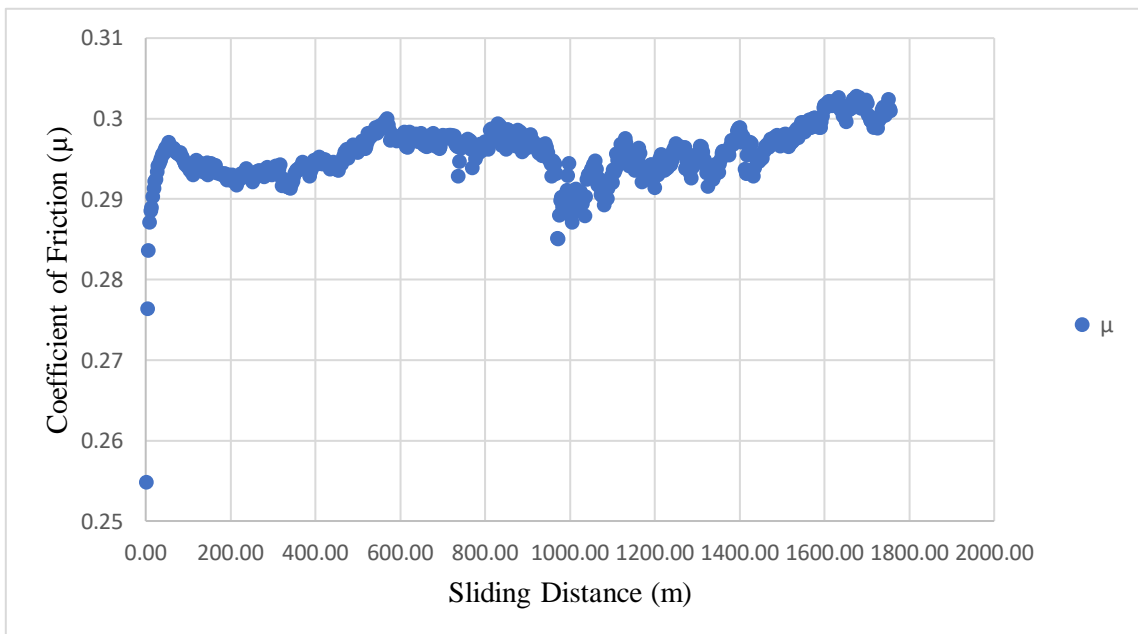


Figure A.6 MX.6. Coefficient of Friction Vs. Sliding Distance of sample 6

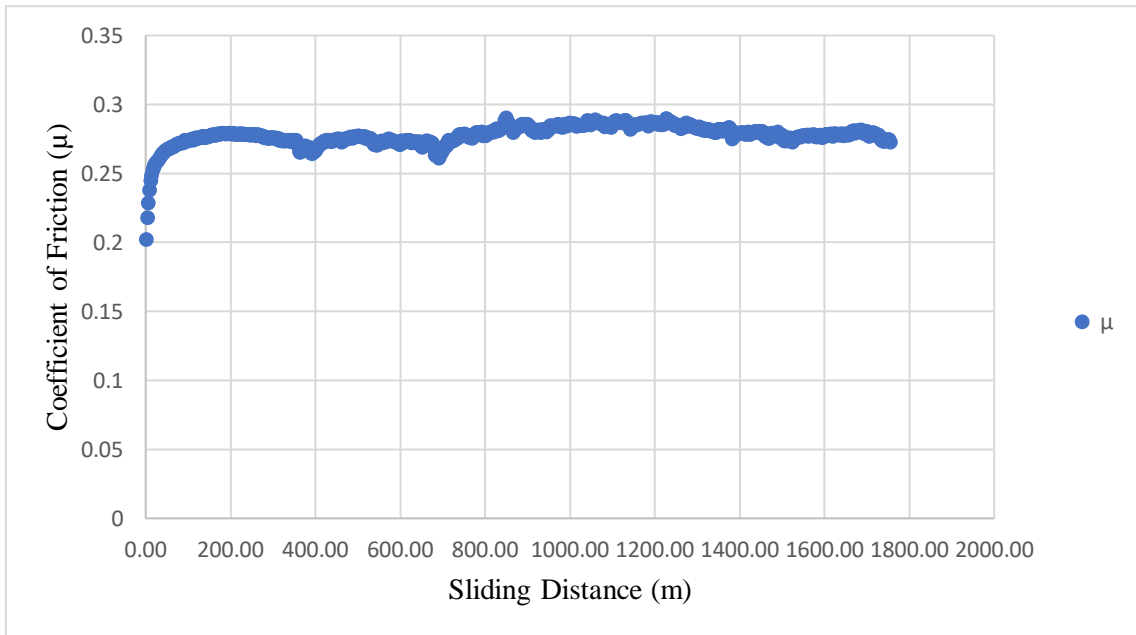


Figure A.7 MX.7. Coefficient of Friction Vs. Sliding Distance of sample 7

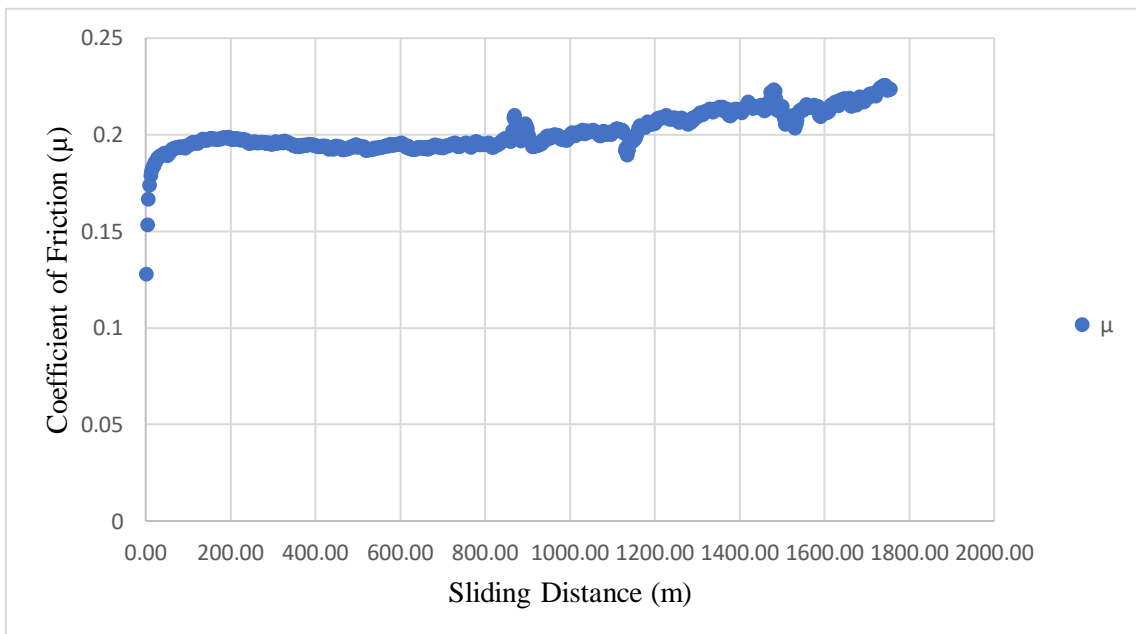


Figure A.8. MX.8. Coefficient of Friction Vs. Sliding Distance of sample 8

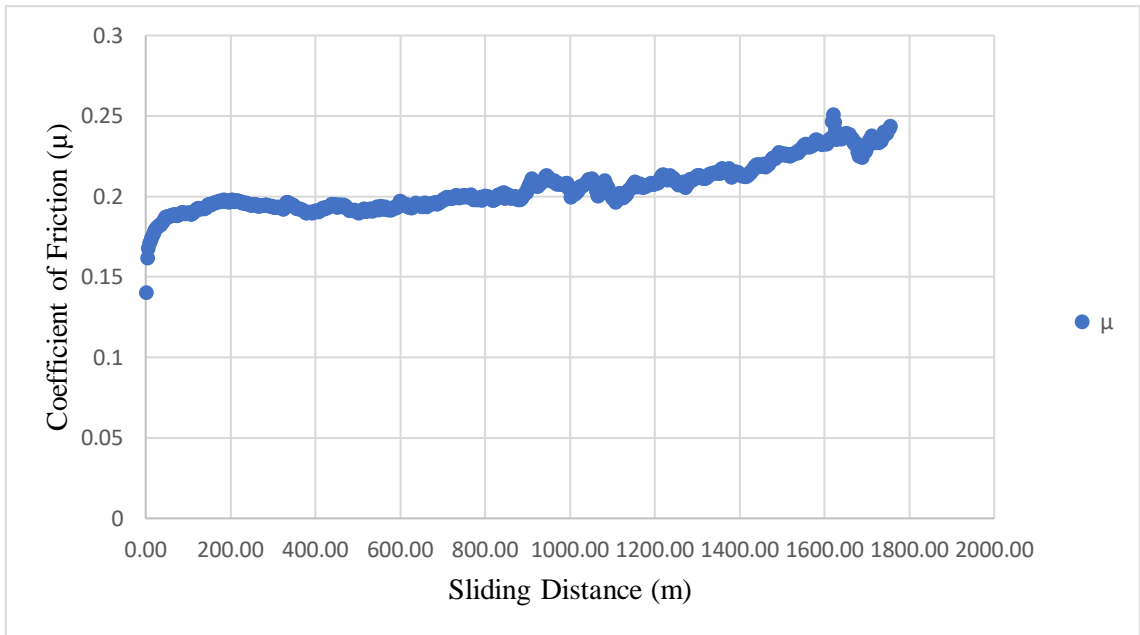


Figure A.9. MX.9. Coefficient of Friction Vs. Sliding Distance of sample 9

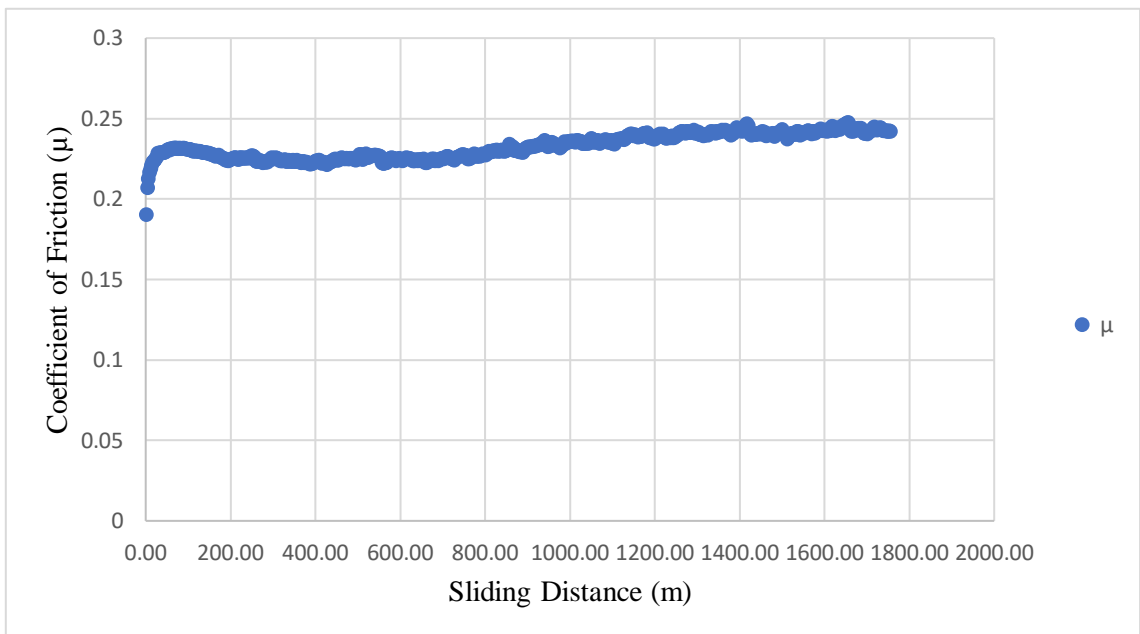


Figure A.10 MX.10. Coefficient of Friction Vs. Sliding Distance of sample 10

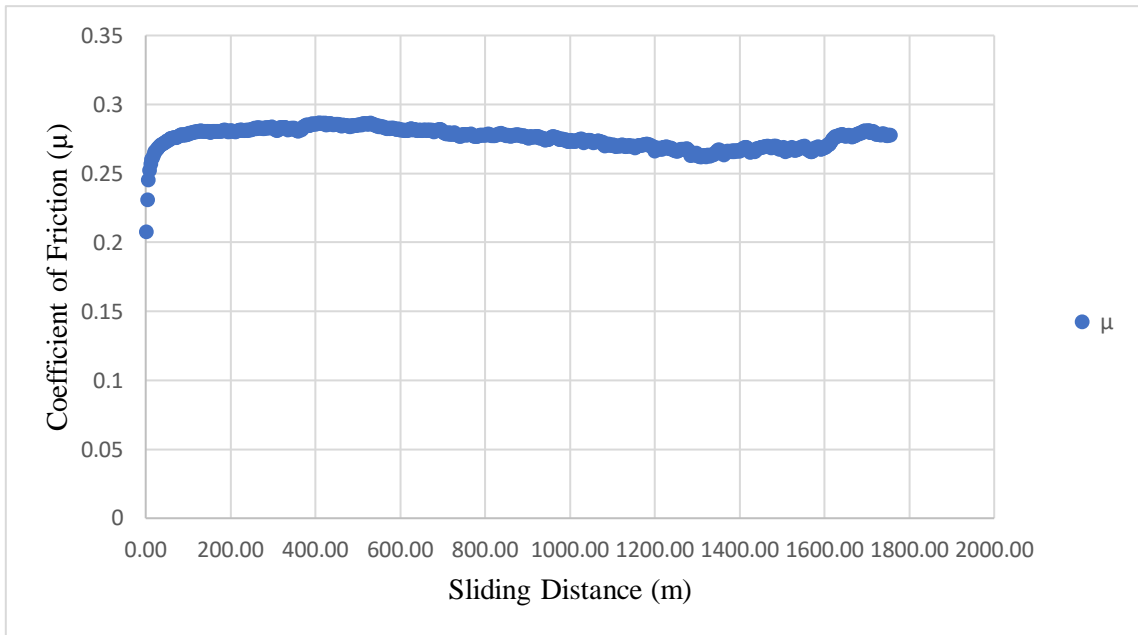


Figure A.11 MX.11. Coefficient of Friction Vs. Sliding Distance of sample 11

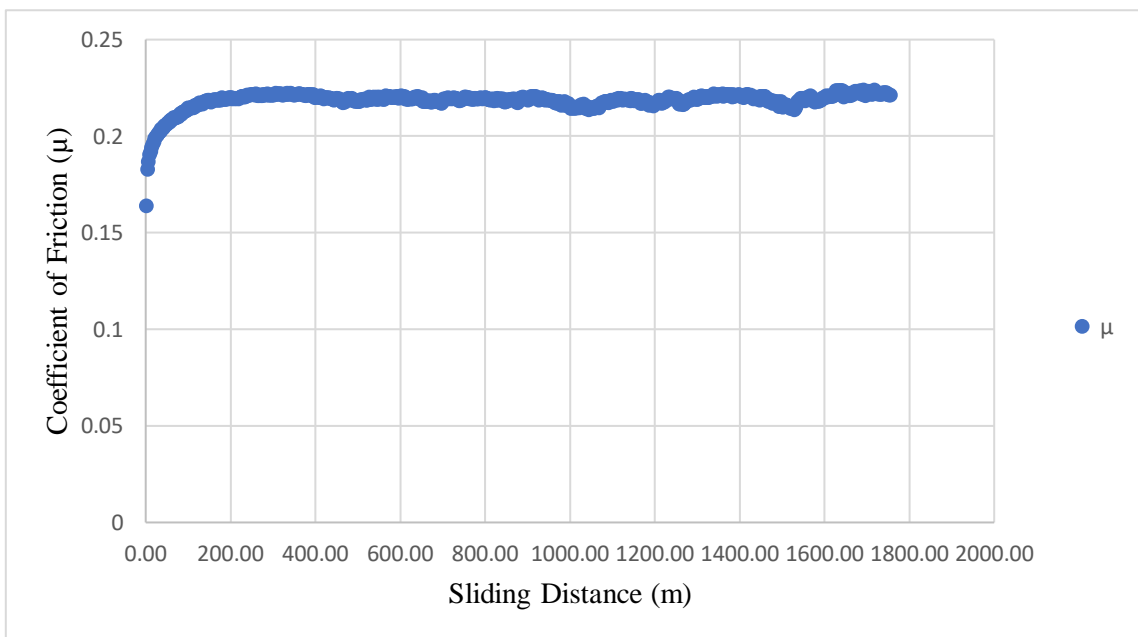


Figure A.12 MX.12. Coefficient of Friction Vs. Sliding Distance of sample 12

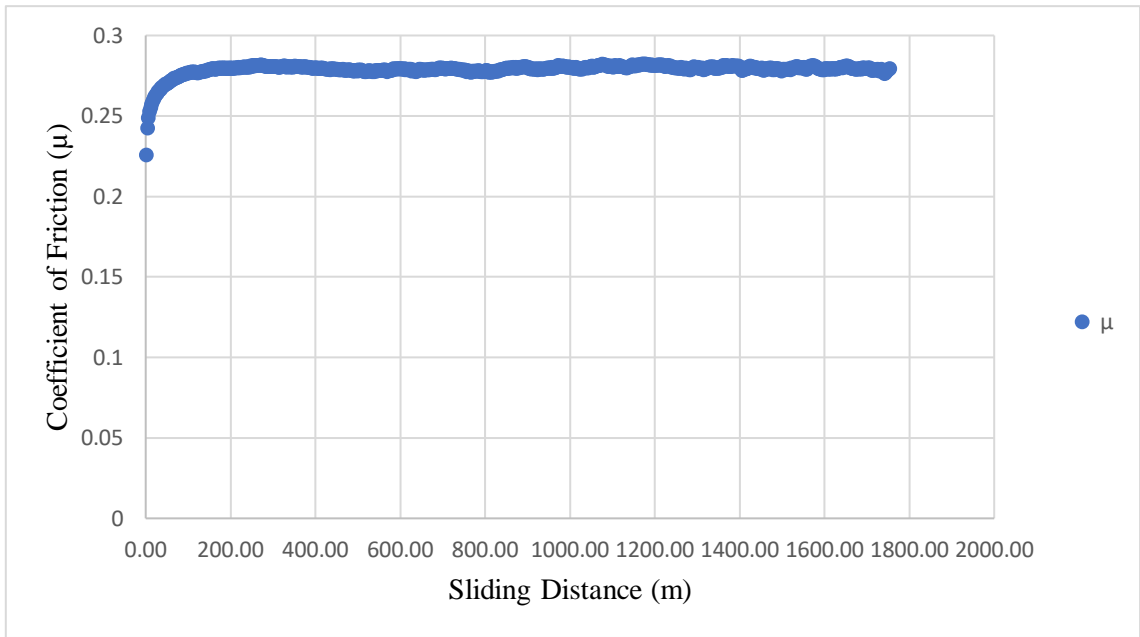


Figure A.13. MX.13. Coefficient of Friction Vs. Sliding Distance of sample 13