EFFECTS OF MECHANICAL ACTIVATION AND SODIUM HEXAMETAPHOSPHATE ON FORMATION OF MAGNESIUM ORTHOSILICATE CERAMIC POWDER

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A project report submitted in partial fulfilment of the
requirements for the award of Bachelor of Mechanical
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DECLARATION

I hereby declare that this project report is based on my original work except for citations and quotations which have been duly acknowledged. I also declare that it has not been previously and concurrently submitted for any other degree or award at UTAR or other institutions.

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APPROVAL FOR SUBMISSION

I certify that this project report entitled **"EFFECTS OF MECHANICAL ACTIVATION AND SODIUM HEXAMETAPHOSPHATE ON FORMATION OF MAGNESIUM ORTHOSILICATE CERAMIC POWDER"** was prepared by **KONG KOON YI** has met the required standard for submission in partial fulfilment of the requirements for the award of Bachelor of Mechanical Engineering with Honours at Universiti Tunku Abdul Rahman.

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ABSTRACT

Magnesium Orthosilicate, a mineral belonging to the olivine group, is composed of magnesium, silicon, and oxygen atoms. Its crystalline structure provided unique characteristics such as a high melting point, biocompatibility, and chemical stability. These properties made Magnesium Orthosilicate suitable for use as substitute parts of hard tissue. Consequently, the quest for efficient synthesis methods for forsterite became increasingly significant due to its potential applications in industries such as construction, electronics, and energy storage. Despite the potential applications of forsterite in industries such as construction and biomedicine, there remained a knowledge gap regarding the development of efficient synthesis methods that could reliably produce high-quality forsterite with precise control over its properties, such as particle size, morphology, and purity. Thus, this experiment explored various synthesis techniques, including high shear mixing and planetary ball milling, to determine optimal conditions for producing Magnesium Orthosilicate. Additionally, the experiment investigated the effects of the addition of Sodium Hexametaphosphate (SHMP) on viscosity to aid in the mass production of Magnesium Orthosilicate, aiming to enhance the scalability of the production. The most effective mechanical activation was found to be using high shear mixing with a mixing duration of 1 hour. Based on these findings, the optimum calcination conditions were determined to be at 1100°C with a holding time of 2 hours. The addition of SHMP allowed better dispersion and was capable of producing pure nanoparticle Magnesium Orthosilicate. As the weight percent (wt%) of SHMP addition increased, the particle size decreased, but this compromised the purity of the Magnesium Orthosilicate. The optimum water-to-solid ratio was found to be 6.871. These findings provided a more efficient way to synthesize Magnesium Orthosilicate, especially for mass production.

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LIST OF SYMBOLS / ABBREVIATIONS

MgO	*Periclase*
SiO_2	*Fumed Silica*
SHMP	*Sodium Hexametaphosphate*
$Mg_3Si_4O_{10}(OH)_2$	*Talc*
$MgSiO_3$	*Enstatite*
$MgCO_3$	*Magnesium Carbonate*
$(NO_3)_2H_2O$	*Magnesium Nitrate Hexahydrate*
$Si(OC_2H_5)_4$	*Tetraethyl Orthosilicate*
HSM	*High Shear Mixer*
XRD	*X-ray Diffraction*
SEM	*Scanning Electron Microscopy*
wt%	*Weight percent of SHMP*

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CHAPTER 1

INTRODUCTION

1.1 General Introduction

The search for biomaterials in human history can be traced back to ancient civilizations which utilizes their surrounding natural resources such as wood, stone, and animal parts for various medical and functional purposes. For instance, ancient Egyptians used linen and honey for wound dressings, while indigenous cultures utilized plant fibres and animal sinews for sutures and wound closures. Scientific progress and technological innovation have resulted in the creation of diverse synthetic and biocompatible materials, which have become essential components in contemporary medical therapies, as well as in the manufacturing of prosthetic devices and implants.

For implantation, the biomaterial that will be commonly used is bioceramics. This is because the bioceramics are capable to attain a balanced amalgamation of physical attributes that closely correspond to those of the replaced tissue, while inducing minimal toxic reactions within the host. The bioceramics has key features of low porosity, micrometric pore size and high mechanical strength which are highly suitable for permanent implants and coating of tissues (Niles, Coletti and Wilson, 1973).

In particular, the usage of silicon and magnesium containing bioceramic like magnesium orthosilicate, (Forsterite, Mg_2SiO_4) increased as the adhesive strength even though there was adhesion failure occurred. The adhesive strength reaches its peak of $57.9MPa \pm 7.0MPa$ and reduce to $41.1 \pm 4.5MPa$ after the adhesion failure. This resilience in adhesive strength highlights magnesium orthosilicate 's suitability as a body implant material, as it maintains its high adhesive strength even in the presence of environmental factors that may lead to adhesion failure (Mardarea, et al., 2002).

Magnesium Orthosilicate is composed of silicon and magnesium bioceramics. Silicon is essential to our bone formation and can easily obtained through dietary intake. In recent studies, the researchers use in vitro studies to show how does silicon helps in bone regeneration and increasing bone density (Arora and Arora, 2017). Magnesium is also an important mineral which help humans to enhance bone density and lower the bone fracturing risk. There was research that show that high magnesium intake may enhance the bone density mineral which helps to prevent osteoporosis (Groenendijk, et al., 2022).

There are various methods for synthesizing magnesium orthosilicate have been explored in previous studies. One approach involves the sol-gel method as demonstrated by Siyu Ni and colleagues (2005), wherein reagent-grade magnesium nitrate hexahydrate and silica are combined, undergoing gelation and milling steps, despite with a lengthy preparation duration (Ni, Siyu, Lee Chou, and Jiang Chang, 2005). There is another method proposed by F. Tavangarian et al. (2010), focuses on heat treatment using talc and periclase precursors that undergo mechanical activation through planetary ball milling, followed by annealing at 1200°C (F. Tavangarian, R. Emadi, 2010). A different avenue, highlighted by S. Ramesh et al. (2013), involves the sonification method to achieve uniform single-layer structures through ultrasonication, followed by ball milling to attain a multilayer lamellar morphology. The resulting mixture is subjected to sintering at 1500°C to eliminate periclase. These synthesis processes is highlighted by these varied techniques, revealing the intricate strategies attempted to produce magnesium orthosilicate which leads to ineffectiveness due to the prolonged production duration and temperature. Researchers opt for methods according to their distinct objectives, limited precursor availability, and the resources they have at their disposal.

Hence, the primary objective of this research is to synthesis magnesium orthosilicate powder in the easiest and most effective way. The main approach to achieve this objective will be designing another way of mixing the precursors through mechanical activation. This research will also focus on evaluating the suitable calcination temperature and time to produce the pure magnesium orthosilicate powder. This is because the presence of enstatite ($MgSiO_3$) will degrade the good mechanical properties of magnesium orthosilicate and should be eliminated from the final product. Beside the mechanical means to produce the powder, this report will also study the effect of the existence of SHMP on the formation of magnesium orthosilicate powder.

1.2 Importance of the Study

This study may provide insight into the development of effective mechanical activation methods which aids the efficiency of magnesium orthosilicate powders production. This applies the mixing or milling mechanism of mechanical activation techniques like ball milling and high shear mixing which promotes better dispersion and enable better rate of reaction during the heat treatment. The synthesis process should be able to produce pure magnesium orthosilicate without any impurities. The result of this present study may have significant impact on the production of high-quality magnesium orthosilicate-based materials, paving the way for their use in various applications such as ceramics, refractories, and advanced composites.

Next, this study holds a vital role in the importance of optimizing calcination parameters. This study specifically related to determination of the suitable temperature and duration for the novel mixing technique. The systematic examination of these factors will enable researchers applied the optimized calcination conditions to yield pure magnesium orthosilicate powders.

Besides that, this study also important for determine the effect of Sodium Hexametaphosphate (SHMP) in the rate of formation of magnesium orthosilicate. The magnesium orthosilicate formation mechanism will be investigated and characterized through various test like X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and Rheology studies. The results of this study will aid the mass production of pure forsterite powder by adding optimal amount of SHMP during the synthesis process.

1.3 Problem Statement

The problem statement of this research is to develop an efficient method for magnesium orthosilicate synthesis using mechanical activation with optimum calcination conditions including the holding temperature and duration. The reaction mechanism of the synthesis of magnesium orthosilicate will be more complicated as different mechanical activation preparation method has been proposed. The synthesis of magnesium orthosilicate process will be further tested in mass production using dispersing agent of SHMP. As the presence of impurities in magnesium orthosilicate will affect its material properties, the enstatite and periclase (MgO) will be produced and wanted to be avoided in the synthesis process.

The mechanical activation serves the purpose of increasing the contact surface area of the precursor by dispersion and de-aggregation of nano powder before undergo calcination. The mechanical activation methods used in powder mixing are ball milling, attrition milling and high energy milling. However, the main problem is the mechanical activation used by previous research which is planetary balling milling method requires 10 hours of ball milling to produce pure magnesium orthosilicate (Tavangarian, Emadi and Shafyei, 2009). This will significantly reduce the overall efficiency of the synthesis process. Hence, it is important to identify the suitable mechanical activation techniques to employ in the synthesis process of magnesium orthosilicate and yield good products.

In the pursuit of synthesizing magnesium orthosilicate through planetary ball milling, extensive research has been conducted. Notably, it has been observed that even after subjecting the magnesium orthosilicate precursor to prolonged planetary ball milling at elevated temperatures, the enstatite and periclase persist in the final product, even following sintering at $1400°C$. This resilient presence of MgO and enstatite implies that the traditional approach of extended ball milling alone may not be an efficient method for eliminating undesired phases. Furthermore, it has been identified that a substantial milling duration of 10 hours is required to effectively diminish the presence of these enstatite and MgO, highlighting the inefficiency and resource-intensive nature of the current mechanical activation technique (Tavangarian, Emadi and Shafyei, 2009).

Despite of these existing research, a significant gap persists in understanding whether the addition of SHMP will further improve the productivity of the synthesis process and a further extend to mass production. The previous studies capable to produce Magnesium Silicate Hydrate (MSH) in the water to solid ratio of 10 (Jia, et al.,2016). This research gap will highlight the need to explore the optimum amount of SHMP required to produce more magnesium orthosilicate without MgO and enstatite.

This research aims to bridge this gap by identifying suitable mechanical activation techniques and exploring the impact of SHMP addition on magnesium orthosilicate synthesis efficiency. The objectives of this project are thus defined by the need to develop a more effective synthesis method that ensures purity of the magnesium orthosilicate powder and enhances overall production efficiency. Addressing these objectives is vital for advancing magnesium orthosilicate synthesis towards mass production, facilitating its widespread utilization across various industries.

1.4 Aim and Objectives

The aim of this research to study the effects of mechanical activation and sodium hexametaphosphate (SHMP) on formation of magnesium orthosilicate ceramic powder. There are three main objectives and listed below:

(i) To develop an efficient method for synthesizing magnesium orthosilicate ceramic powder from periclase and fumed silica through mechanical activation.
(ii) To evaluate the suitable calcination temperature and duration to produce magnesium orthosilicate ceramic powder.
(iii) To characterize the formation of magnesium orthosilicate ceramic with and without the presence of Sodium Hexametaphosphate.

1.5 Scope and Limitation of the Study

The scope of study involves the investigation of and optimize the formation of magnesium orthosilicate through different mechanical activation mean, calcination temperature and duration, and existence of SHMP. The characterization of the magnesium orthosilicate will be examined through X-ray powder Diffraction (XRD), Scanning Electron Microscopy (SEM), and Rheology studies. Besides that, the parameters that affects the formation of magnesium orthosilicate are mechanical activation, annealing temperature and duration, presence of SHMP.

The limitation of the study is the operation of high shear mixer, the controlled parameter like the rotation per minute (RPM) was restricted up until 6000rpm as it cannot operate at a relatively high speed. The sequence of the precursor material was done by trial and error as the viscosity of the mixture is also unpredictable. The high viscosity of the mixture may increase the torque of the motor making it to burn out. Besides that, the usage of SHMP in magnesium orthosilicate production hasn't been done other researchers and the mixing mechanism of the precursor will significantly affect the purity of the magnesium orthosilicate powders.

1.6 Contribution of the Study

This study constitutes a substantial advancement in ceramic mass production by introducing innovative methodologies for synthesizing magnesium orthosilicate powder and facilitating its mass production.

Firstly, this study pioneers the development of a novel and highly effective synthesis method for magnesium orthosilicate powder. This new approach promises increased efficiency and efficacy compared to traditional methods, thus offering a significant breakthrough in the field of ceramic materials synthesis. Moreover, the incorporation of sodium hexametaphosphate (SHMP) into the production process marks a crucial milestone in enabling the mass production of magnesium orthosilicate powders.

Moreover, this study explores the optimization of the calcination process for magnesium orthosilicate powder, a pivotal stage in ceramic manufacturing. Through the experimentation and analysis, the researchers like F. Tavangarian and R. Emadi identify parameters such as temperature and duration influence the properties of the ceramic material. By precisely adjusting these variables, the study achieves notable improvements in the thermal stability of the magnesium orthosilicate ceramics produced. This optimized calcination process not only ensures the production of high-performance ceramic products but also contributes to reduce energy consumption and waste generation during manufacturing.

By using SHMP, the study enhances the dispersion of particles and the homogenity of the mixture, thereby streamlining the production process and ensuring the consistent manufacture of high quality magnesium orthosilicate powders at scale. This contribution highlights the importance of this study in advancing ceramic mass production capabilities and opens up new avenues for the widespread utilization of magnesium orthosilicate in various industrial applications.

1.7 Outline of the Report

In Chapter 1, the introduction was written to explain the application of magnesium orthosilicate in real life due to its mechanical properties. The importance of study and problem statement highlights the significance of previous study and the changes of this study can bring to overcome the difficulties faced in the synthesis of magnesium orthosilicate. The aim and objective are listed, where the main aim is to study the effects of mechanical activation and Sodium Hexametaphosphate on formation of magnesium orthosilicate ceramic powder. Furthermore, the scope and limitation of the study covers the importance and the drawbacks in this study. Lastly, the contribution of this study states how the industry and society will be benefited.

In Chapter 2, the literature review was done based on the research journals and articles on the material, methods ad conditions. It highlights the challenges encountered and discoveries made, serving as a reference for the current study's enhancement or realization. The discussion primarily revolves around mechanical activation in ceramic processing, particularly emphasizing planetary processing and high shear mixing. It examines various precursors utilized for magnesium orthosilicate formation to identify the most suitable option. Furthermore, it explores the concept of calcination to optimize the calcination conditions for synthesizing magnesium orthosilicate effectively. Moreover, the chapter delves into characterizing magnesium orthosilicate to align with the expected outcomes of the experiment design. Additionally, it investigates the role of SHMP in magnesium orthosilicate formation, as it facilitates the mass production of magnesium orthosilicate.

Chapter 3 is regarding the methodology of this study. The general steps for synthesis of magnesium orthosilicate will be introduced. The apparatus and material required for the synthesis of magnesium orthosilicate were specified. The experiment will be separated into three different parts with different approach to achieve respective objectives. The development of an effective mechanical activation method will mainly be based on two methods which are High shear mixing and planetary ball mill. Next, the optimization of calcination temperature and duration will be described. Lastly, the effects of SHMP in the formation of magnesium orthosilicate will be studied with three approaches which are investigating hydration of MgO, effects of different wt% of SHMP on magnesium orthosilicate formation and optimization of SHMP addition. The way of usage of analytical instruments like XRD, SEM and rheometer will be discussed.

In Chapter 4 is mainly about the results and discussion based on the designed experiment in Chapter 3. The development of effective mechanical activation will decide which methods will be practical with minimal duration and does not consist of MgO and enstatite. Besides that, the optimization of calcination temperature and duration will decide based on the XRD results. Moreover, the effects of hydration evolution of MgO are discussed based on the XRD analysis of the precalcinated powder. The effects of different wt% SHMP addition will be discussed through the XRD, SEM and rheology results. Lastly, the optimization of SHMP addition in magnesium orthosilicate for mass production is done based on the previous result obtained whereby increasing the water to solid ratio.

Lastly, this part of the report will conclude the findings of the experiments and determine whether the aim and objective are achieved. There will be recommendations suggested based on the findings of the experiments which allows for potential improvements or further research directions. Additionally, any limitations encountered during the experiments will be discussed, providing insights into areas that may require additional attention or refinement in future studies.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Magnesium Orthosilicate is also known as forsterite ceramics is originate from magnesium rich member of the olivine group solid solution series. These chemicals are abundantly found within the rocks in the Earth crust which is called dunite rocks. These olivine minerals exhibit isomorphism, which gives vary in mechanical properties although sharing the same crystal structure. Magnesium Orthosilicate has high coefficient of thermal expansion which indicates that it can expand significantly when heated and contracts when cooled. This is due to the complex crystal structure of the magnesium orthosilicate which bonded by the silicon-oxygen and magnesium-oxygen tetrahedra (Gillet, et al.,1991).

Besides that, the magnesium orthosilicate also shows good biocompatibility which sees the magnesium orthosilicate to become important bio ceramic material used for body implantation. Magnesium is a crucial element for energy metabolism and enzyme activation. Silicon plays a key role in increasing simultaneously with calcium at lower calcium concentrations but become undetectable as compositions approach hydroxyapatite, indicating a potential association between silicon and calcium during the initial phases of calcification (Carlisle, 1970).

On the other hand, the magnesium orthosilicate also shows good mechanical properties. The hardness of magnesium orthosilicate is highly dependent on the annealing and sintering temperature. The relationship between the sintering temperature and hardness of the magnesium orthosilicate is directly proportional. The Vickers hardness varies from $2.3GPa$ at 1200^oC to $7.7\ GPa$ at 1500^oC. The results also shows that the magnesium orthosilicate samples which were annealed before sintering had poor Vickers hardness and fracture toughness while having a superior Young's modulus. (Ramesh, et al., 2013)

Lastly, the magnesium orthosilicate has high melting point is about $2099K$. This high melting point enables it to become refractory material for furnace lining to withstand the extreme heat. The melting point of magnesium orthosilicate is affected by chemical composition, impurities, and heating rate (Durham and Goetze, 1977) These isomorphism, thermal expansion characteristics, biocompatibility, mechanical versatility, and high melting point properties of magnesium orthosilicate allows it to contribute to its suitability for various applications, particularly in the field of biomedicine.

2.2 Mechanical activation in ceramic process

Mechanical activation is a process that uses mechanical means like grinding, milling, and other mechanical forces to make structural changes of the materials, is pivotal in material science for inducing structural alterations. This technique is often utilized to reduce particle dimensions, amplify contact surface areas, and thereby expedite reaction rates. In ceramic processing, mechanical activation plays a crucial role in various stages of material preparation and fabrication. Different types of mechanical activation involve different intensity of the mechanical forces. Hence, it is important to understand the application in ceramic processing to choose the suitable efficient mechanical activation methods.

The mechanical activation will reduce the particle size for better reactivity. The contact surface area of the particles will significantly increase and increase reactivity. For example, the mixture of ilmenite and graphite uses ball milling before undergoing annealing. Based on the XRD results, it shows that Titanium carbide is formed without Titanium suboxides after heating to $1200\ ^{o}C$. The temperature is much lower than the sample without milling. This indicates that the sample after ball milling will reduce the crystalline structure of the precursors and increase the rate of reaction (Chen, Y., et al, 1997).

Besides that, the mechanical activation can bring changes to the material structural behaviours such as phase transition and crystallinity. When mechanical activation applied over a prolonged duration, can induce a fatigue failure mechanism within the material, leading to the fragmentation of fragile agglomerates. This consequential fragmentation process serves to refine the material's internal structure, ultimately resulting in the establishment of stable particle sizes, which contributes to the overall structural stability and uniformity of the material (Živojinović, et al., 2017).

Mechanical activation is used in synthesizing ceramic-based composites causing the reduction of particle size, increases surface area, and drives structural alterations for enhanced reactivity and material performance. By elevating the energy level of the system, mechanical activation facilitates more frequent and energetic collisions between particles, leading to increased conversion of reactants into products. The mechanical activation alters the thermal behaviour of starting powders but also affects reaction thermodynamics and kinetics, making it an indispensable tool for enhancing the efficiency and effectiveness of various chemical processes involving powdered reactants (Farhanchi, et al., 2015).

2.2.1 Ball milling

Planetary ball milling is a high energy ball milling where the container filled grinding balls and mixture will be rotated continuously. The grinding balls are made ceramics which are chemically inert and have different size for specific purpose. The purpose of using different size of grinding balls is to manipulate the final particle size of the material being processed. The small grinding balls is capable to reduce the size of large particles into finer size. The combination of both large and small grinding balls, the ball milling process will be very efficient as the small spaces will be occupied by the small grinding balls. The efficiency of ball milling using homogeneous size of the ball mill will be lesser (AmanNejad and Barani, 2020).

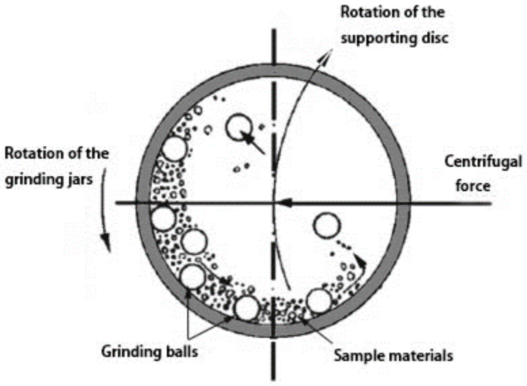


Figure 2.1: Demonstration of Planetary Ball Milling (Baheti, Abbasi and Militky, 2012).

The parameter that can be monitored to produce desired results through planetary ball milling is milling duration, rotational speed of the roller machine and the choice of grinding media. Longer milling duration will bring produce finer product. For example, the carbon nanotubes undergo prolonged ball milling of 200 hours, the results shows that the ball milling is equally effective as high impact methods but offers certain benefits. It allows more precise tuning of the nanotubes properties by changing the milling duration. This level of control enables manufacturers to tailor made the nanotubes according to the material properties and application (Kukovecz, et al., 2005).

In addition, the factor of rotational speed will be crucial for achieving homogeneity. The high rotational speed will increase the collision frequency of the balls which leads to effective milling of the powder. It is important to increase the rotation to revolution speed ratio as it will result more energy being transferred during ball milling. This energy will be vital for material deformation and structural changes. The previous research uses talc to study the effect of rotational speed in ball milling, the results suggested that the specific impact energy of balls rise as the rotation-to-revolution speed ratio increases. However, it declines once the critical speed ratio is reached, primarily because of the introduction of rolling motion (Hiroshi, et al., 2001).

Furthermore, the choice of grinding media is very important as the media must be chemically inert and has minimal difficulty to extract the final product. This research suggested that the use of grinding media can enhance the generation of fine particles, yet the production efficiency per unit of energy expenditure remains constant. A crucial factor in this context is the critical pulp viscosity, which plays a pivotal role in influencing the efficiency of the ball milling process. This critical viscosity pertains to a point where the milling balls are unable to disengage from the mill wall during rotation. If the grinding media can reduce the critical pulp viscosity, this allows the mill to draw more energy and produce finer powder (D.W. Fuerstenau, K.S. Venkataraman, B.V. Velamakanni, 1984).

Planetary milling plays a crucial role in magnesium orthosilicate synthesis by refining crystalline size and enhancing lattice strain. It enables achieving a uniform grain size distribution, essential for consistent material properties. Prolonged milling will fragment larger particles, refining the particle size distribution, and generates lattice defects within magnesium orthosilicate's structure. In contrast, the XRD peaks of magnesium orthosilicate will eventually decrease in intensity due to refinement of crystalline size. The smaller crystalline sizes will increase the specific surface area and enhancing reactivity and densification during processing. Moreover, the presence of lattice strain can influence the mechanical, thermal, and electrical properties of magnesium orthosilicate, making it suitable for a wide range of applications (Bafrooei, Ebadzadeh and Majidian, 2014).

In conclusion, the planetary ball milling proves to be a good technique for finely grinding materials. It operates by rotating a container filled with grinding balls, which effectively break down the material into smaller particles. Crucial factors such as milling duration, rotational speed, and the type of grinding media utilized all significantly impact the process efficiency. The longer milling duration will produce finer particles. By increasing the rotational speeds of planetary ball mill, this enhance grinding efficiency through more collisions, and selecting appropriate grinding balls is vital for optimal results. For materials like magnesium orthosilicate, planetary milling serves to reduce particle size and enhance reactivity, thus expanding its utility across various applications.

2.2.2 High shear mixing

High shear mixer is composed of a high-speed rotor with a narrow gap a stator. The speed of the rotors varies depending on their application. It was normally used for deagglomeration and applied in food production, healthcare, agriculture and biochemical. The local energy dissipation and shear rate are generated to disperse the bulky powder into finer powder in an efficient way (Yudong, et al., 2022).

The high shear mixer work on the mechanical force applied to the materials being mixed. The powder needs to be added with the grinding media before operation to provide effective energy transfer for better dispersion. The high shear mixer is composed of long rotor shaft which connect the high-speed rotating blade and a stator covers around the blade. When the rotor starts to rotate, the high-speed rotating blades tends to create turbulence within the mixture and forcing the mixture through the small gaps of the stator. This creates shearing action with the mixture which breakdown the particles into smaller particles and simultaneously achieving mixing and dispersion.

There are different types of high shear mixer exist in the market for different industries and purpose. Batch high shear mixer is commonly used for medium scale mixing application especially in food processing industries. The production of mayonnaise uses the batch high shear mixer emulsify the rape seed oil in aqueous sugar solution homogeneously.

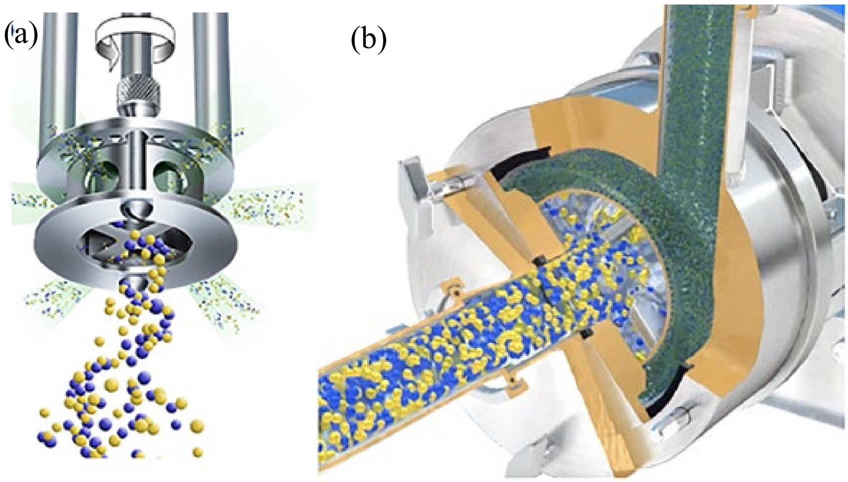


Figure 2.2: (a)Inline High Shear Mixer, (b) Batch High Shear Mixer.

The inline high shear mixer is designed for continuous process as the material will be transferred into the mixing chamber through the rotor and stator assembly (Jintao, et al., 2013).

The high shear mixer is used for dispersing one of the precursor silica nano particles. The hydrophobic and hydrophilic silica nano powders are dispersed. Although they exhibit similar size distribution and morphology, the level of deagglomeration is different. The hydrophobic silica displays faster de-agglomeration, with nanoaggregates forming earlier and requiring lower energy dissipation rates compared to hydrophilic silica. The critical energy density for effective de-agglomeration varies between the two, with hydrophilic silica requires higher energy densities for dispersion. These findings provide valuable insights into optimizing processing parameters for silica nano-powders, crucial for this experiment setup (Ding, Orwa and Pacek, 2009).

The parameters that affect the efficiency of the high shear mixer are number of rotor teeth, distance between rotor and stator, rotor teeth height and more. Previous study shows that the higher number of rotor teeth will increase the generation of fine agglomerates (Liu, et. al., 2021). This indicates that the contact area for rotor teeth deagglomerate the powder. The high rotational speed of the rotor results higher collision frequency and shear effect between the mixture and the slots in the stator.

Besides that, the energy density is also one of the factors that will influence the mixing efficiency. This study uses hydrophilic silicon dioxide powder to test the effect of energy density in a high shear mixer. The results reveal that silicon dioxide will aggregate in two stages. The large aggregates will be broken down into smaller sizes when energy density increases. At high energy density, the breaking process shifts to the erosion of primary aggregates from the surface of secondary aggregates. When the energy density is adequate, the secondary aggregate disappears, leaving only tiny primary aggregates in the mixture. This study highlights the significant impact of energy density on the aggregation behaviour and mixing efficiency of silicon dioxide powder, offering valuable insights for optimizing industrial processes in ceramic material production and related applications (Pacek, Ding and Utomo, 2007).

Overall, the high shear mixers play a crucial role in various industries such as food production, healthcare, agriculture, and biochemical processes, offering efficient dispersion and mixing capabilities through mechanical force and shear action. There are different types of high shear mixers, such as batch and inline models, are tailored to suit specific mixing requirements. The parameters like rotor design and energy density play crucial roles in determining the efficiency of the mixing process. Moreover, recent studies, particularly on the dispersion of silica nano powders, have highlighted the importance of optimizing processing parameters, including energy density, to achieve effective de-agglomeration. By understanding the relationship between energy density and aggregation behaviour, as demonstrated in experiments with hydrophilic silicon dioxide powder, provides valuable insights for enhancing industrial processes in ceramic material production which includes magnesium orthosilicate.

2.3 Synthesis of Magnesium Orthosilicate Ceramic powder

In the synthesis of magnesium orthosilicate, the traditional methods used are heating of talc ($Mg_3Si_4O_{10}(OH)_2$) and Magnesium Carbonate ($MgCO_3$), co-precipitation method, molten salt approach, and more. As the magnesium orthosilicate formation process is a two-phase process, there will be enstatite, $MgSiO_3$ and Magnesium oxide, MgO produced during the synthesis of magnesium orthosilicate. It is important for the synthesis process to be capable to produce pure magnesium orthosilicate without MgO and enstatite as it will reduce the quality of the magnesium orthosilicate. Different approach for the synthesizing of magnesium orthosilicate has their respective difficulty and advantages. The difficulty of those processes is focusing on the extraction of the pure single-phase magnesium orthosilicate. Hence, it is important to utilize the production process to increase the efficiency of pure magnesium orthosilicate production.

First and foremost, the heating of magnesium carbonate and talc is one of the common novel methods to produce magnesium orthosilicate. The precursors material will be mixed according to the molar ratio of 2:1 which correspond to the theoretical value of pure magnesium orthosilicate. The mixture will be mechanically activated by planetary ball milling under ambient condition. The ball weight to mixture ratio will be 10:1 and run under 500 rpm speed. The parameters that can affect the ball milling process are revolution speed, milling time, filling ratio of milling balls of different size and ball to powder ratio. These parameters are interrelated and important to increase the surface area of the precursors. After the ball milling process, the heat treatment will be carried out under ambient pressure and elevated temperature with gradual heating rate and extended holding time. The prolonged ball milling process does not guarantee as MgO will appear despite $1500\ ^{o}\ C$ of sintering temperature (Tavangarian, Emadi, and Shafyei, 2009).

Next, the co-precipitation method involves precursors of Magnesium Nitrate Hexahydrate, $(NO_3)_2H_2O$ and Tetraethyl Orthosilicate, $Si(OC_2H_5)_4$ which are mixed and dissolved in solution composed of 40ml ethanol and 3ml of nitric acid. The solution will be stirred vigorously for 30 minutes and dipped into 120ml of ammonium hydroxide for continuous stirring. The solution will be left out for certain duration to precipitate and form magnesium orthosilicate.

This method is capable to produce single phase magnesium orthosilicate powder. The extraction of magnesium orthosilicate from other co-precipitated substances and complicate the industrial process (Zampiva, et al.,2017).

Besides that, the molten salt approach is the use of molten salts like sodium chloride, NaCl add into the mixing of MgO and silica (SiO_2) according to the desired molar ratio. The mixture will be grinded for 30 minutes and undergo heat treatment for $700 - 1100^oC.$ The mixture will be rinsed with deionized water until no chlorine ions can be detected. This becomes the main obstacle need to be overcome to obtain pure magnesium orthosilicate. This method allows the researchers to produce magnesium orthosilicate of desired crystal size by undergoing heat treatment in the range of $700 - 1100^oC$ (Wang, Z.F., et al, 2016).

On the other hand, there is also research uses Talc and MgO as precursor to synthesis magnesium orthosilicate. The combination is made using a 5:1 molar ratio of Talc to MgO molar ratio of 5:1. The mixture will be subjected to planetary ball mill under ambient conditions. The milling media is using ceramic grinding ball with ball to powder weight ratio of 10:1. The rotational speed will be set at 500 rpm. By undergo 5 hours of mechanical activation and calcinated at 1200^oC , pure nanocrystalline magnesium orthosilicate is obtained (Emadi, et al., 2011).

In summary, there are several ways to synthesizing magnesium orthosilicate whereby each methods had its own limitation and advantages. The tradiational methods include heating talc and magnesium carbonate, co-precipitation, the molten salt procedure and talc and MgO as precursors, provide options for manufacturing pure nanocrystalline magnesium orthosilicate by planetary ball milling and subsequent heat treatment. To maximize magnesium orthosilicate manufacturing efficiency, each process must take into account characteristics such as milling conditions, precursor ratios, and heat treatment temperatures. Despite the hurdles, advances in synthesis processes continue to expand the potential applications of magnesium orthosilicate in a variety of sectors.

2.3.1 Precursor

There is different precursor to be used in the previous studies as mentioned in section 2.3. The precursors are $MgCO_3$ and talc, magnesium nitrate hexahydrate and tetraethyl orthosilicate, MgO and silica, and talc plus MgO The ratio of each precursor needs to be calculated using derivation of molar ratio to molar mass.

The purity of precursor materials directly impacts the impurity content of the final magnesium orthosilicate product, making high-purity precursors desirable for achieving the desired material properties and minimizing unwanted secondary phases. Therefore, careful selection and processing of silica and periclase precursors are essential for tailoring magnesium orthosilicate properties to meet specific application requirements. Research suggests that the use high purity precursor enable to synthesis pure magnesium orthosilicate by ordinary sintering without additional pressure (Satoru, et al., 2006).

2.3.2 Calcination duration and temperature

Calcination is a process of heating a substance at elevated temperature in the absence of air or oxygen. This brings alterations in the physical and chemical characteristics of a material take place with the aim of removing volatile substance such as ethanol, water and more This method is commonly used in cement production and preparation of metal oxide. The release of volatile components will minimize the internal shrinkage which aids the reduction of internal stress eventually causing cracks when used for 3D printing.

According to previous study, the calcination temperature ranges between $1000^o\ C$ and $1200^o\ C$ with the heating rate of $10\ {}^oC/min$ with short heating time and long holding time. Annealing will cause the weight loss during the magnesium orthosilicate formation process. The weight loss can be divided into three stages and different composition of breakdown of substance. The weight loss of the process is investigated through the thermal analysis. When the temperature reaches to $300^o\ C$, there will be the first weight loss caused by dehydration occurred. When the temperature ranges between $500^o\ C - 1200^o\ C$, the second weight loss occurs due to the crystallization of periclase.

Lastly for the temperature more than $1200^o\ C$, the final weight loss will be caused by the crystallization of magnesium orthosilicate (Tavangarian, Emadi, and Shafyei, 2009). Hence, it is important to study the reaction mechanism and material property.

The study of quicklime reactivity and its relationship with calcination temperature has significant consequences for ceramic manufacture. The findings highlight the significance of quicklime features such as crystal size, dispersion, and compactness in determining ceramic qualities. A more reactive quicklime, characterized by smaller crystal sizes and greater specific surface areas, can have a substantial influence on ceramic body formation, glaze preparation, and calcination processes. Furthermore, the inverse link between calcination temperature and quicklime reactivity emphasizes the need of carefully selecting fire conditions in ceramic kilns to maximize product quality. Understanding and harnessing these linkages allows ceramic producers to improve the efficiency and quality of their manufacturing processes, resulting in stronger, more durable, and more appealing ceramic products (Antoni, et al., 2001).

Additionally, calcination allows manufacturers precise control over the microstructure of ceramics. By adjusting calcination duration and temperature, they can tailor the material's properties to meet diverse application requirements such as hardness, thermal conductivity, and more, making it a versatile and indispensable process in ceramic production. For instance, the crystallization control of the grain boundary phase improves the mechanical property of silicon carbide ceramics (Cheong, et al., 2002).

Lastly, in the calcination process for hydroxyapatite (HA) nanoparticles, microscopic crystals form and proliferate throughout the material, rather than simply on the surface. Due to the abundance of HA molecules within the material, new crystals may quickly form. However, because molecules travel slowly in solid materials, the crystals that form are smaller but more abundant. Understanding how nucleation and growth occur during calcination is critical for creating ceramic materials. It aids in controlling the size and quantity of crystals in the ceramic, influencing qualities like as strength and durability. So, by refining the calcination process using these insights, we may produce superior magnesium orthosilicate ceramics for a variety of uses (Scalera, et al., 2013).

2.4 Effects of addition of Sodium Hexametaphosphate in Magnesium Orthosilicate formation

Sodium Hexametaphosphate $(NaPO_3)_6$, (SHMP) is a chemical compound that is widely used in ceramic processing as dispersing agent. The SHMP is employed as dispersant to improve the suspension properties of the ceramic slurries. It prevents prevent agglomeration and promotes dispersion of particles in the liquid medium. This enhances the flow and rheological behaviour of the ceramic suspension. This is particularly important for achieving homogeneous coatings or layers during processes such as magnesium orthosilicate synthesis.

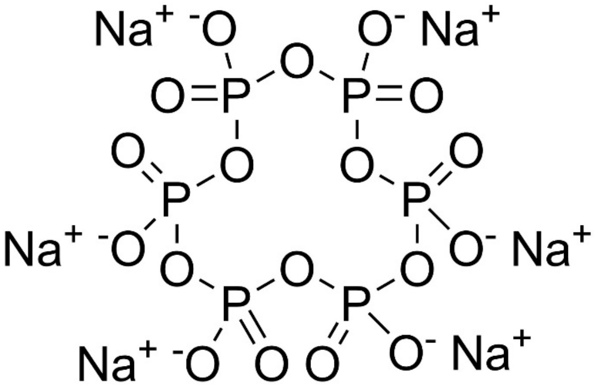


Figure 2.3: Chemical Structure of Sodium Hexametaphosphate (SHMP).

In response to previous limitations in magnesium orthosilicate production, researchers have introduced the concept of incorporating sodium hexametaphosphate (SHMP) into cement production processes to enable mass production of magnesium orthosilicate. This innovation aims to enhance both the rheological and mechanical properties of the magnesium oxide (MgO) and fumed silica paste. Previous studies have investigated the hydration of MgO by immersing it in distilled water with and without SHMP. They found out that MgO hydration accelerates the reaction kinetics between magnesium oxide and silica. Initially, when MgO reacts with water, it forms magnesium hydroxide $(Mg(OH)_2)$, which can then react with silica to form magnesium silicate phases, crucial for magnesium orthosilicate formation. Consequently, this inhibition leads to increased dissolution of MgO and a higher concentration of Mg^{2+} ions in the solution. Moreover, the MgO particles exhibit a tendency to agglomerate more readily and consist of finer primary particles in the presence of SHMP. These findings highlight the potential of SHMP as a key factor in influencing the hydration behaviour and particle morphology of MgO, paving the way for improved processes in both cement and ceramic production. This aligns with the observed inhibition of $Mg(OH)_2$ formation, the significant increase in pH, the elevated concentration of Mg^{2+} ions in solution, and the alteration in particle morphology observed when SHMP is present during MgO hydration (Yuan, et al. 2016).

Besides that, there are research that shows SHMP will be beneficial to the immobilization of Magnesium rich Magnox sludge (MSH). This work allows MSH to be used as encapsulant for both low and high-water content without producing bleed water. The X-ray diffraction analysis revealed the formation of hydration products, primarily brucite and silica fume, consumed to varying degrees with phosphate addition, resulting in the production of M-S-H. Poorly crystalline M-S-H was evident in the diffractograms, with excess silica fume identified. Interestingly, the formation of M-S-H was delayed with higher levels of phosphate addition (Sam, et. al, 2015). This finding will be helpful for the production of olivine group materials including magnesium orthosilicate.

In conclusion, sodium hexametaphosphate (SHMP) emerges as a versatile compound with significant implications across various industries, particularly in ceramic and cement production as well as in waste immobilization processes. Its role as a dispersing agent enhances suspension properties, facilitating homogeneous coatings and layers crucial for processes like magnesium orthosilicate synthesis and the immobilization of magnesium-rich Magnox sludge (MSH). Moreover, the SHMP inhibits the $Mg(OH)_2$ formation during MgO hydration leads to increased dissolution of MgO, elevated Mg^{2+} ion concentration, and alterations in particle morphology, promising improvements in both cement and ceramic production processes. Additionally, the incorporation of SHMP in MSH immobilization shows potential benefits, including the formation of poorly crystalline M-S-H phases, suggesting its utility in mitigating environmental concerns associated with waste disposal. These findings highlight the SHMP's role in enhancing material properties and process efficiency, highlighting its significance in various industrial applications.

2.5 Characterization of Magnesium Orthosilicate

The characterization of magnesium orthosilicate powder usually needs to be done by scientific analytical tools like XRD, SEM, and rheology. This is because the microstructure and the unique properties of the magnesium orthosilicate powder makes the research works cannot be observed through observations.

2.5.1 X-Ray Diffraction (XRD) analysis

X-ray Diffraction (XRD) is a powerful analytical technique with a primary purpose of determining the crystal structure and atomic arrangement within crystalline materials. Previous research uses XRD to identify the presence of MgO and enstatite and phase transformation after calcination process. The XRD results show that the first magnesium orthosilicate peak appears when the calcination temperature reaches $750\ ^{o}C$. As the temperature increase until reaches $1000°C$, there was a noticeable increase in the intensity of the magnesium orthosilicate peaks. This phenomenon suggests the progressive alleviation of internal strain within the material and the enlargement of crystallite size. Notably, no additional phases were detected in the XRD pattern. Within the $750°C$ to $1000°C$ range. This study indicates a series of phase changes wherein magnesium oxide first originates within the structure. Then, it undergoes a reaction at the surface of SiO_2, resulting in the formation of enstatite. Ultimately, MgO permeates through the enstatite layer, culminating in the creation of magnesium orthosilicate. As a reference of purity of the magnesium orthosilicate, the MgO and $MgSiO_3$ will have significant peaks at 29.9^{o} and 37.4^{o} respectively (Tavangarian, Emadi, and Shafyei, 2009).

2.5.2 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) serves a crucial purpose in materials science, biology, geology, and numerous other fields by delivering high-resolution imaging of material surfaces. It shows high resolution image of the samples’ surface morphology, elemental composition, and microstructure

(Seyforth, 2015). The researchers have utilized SEM to study the morphology of the before and after calcinated powders by observing the powder size. The samples will be coated with a conductive material such as platinum to preserve the original surface morphology. It provides a conductive surface which allows the electron beam to interact with reduced charging effects in order to improve image resolution (Cheney, 2007). However, there is insufficient knowledge and research that suggest the effect of SHMP on the Magnesium Orthosilicate powder size. Therefore, it is important to conduct further investigation to determine whether the SHMP addition will cause any effect on the powder size when the wt.% is different.

2.5.3 Rheology viscosity test

Rheology studies is primarily helping the investigation of the flow and deformation behaviour of material under various conditions, typically under stress or strain. The rheological properties such as viscosity, elasticity, and viscoelasticity are characterized to understand how material respond to external forces. In the context of magnesium orthosilicate synthesis with SHMP, the understanding rheological behaviour is crucial. As illustrated in Figure 2.5 below, diluted ceramic suspensions exhibit Newtonian behaviour, meaning their viscosity remains constant regardless of the applied shear rate. However, as the concentration of the solution increases, the ceramic suspension gradually exhibits shear thinning behaviour, where viscosity decreases with increasing shear rate. At higher concentrations, the suspension may undergo shear thickening, wherein viscosity increases with shear rate (Camargo, et al., 2021).

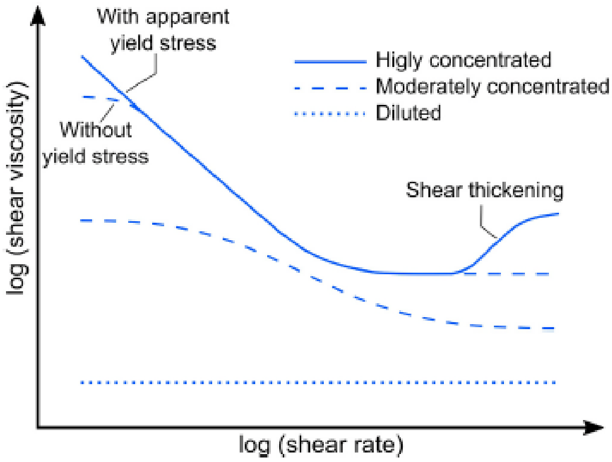


Figure 2.4: Graph of Viscosity against Shear rate (Camargo, et al., 2021).

These rheological properties are vital for optimizing the processing conditions during magnesium orthosilicate synthesis with SHMP to ensure proper flow and deformation behaviour of the ceramic suspension, ultimately influencing the quality and properties of the synthesized material.

2.6 Summary

Magnesium Orthosilicate, also known as magnesium orthosilicate, is a ceramic material that originates from magnesium-rich members of the olivine mineral group. It is abundantly found in dunite rocks within the Earth's crust. magnesium orthosilicate exhibits isomorphism, resulting in varying mechanical properties while sharing the same crystal structure. It possesses a high coefficient of thermal expansion, making it capable of significant expansion when heated and contraction when cooled due to its complex crystal structure. Notably, magnesium orthosilicate is biocompatible and has excellent mechanical properties, including high hardness and fracture toughness. Its high melting point also makes it suitable for use as a refractory material in furnace linings.

The concept of mechanical activation is introduced, with a primary focus on two practical methods: high shear mixing and planetary ball milling. Regarding the synthesis of magnesium orthosilicate, various methods are discussed, including heating talc and magnesium carbonate, co-precipitation, and the molten salt approach. However, each method has its limitations, such as milling duration, high calcination duration, difficulty in obtaining single-phase pure magnesium orthosilicate, and limited scalability.

The effects of SHMP in magnesium orthosilicate synthesis are examined, with a particular emphasis on the hydration of MgO with increasing wt% of SHMP. Additionally, the impact of SHMP on magnesium orthosilicate properties before and after calcination over a 60-day period is investigated. These findings are crucial for enhancing process efficiency and obtaining pure magnesium orthosilicate.

Lastly, the characterization of magnesium orthosilicate is conducted using various experimental setups, including rheology studies, XRD analysis, and SEM imaging. These characterizations provide valuable insights into the properties and behaviour of magnesium orthosilicate synthesized under different conditions.

CHAPTER 3

METHODOLOGY AND WORK PLAN

3.1 Introduction

The experiment will be conducted in the university laboratory as the formation of magnesium orthosilicate involves high calcination temperature. The analytical works will be like X-ray Diffraction (XRD), Scanning electron microscope (SEM), and rheology will be carried out using the existing analytical machines in the university.

3.1.1 General Steps for Synthesis of Magnesium Orthosilicate ceramic powder

The experiment will be divided into two parts which mainly consist of synthesis of magnesium orthosilicate and characterization of magnesium orthosilicate formation. The addition of Sodium Hexametaphosphate (SHMP) was calculated according to the requirement of the experiment. The overall process will be listed in figure below:

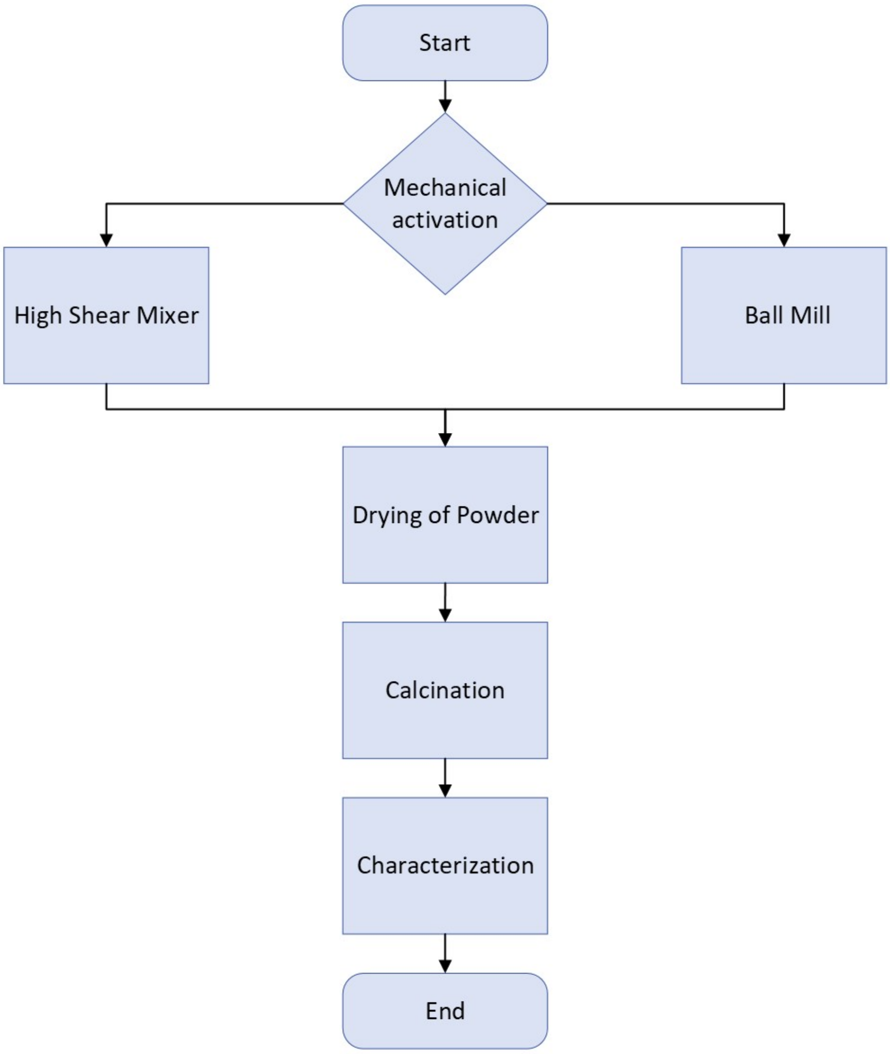


Figure 3.1: Overall Process of Magnesium Orthosilicate synthesis.

The synthesis of magnesium orthosilicate was started by preparation of precursor powder. The precursor of this experiments are Periclase (MgO) and Fumed Silica. The precursors were prepared according to the molar ratio of 2:1 as shown below:

$$2MgO + SiO_2 \rightarrow Mg_2SiO_4 \qquad (3.1)$$

The sequence of adding the precursor is important for the synthesis process as it will affect the viscosity of the mixture and mixing effect. The mixture went for different mixing method either conventional Ball Milling or High Shear Mixing depending on the requirement of the experiments. Then, the mixture was put inside the oven for drying for about 1-2 days. The dried powder was pounded and undergo calcination process according to the specific calcination conditions of the experiment setup. Lastly, the calcinated powder was characterized through analytical instruments.

3.1.2 Experiment Apparatus

Laboratory Oven

A laboratory oven was used for high-volume thermal convection applications. These ovens were designed to provide precise and controlled temperature environments within a sealed chamber to meet the specific needs of scientific experiments and processes. The oven was used for the drying process of the mixture before calcination. The temperature of the oven was set at 90^oC and the duration of drying took at least 24 hours.

LT Furnace

A chamber furnace is a type of industrial heating device or oven that is designed for high-temperature applications, such as heat treatment, annealing, sintering, and various industrial processes that require controlled heating environments. In this experiment, the furnace was used for calcination of the milled mixture. The parameters manipulated are calcination temperature and holding time. In general, the heat rate was set at 10^oC and 2 hours of ramping time to reach desired temperature of 1200^oC. The holding time was 3 hours and no restriction for the subsequent cooling.

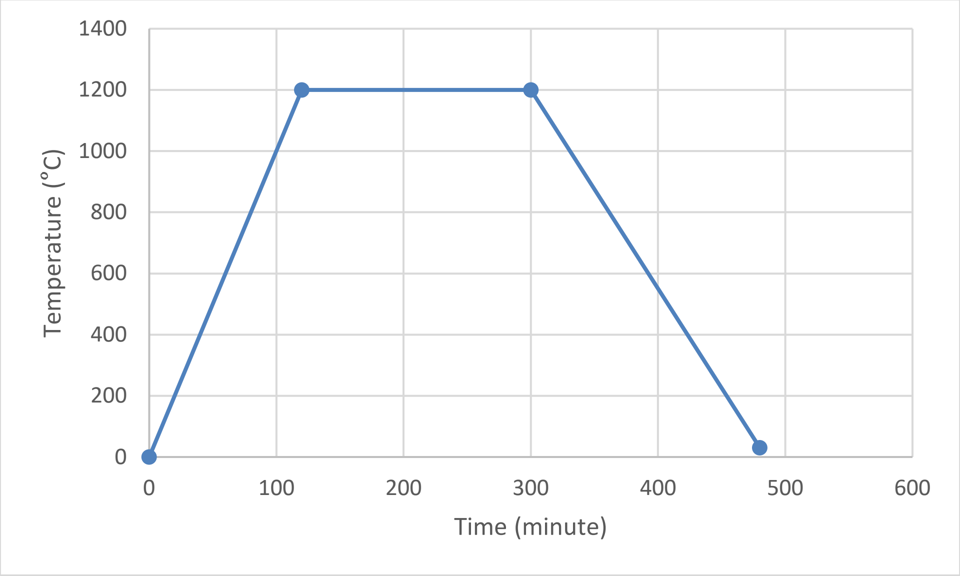


Figure 3.2: Illustration of calcination process.

Figure 3.3: LT Furnace.

High Shear Mixer

The High Shear Mixer was used for the mechanical activation of the precursors. The rotation speed of the mixer was set at 4800 RPM across all the experiment for consistency of the result.

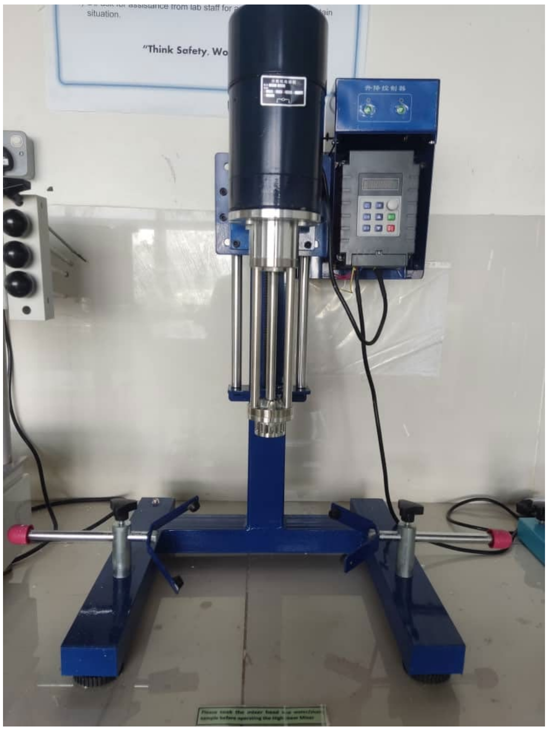


Figure 3.4: High Shear Mixer.

Roller Machine

The roller machine is a type of milling equipment that used in various industries for reducing the particle size of materials. The mixing bottle filled with the ceramic grinding balls and the precursor powder was put on the roller machine for prolonged milling time of 24 hours.

3.2 Materials

The materials used in this experiment were carefully selected to optimize the synthesis of magnesium orthosilicate. The precursor of the magnesium orthosilicate synthesis are Fumed silica and Periclase (MgO). The SHMP act as dispersing agent and the ethanol and deionized water were use for facilitating the mixing process.

3.2.1 Fumed silica

Fumed silica, SiO_2 was produced through high temperature method where the silicon tetrachloride vapor undergoes hydrolysis within flame fuelled by hydrogen and oxygen. It has large surface area and extremely small particle size. It is also a thickening agent as it will increase the viscosity of the mixture without changing their properties. (Changhua C. Liu, Gary E. Maciel, 1996)

In this experiment, the SiO_2 was prepared under the molar ratio of $MgO\ to\ SiO_2$, 2:1 which means one mole of fume silica is required for formation of magnesium orthosilicate. The SiO_2 was added into the mixing bottle containing deionized water. As the particle size of fumed silica is extremely small and light, the addition of SiO_2 was done in the fume chamber to prevent any respiratory intake of the powder. To prevent the mixture from becoming too viscous and affecting the dispersion by the high shear mixer, SiO_2 was added first, followed by MgO. Adding MgO after SiO_2 ensures that the mixture from being too viscous and the dispersion effect is maintained.

3.2.2 Periclase

Periclase, MgO is also known as magnesium oxide is normally found in marble produced by the process of metamorphism of limestone. The periclase was used as precursor for synthesis of magnesium orthosilicate.

3.2.3 Sodium Hexametaphosphate

In investigating the impact of sodium hexametaphosphate (SHMP) on magnesium orthosilicate formation, the addition sequence of SHMP is crucial, given its role as a dispersant widely employed in mineral processing and clay industries to optimize surface morphology. By adding SHMP prior to the precursor powder, the mixing efficiency was enhanced, and weight loss minimized, as the dispersant facilitates even dispersion throughout the mixture. This strategic sequence ensures that the precursor powder does not accumulate on the mixture top surface, promoting thorough integration and uniform distribution of SHMP, thereby potentially influencing the formation process of magnesium orthosilicate.

3.2.4 Ethanol

Ethanol, C_2H_5OH is a polar solvent which enables it to dissolve in wide range of substances. This property make it become useful for grinding and dispersion of material. Ethanol used for flushing purpose when addition of precursors into the container which contains deionized water. It also has low boiling point which is around 78^oC , hence the drying temperature of the grinded material to set around 80^oC.

3.2.5 Deionized water

Deionized water is high purified and removed most of the ions. This reduces the risk of contamination of the grinded material.it also helps to dissipate heat generated by friction. This is beneficial for preventing damage to sensitive materials or maintaining the integrity of heat-sensitive compounds. Hence, the deionized water has a certain period of before it degrades to become distilled water.

In general synthesis of magnesium orthosilicate, the addition of deionized water is not limited to prevent the mixing mixture too viscous and bulky which may cause the mixer to overheat. It is notable that in the experiment setup to study the effect of SHMP on the magnesium orthosilicate formation, the addition of deionized water must follow the water to solid ratio of 10:1.

3.3 Developing effective mechanical activation method for Magnesium Orthosilicate synthesis

The mixing method used in this experiment were High Shear Mixing and Planetary Ball Mill. To compare the effectiveness of these methods, the mixing duration and the presence of the MgO and enstatite will be a crucial factor for developing the efficient methods.

3.3.1 High shear mixer

High Shear Mixing involves subjecting the magnesium orthosilicate precursor materials to intense shearing forces, typically achieved by using a high-speed rotor-stator mixer. Initially, 500ml of deionized water was prepared in the mixing container. The fumed SiO_2 was added in the container for initial mixing at the speed of 2000 RPM for 10 minutes. The MgO was added to mixture and the speed of the mixer was operating at the speed of 4500 RPM. Ethanol will be added for flushing purpose to reduce powder loss which sticks on the container walls and stator.

The parameter was manipulated is the mixing duration which are 2 hours and 1 hour. The mixture was dried and calcinated at $1200\ ^{o}C$ for 3 hours. The calcinated powders was went for characterization using XRD to identify the purity of magnesium orthosilicate powder.

3.3.2 Ball mill

The planetary ball mill required a mixing bottle filled with different sizes of ceramic grinding balls. The ratio of large to small ceramic grinding balls was 1:10. Firstly, ethanol was prepared with a total precursor volume ratio of 10 and loaded into the mixing bottle. The mixing bottle was then loaded onto the rolling machine and run at 1000 RPM for 12 hours. After the mixing, the mixture was submerged into an ultrasonic bath for 5 minutes. The mixture and the grinding balls were separated for subsequent drying and calcination at 1200°C for 3 hours. The calcinated powders were then characterized using XRD to identify the purity of the Magnesium Orthosilicate.

3.4 Optimization of calcination temperature and duration

The optimization of the synthesis process involves the determination of the suitable calcination temperature and duration to produce pure magnesium orthosilicate. The synthesis of magnesium orthosilicate will be same as the general method as mentioned in Figure 3.1, but the mechanical activation was done using High Shear Mixer. The calcination was done at $1000^o C, 1100^o, and\ 1200^o C$ with different rise time but same holding time of 3 hours. The details are shown below:

Table 3.1: Calcination Temperature and ramp time.

Calcination Temperature	Ramp Time (minutes)
$1000^o C$	100
$1100^o C$	110
$1200^o C$	120

For the optimization of the calcination duration, the synthesis of magnesium orthosilicate is same as the general method as mentioned in Figure 3.1 using High Shear Mixing calcinated at $1200^o C$ but different holding of 2, 3, and 4 hours. The calcinated magnesium orthosilicate powders tested through XRD analysis to determine the magnesium orthosilicate phase and the intermediate phase.

3.5 Effect of SHMP in the formation of Magnesium Orthosilicate

The study on the effects of SHMP on the magnesium orthosilicate formation will be divided into three parts which contains:

1. Hydration evolution of MgO
2. Effects of different concentration of SHMP in magnesium orthosilicate formation
3. Optimization of SHMP addition in magnesium orthosilicate formation

3.5.1 Effects of SHMP in the hydration evolution of MgO

To investigate the influence of sodium hexametaphosphate (SHMP) on magnesium orthosilicate formation, various weight percentages (wt%) of

SHMP was added during the magnesium orthosilicate synthesis process. For the synthesis of magnesium orthosilicate, SHMP weight percents of 0wt%, 1wt%, and 2wt% will be added relative to the 100g precursor weight. The SHMP was introduced into deionized water before mixing the precursor powders. This is due to the properties of SHMP acting as a dispersing agent, SHMP facilitates improved mixing and preventing powder aggregation in the initial stage of mixing. Mixing was carried out for 1.5 hours using a High Shear Mixer at 4500 RPM. Subsequently, the mixture was divided equally into five portions to be stored for Day 1, 7, 14, 30, and 60. This is aimed to study the effects of hydration evolution of MgO in magnesium orthosilicate formation. The mixture after left for hydration for 1, 7, 14, 30 and 60 days was dried and send for XRD analysis. The dried powder was stored in the freezer to prevent the hydration continue to occur before drying and calcination to occur.

Table 3.2: Mass composition of Mixture 1, 2, and 3.

Mixture	MgO (g)	SiO_2 (g)	Deionized water (g)	SHMP (wt%)	SHMP (g)
1	68.754	51.248	1000	0	0
2	68.754	51.248	1000	1	1.2
3	68.754	51.248	1000	2	2.4

3.5.2 Effects of different concentration of SHMP in Magnesium Orthosilicate formation

To study the effects of the SHMP on magnesium orthosilicate formation, the mixture after 1, 7, 14, 30, and 60 days of hydration was dried and stored in the freezer to prevent further hydration and causing the results to be inaccurate. The volume discrepancies among powders of equal mass were observed. The XRD analysis was carried out on the precalcinated powders for study if hydration of MgO. Subsequently, the mixtures went for calcination at $1200°C$ and $1100°C$ for each SHMP concentration and time interval. Finally, XRD and SEM analyses was conducted to characterize the magnesium orthosilicate. The focus of this section will be the purity of the magnesium orthosilicate phase.

3.5.3 Optimization of SHMP addition in Magnesium Orthosilicate formation

The viscosity of the samples subjected to 0, 1, 7, 14, 30 and 60 days was tested through rheometer. The 0 day sample means that the sample will be tested immediately once the mixing was done. In the pursuit for optimization of the water to solid ratio, the SHMP addition will be set at highest of 2wt%. The water to solid ratio that will be tested ranging from 10 to 6 until achieving the viscosity of 0wt% SHMP to synthesis magnesium orthosilicate. Hence, the viscosity data of 0wt% SHMP mixture will become benchmark for this optimization process based the 2wt% mixture.

In this part of the experiment, 100ml of deionized water was loaded into the mixing bottle and running at 500 RPM. The SHMP will be loaded first, followed by the precursors according to the water to solid ratio as shown below:

Table 3.3: Mass of precursor.

Water to Solid ratio	Deionized water (ml)	MgO (g)	SiO_2 (g)
10:1	100	5.7295	4.2706
9:1	100	6.3661	4.7451
8:1	100	7.1618	5.3383
7:1	100	8.1849	6.1009
6:1	100	9.5491	7.1177

During the mixing process, the viscosity test was carried out simultaneously. The samples were extracted from the mixing bottle for rheology test for every 10 minutes after proceeding to the next water to solid ratio. When the viscosity against shear rate curve was obtained for various water to solid ratio, the comparison was done to find the optimum viscosity using interpolation method.

3.6 Characterization of Magnesium Orthosilicate

3.6.1 X-Ray Diffraction (XRD) Analysis

XRD patterns of the calcinated powder were obtained using X-Ray diffractometer (XRD-6000, Shidmazu) to study to characterize its phase composition. The diffraction patterns were obtained within a 2θ ranges from 10^o to 60^o with a measuring step of 0.02^o and measuring speed of $2^o/min$. The data obtained was plotted into a graph in Y-stacked pattern using OriginPro software and the peaks are identified using the HighScorePlus software.

Figure 3.5: X-ray Diffractometer.

3.6.2 Scanning Electron Microscope (SEM)

Scanning Electron Microscopy (SEM) is a popular technique for studying the surface morphology, topography, and composition of materials at high resolution. Because the samples are in powder form and not classified as transition metals, a thin layer of gold and vanadium will need to be coated using a sputter coater. The coating is primarily employed to retain the original surface shape by providing a conductive surface with which electron beams may interact, as well as to limit charging effects and so increase picture quality. The test included three different wt% (Mixture 1, Mixture 2, Mixture 3) of SHMP with varying time intervals to evaluate the impact on powder size. The photos were recorded at an accelerated voltage of 15.0kV with magnifications ranging from 4.0k to 20.0k.

Figure 3.6: Scanning Electron Microscopic.

3.6.3 Rheology

Anton Paar Rheometer (Physica MCR301) and Anton Paar Rheocompass software were used to evaluate the viscosity of the mixture which added different wt% of SHMP during the magnesium orthosilicate synthesis process. In terms of setup, a 25.0mm stainless steel parallel plate with a 1.0mm gap between the parallel plates was used for the test. Furthermore, it is important to load appropriate amount of mixture sample to avoid underfilling and overfilling, which will cause inaccurate data. The excess sample been squeezed out from the gap should be trimmed away before starting the test.

The viscosity test was performed by gradually increasing the shear rate from $0.01\ 1/s\ to\ 100\ 1/s$ under constant ambient temperature. The graph generated should have the viscosity (mPas) against shear rate with the viscosity will be using a logarithmic ramp.

Figure 3.7: Rheometer.

3.7 Summary

This experiment involved synthesizing magnesium orthosilicate through various mechanical activation methods and calcination conditions. The setup details and materials usage were outlined to suit each experiment segment's requirements. Effective mechanical activation methods, including high shear mixing and planetary ball milling, were employed, with operational procedures specified. Optimization of calcination conditions entailed testing three different temperatures with identical holding times and durations with a consistent temperature of $1200^{o}C$. Additionally, the study on SHMP's impact on magnesium orthosilicate formation encompassed examining hydration evolution, evaluating different SHMP concentrations, and optimizing SHMP addition. By manipulating SHMP percentages during synthesis, improved mixing and prevention of aggregation were achieved, with viscosity testing and XRD analysis unveiling their effects on magnesium orthosilicate formation. This comprehensive investigation aimed to elucidate SHMP's role in enhancing magnesium orthosilicate synthesis, contributing to materials science advancements. Furthermore, magnesium orthosilicate characterization involved XRD analysis within a specified range and speed, SEM examination of surface morphology post-coating, and rheological evaluation using an Anton Paar Rheometer, with viscosity plotted against logarithmic shear rate.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

This chapter will present and discuss the experimental results and various tests conducted. The discussion will commence with the investigation of effective mechanical activation through XRD analysis. Following this, optimization of calcination conditions will be explored by determining the optimal calcination temperature and holding duration necessary for obtaining pure magnesium orthosilicate, with XRD analysis serving as the primary analytical tool. Subsequently, the influence of SHMP on the hydration of MgO will be examined to determine whether it accelerates reaction kinetics. The samples will be analysed for phase composition, rheological properties, and surface morphology using XRD, SEM, and rheology techniques. Finally, the optimization of SHMP addition in magnesium orthosilicate production, based on the findings in section 4.5, will be conducted to determine the optimal weight percentage crucial for mass production of magnesium orthosilicate.

4.2 Evaluation of mixing methods

In this section, there will be evaluating the effectiveness of different mixing methods for the synthesis of magnesium orthosilicate. The Figure 4.1 and Figure 4.2 show the XRD patterns of the samples of undergoing high shear mixing and planetary ball mill respectively.

The XRD pattern displayed the formation of magnesium orthosilicate obtained through different methods. The sharp peaks and low background suggest the powder were highly crystalline. The samples were calcinated at $1200^{o}C$ and holding time of 3 hours. The Figure 4.1 and Figure 4.2 show the XRD patterns of the samples of undergoing high shear mixing and planetary ball mill respectively. In high shear mixing, the mixing duration of 1 hour and 2 hours were tested. The XRD analysis shows that pure magnesium orthosilicate is obtained in both samples. There was no presence of MgO appeared in the samples. The intensity of magnesium orthosilicate peaks for both samples were the same indicates that the optimization of the mixing duration for the precursors can be further reduced to 1 hour.

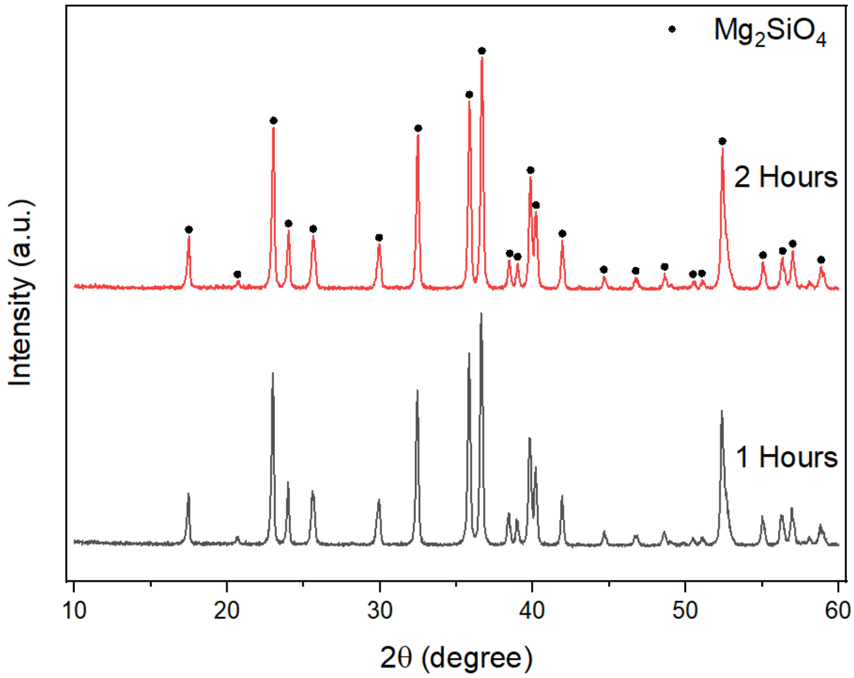


Figure 4.1: X-Ray Diffraction pattern of High Shear Mixing calcinated at 1200^o $C$and holding time 3 hours.

In contrast, the Figure 4.2 showed that ball milling sample contained MgO. The MgO peaks appeared at $2\theta \approx 43^o$ indicates that there was an incomplete reaction of MgO and SiO_2. According to report from Pacek, Ding and Utomo (2007), it was found that high shear mixing is utilized to disperse nanoparticles effectively in an aqueous solution, ensuring homogeneity and stability. This mixing process involves breaking down aggregates within the nanoparticles into smaller primary nanoparticles. The forces generated by high shear surpass the cohesive bonds between particles or smaller aggregates, causing the breakdown of ionic and hydrogen bonds present in the precursors. By comparing both mixing methods, the ball milling cannot achieve homogenous mixture as proven that periclase was detected in the calcined powder. Besides that, the ball milling consumed more energy compared to high shear mixing, especially for size reduction. The rotational motion of the milling chamber and the grinding balls requires significant energy input. and cannot achieve homogenous formation of magnesium orthosilicate. Overall, high shear mixing emerged as a more efficient method for synthesizing magnesium orthosilicate, offering the potential for shorter processing times and reduced energy consumption compared to ball milling.

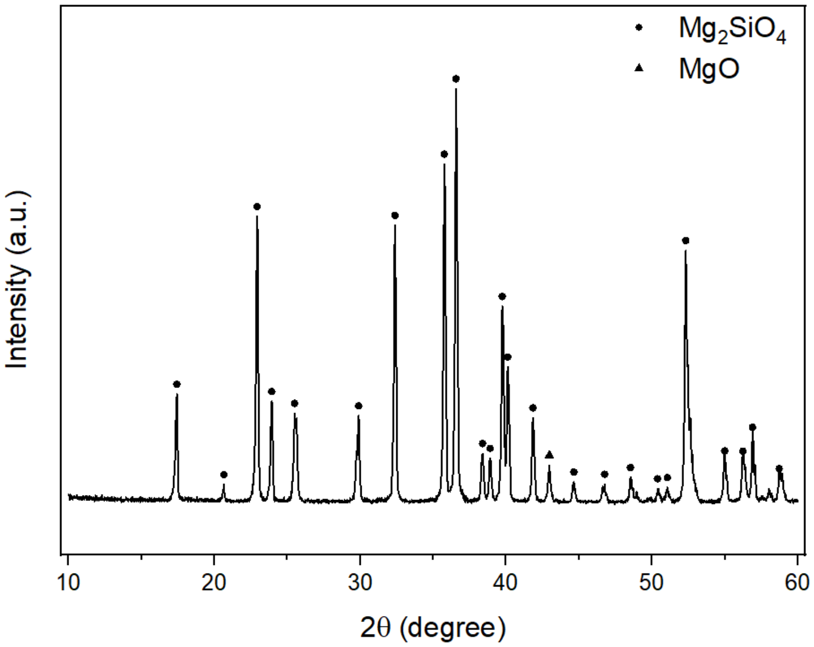


Figure 4.2: X-Ray Diffraction of Ball Mill calcinated at $1200^{o}C$ and holding time 3 hours.

4.3 Study of Optimum calcination temperature and duration

To investigate the potential formation of magnesium orthosilicate during subsequent heat treatment, nano MgO and fumed silica were mechanically mixed by using High Shear mixing for 2 hours at a speed of 4500 RPM and then calcinated at various temperatures. The Figure 4.3 below showed the XRD pattern of the samples undergo calcination at different temperature.

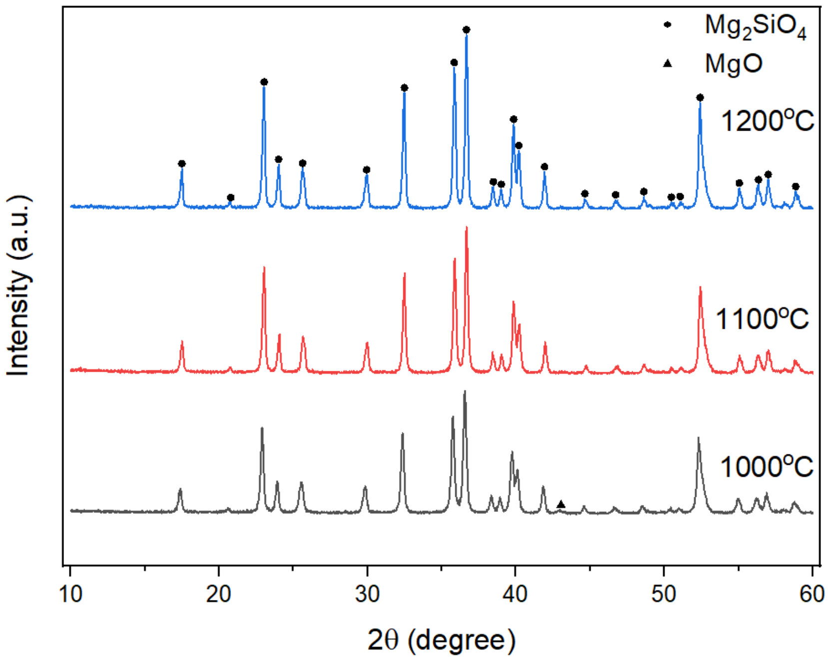


Figure 4.3: XRD pattern of the samples undergo calcination at different calcination temperature.

Pure magnesium orthosilicate was successfully obtained at temperatures of $1200°C$ and $1100°C$ indicating complete conversion of the precursor materials. However, at lower calcination temperatures of $1000°C$, the XRD analysis revealed the presence of MgO peaks at $2\theta \approx 43°$. This indicates incomplete reaction to form magnesium orthosilicate. This incomplete conversion at lower temperatures suggests a multi-step reaction pathway. Initially, MgO reacts with SiO_2 to form $MgSiO_3$:

$$MgO + SiO_2 \rightarrow MgSiO_3 \tag{4.1}$$

Then, enstatite reacts with the remaining MgO to form magnesium orthosilicate powder at the elevated temperature:

$$MgSiO_3 + MgO \ \rightarrow Mg_2SiO_4 \tag{4.2}$$

Moreover, the reaction rates at $1200°C$ and $1100°C$ are found to be better compared to $1000°C$ indicating that MgO and SiO_2 initially form $MgSiO_3$, followed by the reaction of enstatite with the remaining MgO to form magnesium orthosilicate. This finding aligns with previous studies by Brindley and Hayami, who suggested that MgO initially diffuses into the surface of SiO_2 to form enstatite, and then continues to diffuse through the enstatite layer to form magnesium orthosilicate. The process can be promoted by dynamically maintained high reaction interface areas, which can be achieved through high shear mixing (Brindley, G.W. and Hayami, R., 1965). The absence of periclase and enstatite in XRD patterns suggests that during mechanical activation, a homogeneous powder mixture was achieved. This finding matches the previous studies from F. Tavangarian and R. Emadi (2009), which proposed the formation of periclase and enstatite phases due to the lack of reactant homogeneity and shorten milling duration.

From an optimization perspective, the similarity in magnesium orthosilicate phases observed at $1200°C$ and $1100°C$ opens up opportunities for process optimization. By lowering the calcination temperature to $1100°C$ could help reducing energy consumption and process duration while maintaining the desired product quality. To perform optimization of calcination holding time, the nano MgO and fumed Silica (SiO_2) were mixed through High Shear mixing for 2 hours and calcinated at 1200^oC with various holding time of 2, 3, 4 hours. The Figure 4.4 below shows the XRD pattern of the samples undergo calcination at different temperature.

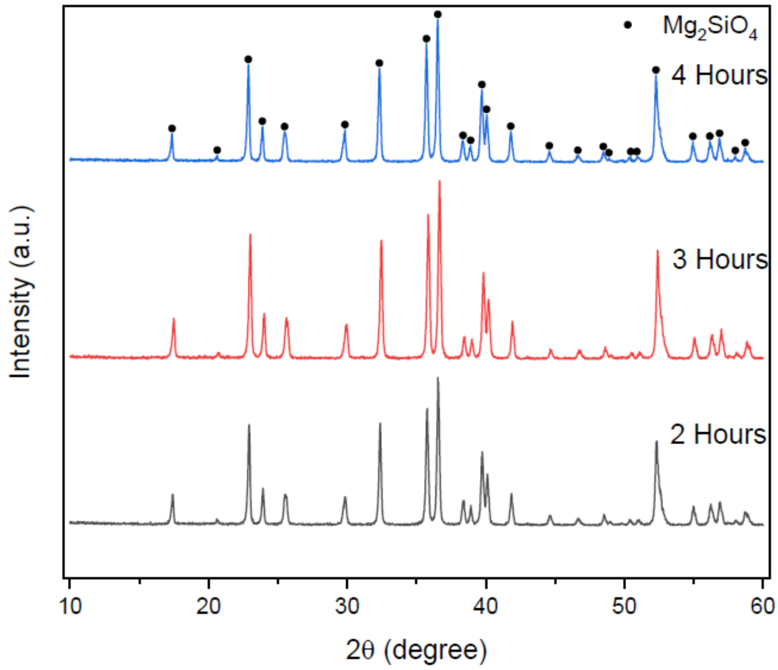


Figure 4.4: XRD pattern of Magnesium Orthosilicate powders calcinated at 1200^oC for different holding time.

The results demonstrated that pure magnesium orthosilicate phase can be successfully obtained with calcination holding durations of 2, 3, and 4 hours, with no MgO or enstatite observed in the samples. Interestingly, the sample subjected to a 3 hour calcination period exhibited higher crystallinity compared to the 2 hour duration, indicating that adequate time is essential for achieving optimal phase formation.

These findings align with previous research suggesting that the formation of magnesium orthosilicate involves a slow diffusion process between precursors, emphasizing the importance of sufficient reaction time. The calcination holding time emerges as a critical factor influencing crystallinity, with longer durations facilitating more complete solid-state reactions.

The phase transition of the mixed precursor initiates with the rapid consumption of the MgO phase, followed by the formation of a thin layer of magnesium orthosilicate at the interface of enstatite and remaining MgO through a slower diffusional process. With prolonged calcination, the thin layer of magnesium orthosilicate continues to develop, providing longer reaction times for MgO despite lower diffusion rates.

Considering these results, the optimization of the calcination holding time to 3 hours appears favourable for maximizing crystallinity. Longer durations may lead to enhanced phase purity and crystallinity but reduces energy consumption compared to the 4 hours of calcination holding time. Overall, the optimum calcination conditions to obtain pure magnesium orthosilicate will be $1100^{o}C$ holding for 3 hours and subsequent cooling to room temperature as illustrated in the graph below.

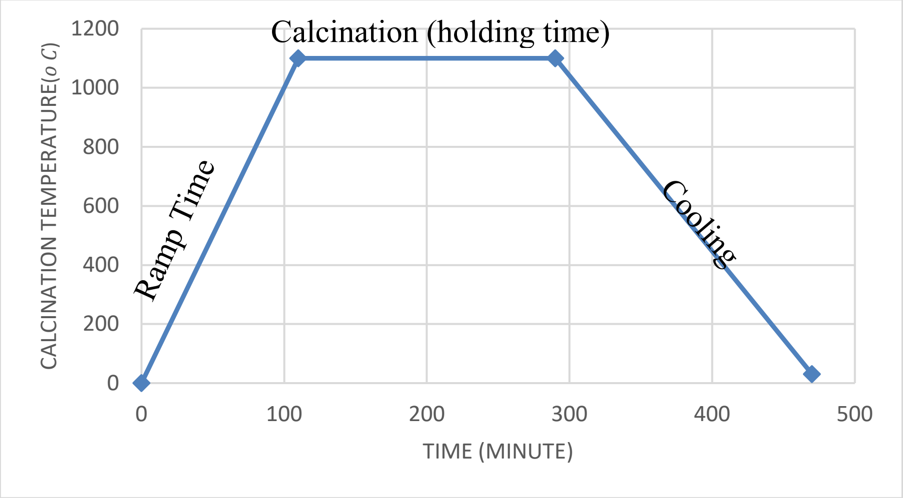


Figure 4.5: Illustration of Optimized Calcination Process.

4.4 Effects of SHMP on hydration evolution of MgO

4.4.1 XRD analysis

The XRD patterns elucidate the intricate dynamics of MgO hydration under varying concentrations of SHMP. In Figure 4.6, Mixture 1 with the absence of SHMP ($0wt\%$), the early onset of MgO hydration is obvious, evidenced by the emergence of $Mg(OH)_2$ peaks as early as Day 1. However, despite this prompt reaction, there persists a small MgO peak at $2\theta \approx 42^o$, which disappears completely after 30 days. The hydration of MgO can divided into dissolution of MgO and precipitation of $Mg(OH)_2$. The dissolution of MgO involves the release of Mg^{2+} and OH^- ions. The chemical reaction equation of precipitation of $Mg(OH)_2$ is shown below:

$$Mg^{2+} + 2OH^- \rightarrow Mg(OH)_2 \tag{4.3}$$

The formation of $Mg(OH)_2$ will provide a better reaction kinematics which helps in the study of magnesium orthosilicate synthesis.

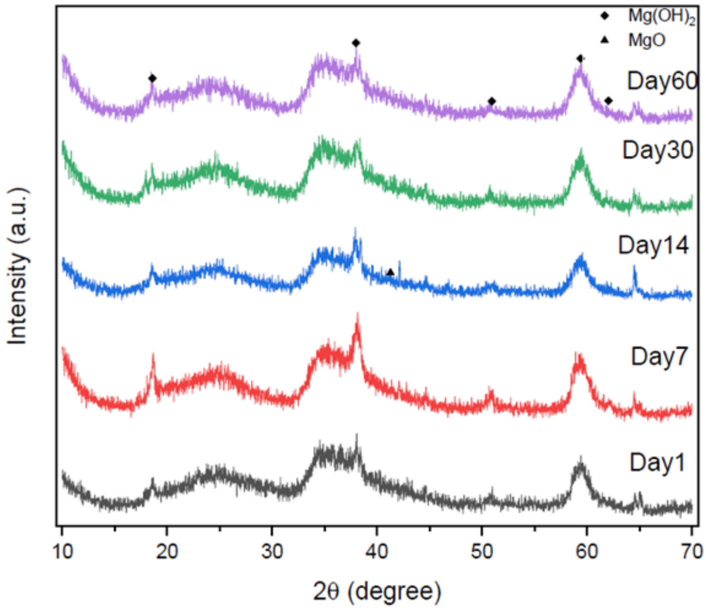


Figure 4.6: XRD Patterns of 0wt% SHMP after 1,7,14,30,60 days of Hydration of MgO.

Conversely, In Figure 4.7 showed the Mixture 2 which was subjected to 1wt% of SHMP. The hydration of MgO occurred as the $Mg(OH)_2$ appeared since Day 1. The $Mg(OH)_2$ peaks at $2\theta \approx 62^o$ steadily increases while the peak of MgO at $2\theta \approx 42^o$appeared and steadily decreased. Throughout Day 1 to Day 60. The continuous increment in $Mg(OH)_2$ peaks over 60 days indicates ongoing hydration, suggesting that SHMP helps create favorable conditions for MgO dissolution and the formation of $Mg(OH)_2$. However, the persistent presence of the MgO peak at $2\theta \approx 42°$ throughout the 60 days period, although gradually decreasing, this highlights the delicate balance between SHMP's benefits for workability and its possible limiting the hydration of MgO.

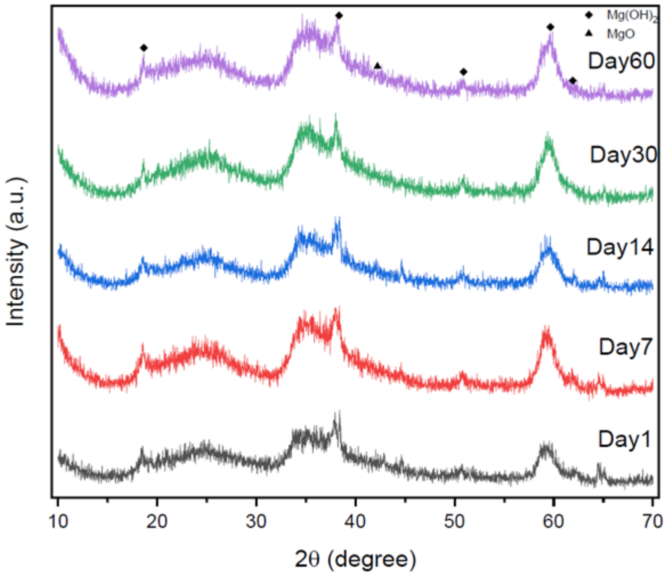


Figure 4.7: XRD Patterns of 1wt% SHMP after 1,7,14,30,60 days of Hydration of MgO.

Additionally, the Mixture 3 with the addition of 2wt% SHMP leads to a starkly different outcome for MgO hydration as demonstrated in Figure 4.8. This is evident from the sustained presence of MgO peaks at $2\theta \approx 42°$ throughout the entire 60 days observation period. The continuous presence of these peaks, coupled with the subdued $Mg(OH)_2$ peaks at $2\theta \approx 62°$ from Day 30 to 60, suggests inhibition to MgO hydration caused by the higher concentration of SHMP. This inhibition may stem from SHMP's role in reducing the release of Mg^{2+} and OH^- ions during dissolution of MgO, consequently impeding the formation of $Mg(OH)_2$. This phenomenon underscores the regulatory role of SHMP in modulating the hydration kinetics of MgO, thereby influencing the composition and characteristics of the resultant hydration products over time.

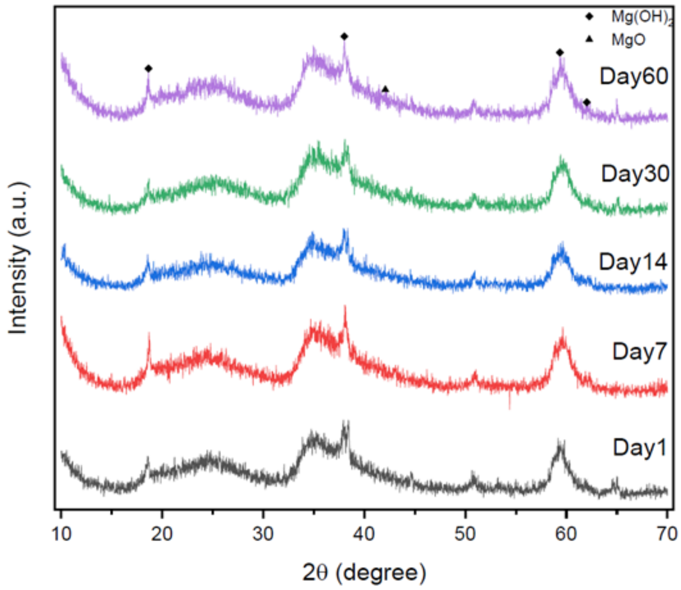


Figure 4.8: XRD Patters of XRD Patterns of 2wt% SHMP after 1,7,14,30,60 days of Hydration of MgO.

For 1wt% and 2wt%, the incomplete consumption of MgO is not solely attributed to the presence of SHMP but also to the stratification phenomenon observed in the mixture over time as shown in Figure 4.10. This stratification leads to insufficient reactants, hindering MgO hydration. As time progresses, stratification occurs, with the upper layer comprising distilled water and the lower layer containing the precursor. Consequently, MgO may remain not hydrated as water accumulates at the top, leaving MgO at the bottom without ample hydration medium. This lack of excess water prevents the complete hydration of MgO, contributing to its persistence in the XRD patterns. The 0wt% of SHMP addition, the stratification is not significance which allows hydration of MgO to occur.

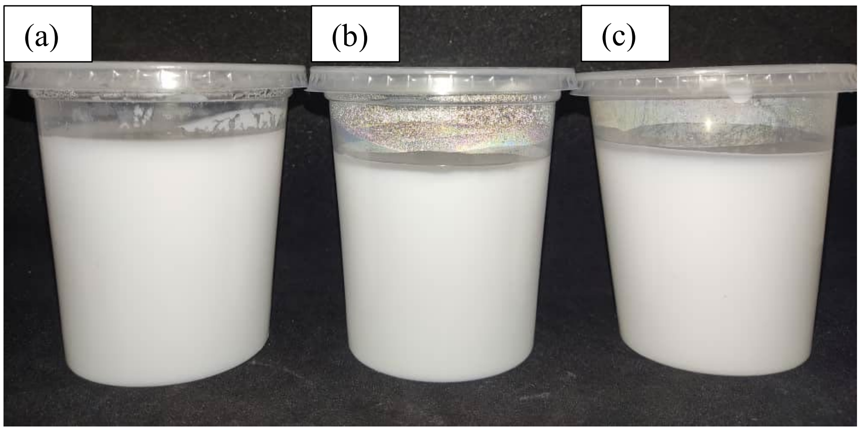


Figure 4.9: Demonstration of stratification (a)0wt%, (b)1wt%, (c)2wt%.

In summary, the XRD analysis of samples undergone MgO hydration with different SHMP addition reveals the possibility of producing pure magnesium orthosilicate. The 1wt% and 2wt% SHMP addition shows balance between SHMP's benefits and its potential limitations on MgO hydration.

4.4.2 SEM analysis

The observed differences in the FE-SEM images between the 0wt% and 1& 2wt% is shown in Figure 4.10 whereby SHMP conditions provide valuable insights into the hydration evolution of MgO. In the absence of SHMP (0wt%), the formation of crystalline $Mg(OH)_2$ solid particles are evident by Day 14, indicating significant progress in the hydration process. However, by Day 30, irregularly flocculated particles are observed, suggesting a complex and perhaps less controlled hydration pathway. This irregular morphology may indicate variations in hydration kinetics and product formation, potentially affecting the overall performance of the material.

Conversely, with the addition of 1 and 2wt% SHMP, the presence of $Mg(OH)_2$ crystalline solid particles are noted, but with a finer crystalline formation compared to the 0wt% condition. This finer crystalline formation suggests a more controlled and possibly accelerated hydration process, with SHMP likely playing a role in regulating the nucleation and growth of $Mg(OH)_2$ crystals. Furthermore, the observed growth of crystals, with a finer size distribution, implies improved homogeneity due the addition of SHMP.

Notably, in the 1 and 2wt% SHMP condition, uniform fluffy spheres of particles are formed, indicating a more uniform and organized hydration product compared to the irregular flocculated particles observed in the 0wt% scenario. This uniform morphology suggests a more controlled and optimized hydration process facilitated by the presence of SHMP. These observations highlight the significant influence of SHMP on the hydration evolution of MgO.

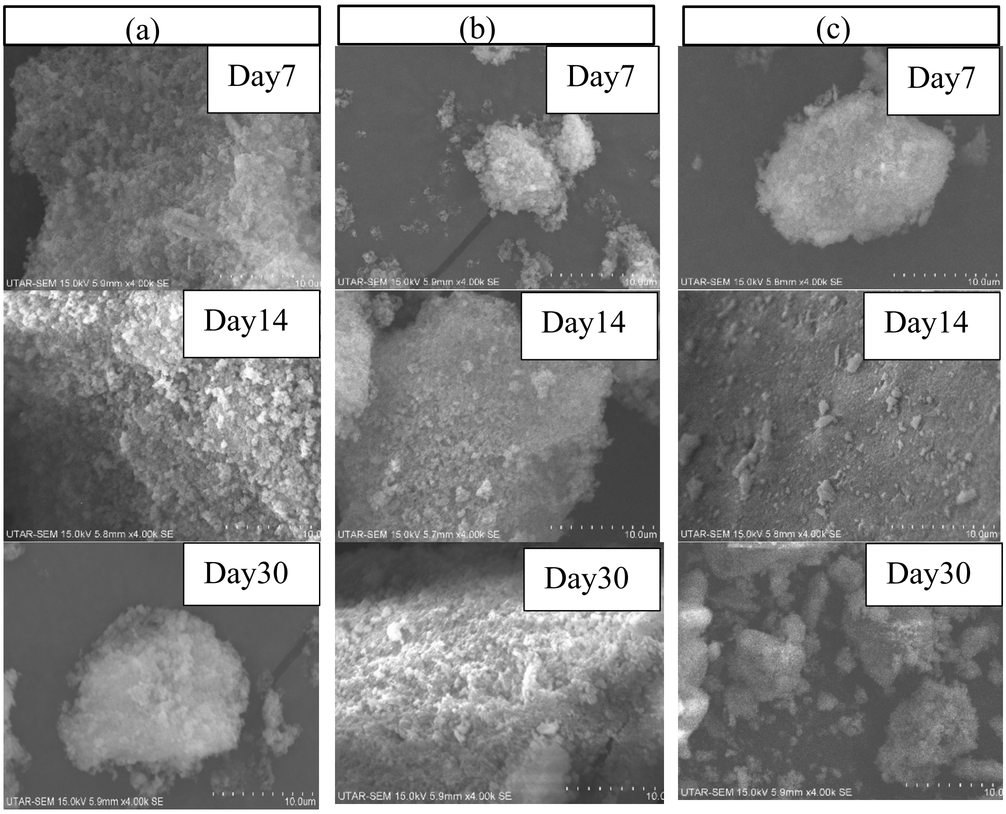


Figure 4.10: FESEM result of precalcinated powders (a)0wt%, (b)1wt%, (c)2wt%.

4.5 Effects of different concentration of SHMP in Magnesium Orthosilicate formation

4.5.1 Physical Observations

The physical observation was done after the drying process of the samples. It is worth noting there was volume difference between each wt% of SHMP in the samples. Figure 4.11 shows the volume difference of each wt% under the same mass of 30g. The 0wt% samples have a largest volume followed by 1wt% and 2 wt%.

From the literature review, it is evident that SHMP serves as a dispersant, enhancing particle dispersion and preventing agglomeration. Consequently, the addition of SHMP should theoretically diminish volume discrepancies by fostering a more uniform distribution of nanoparticles within the samples.

Moreover, SHMP's high solubility in deionized water leads to the formation of solutions with elevated water content. Provided that the hydrophilic nature of fumed silica in the samples, the inclusion of SHMP introduces additional water into the mixture. Upon drying, this elevated water content caused more pronounced shrinkage compared to samples with lower or no SHMP content.

In summary, while SHMP's dispersant properties theoretically promote uniformity and diminish volume discrepancies, its solubility in water and the resulting introduction of moisture can exacerbate shrinkage during the drying process, particularly in samples with higher SHMP content.

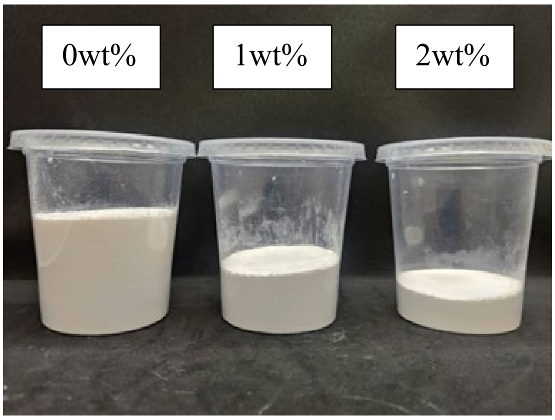


Figure 4.11: Volume difference of 0, 1,2wt% SHMP addition.

4.5.2 XRD analysis

The experimental investigation focused on the synthesis of magnesium orthosilicate through addition of varying weight percentages (wt%) of sodium hexametaphosphate (SHMP) as a dispersant. The samples were calcinated at $1100^{o}C$ and $1200^{o}C$ respectively to determine its composition phase. The XRD results showed different outcomes for each amount of SHMP added. This helps us understand how adding SHMP affects things like the way MgO reacts with water, how well everything gets mixed together, and how quickly certain phases form.

The calcinated powder from mixture 1 without SHMP (0wt%), the XRD analysis demonstrated the successful synthesis of pure magnesium orthosilicate at both $1100^{o}C$ and $1200^{o}C$. This outcome was attributed to the maximum hydration of MgO in deionized water, leading to the formation of $Mg(OH)_2$ as a precursor for magnesium orthosilicate synthesis. $Mg(OH)_2$ acts as a precursor for magnesium orthosilicate formation. It provides a more favourable starting point for the synthesis process, facilitating the conversion into magnesium orthosilicate more effectively than MgO alone.

In contrast, the Mixture 2 with the addition of 1wt% SHMP introduced complexities in the synthesis process. While the 1wt% sample also exhibited the capability to produce pure magnesium orthosilicate for both calcination temperature of $1100^{o}C$ and $1200^{o}C$, the appearance of enstatite peaks at specific time intervals (Day 30 and 60) hinted at challenges related to mixing homogeneity. According to previous study, homogeneous mixing may not achieve although dispersant (SHMP) is added. This is because despite the presence of dispersants, particles may agglomerate or settle due to insufficient agitation or incompatible chemical properties. Therefore, achieving true homogeneity may require optimization of mixing conditions, such as adjusting agitation speed, duration, or introducing additional dispersants tailored to the specific characteristics of the mixture (Du, et al., 2016). The optimization of the SHMP wt% will be done in section 4.6. Despite this, the findings suggested an improvement in the mechanism of magnesium orthosilicate formation attributed to the hydration of MgO facilitated by SHMP.

The Mixture 3 with the addition of 2wt% SHMP resulted in a distinct outcome. Enstatite peaks were consistently observed throughout the experimental duration and calcinated at $1100^{o}C$ and $1200^{o}C$. This indicates inhibition of $Mg(OH)_2$ formation due to the higher SHMP content. The abundance of MgO in the mixture, coupled with hindered $Mg(OH)_2$ formation, impacted the reaction kinetics, potentially leading to incomplete conversion to magnesium orthosilicate.

Overall, the experimental findings highlighted the significant influence of SHMP concentration on the synthesis of magnesium orthosilicate. While SHMP facilitated MgO hydration and improved the mechanism of magnesium orthosilicate formation, higher concentrations introduced challenges such as non-homogeneous mixing and inhibition of $Mg(OH)_2$ formation. Fine-tuning the SHMP concentration and optimizing mixing conditions emerge as critical factors for achieving desired phase purity and homogeneity in the synthesized magnesium orthosilicate products.

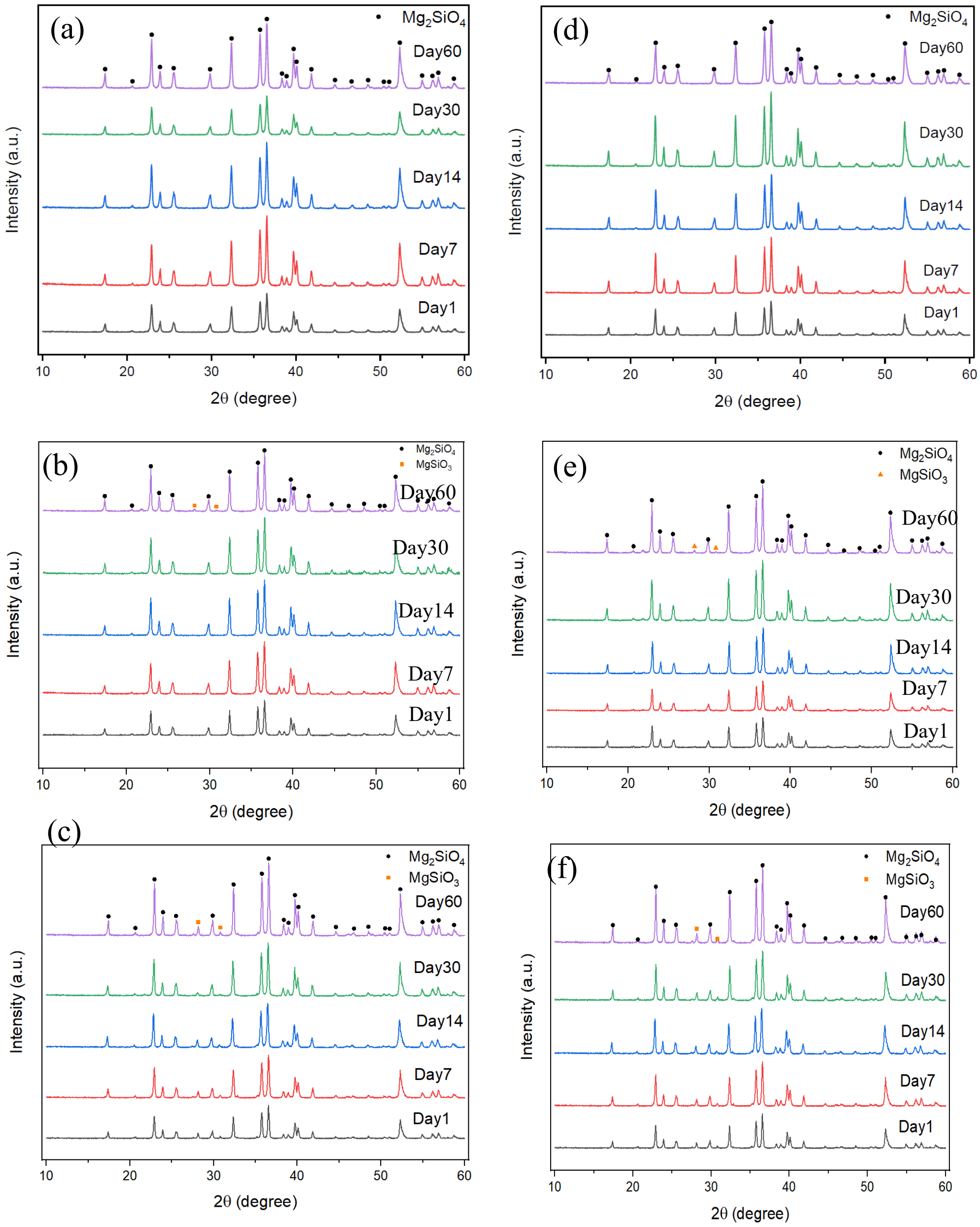


Figure 4.12: XRD Patterns of samples calcinated at 1100 ^{o}C (a)0wt%, (b)1wt%, (c)2wt% and 1200 ^{o}C (d)0wt%, (e)1wt%, (f) 2wt%.

4.5.3 SEM analysis

The experimental results demonstrate the significant influence of sodium hexametaphosphate (SHMP) concentration and calcination temperature on the morphology and size of magnesium orthosilicate particles. The particle and agglomerate size of 0wt% was the largest compared to 1wt% and 2 wt%. The addition of higher SHMP concentrations, particularly at 2wt%, lead to smaller particle sizes which attributed to SHMP's excellent dispersing and its ability to inhibit particle agglomeration and promote finer particles as shown Figure 4.13. Additionally, Figure 4.14 showed that magnesium orthosilicate particles calcinated at 1200°C exhibit smaller sizes compared to those at 1100°C, indicating calcination temperature affects the particle size reduction during calcination. Moreover, the prolonged hydration durations result in increased particle sizes and the formation of crystalline magnesium orthosilicate phases, with SHMP at 2wt% accelerating crystallization and promoting finer particle sizes earlier than lower concentrations after calcinated at the same calcination temperature of $1200^{o}C$ as shown in Appendix A. These findings highlight the critical role of SHMP in controlling magnesium orthosilicate particle morphology and size, offering insights into optimizing synthesis conditions for various applications in materials science and engineering.

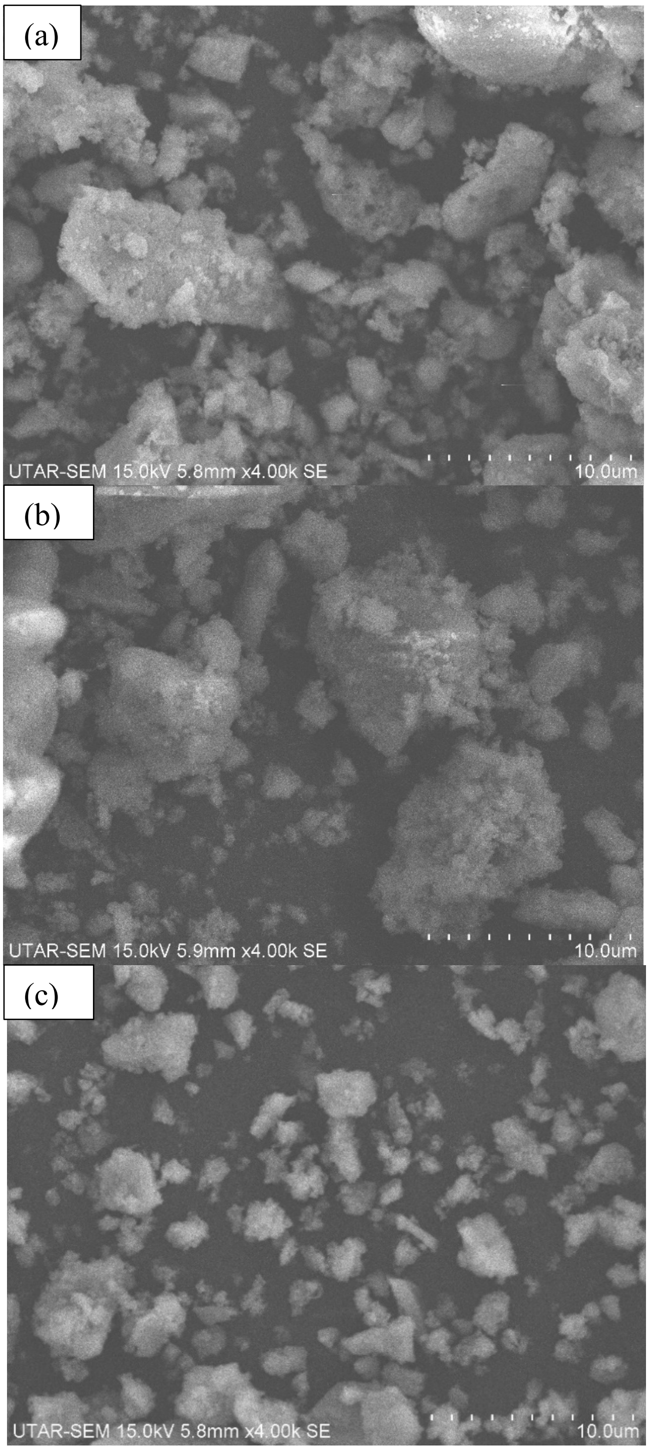


Figure 4.13: SEM image of powder (a)0wt%, (b)1wt%, (c)2wt% calcinated at $1100^{o}C$ after 30 days of hydration.

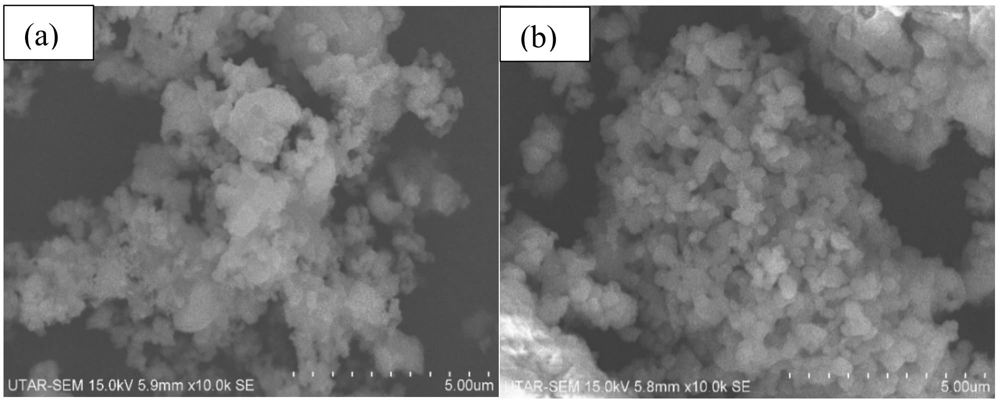


Figure 4.14: SEM image of powder with 1wt% SHMP addition calcinated at (a) $1100^{o}C$, (b)$1200^{o}C$.

4.6 Optimization of SHMP addition in Magnesium Orthosilicate formation

4.6.1 Rheology results

The viscosity study was carried out by comparing the viscosity at each time interval using the rheometer. Based on the observations, the addition of different wt% of SHMP significantly influenced the viscosity evolution of the mixture during the hydration of MgO. The figure illustrated the viscosity trend of different wt% after a certain time interval of hydration of MgO. The absence of SHMP in the 0wt% sample led to a steady increase in viscosity over the time intervals. There was a significant increment in viscosity on Day 14. This result suggested that without SHMP, there was a lack of surface modification, which allowed the particles to aggregate easily. Similarly, for the 1wt% samples, the SHMP samples exhibited a steep increment between Day 0 and Day 7, followed by a stagnation in viscosity increment. This indicated that there was partial dispersion but limited stability. In contrast, for the 2wt% samples freshly taken out from mixing, the viscosity curve showed noise due to incomplete dispersion, followed by a steady increase in viscosity over the duration of the experiment, suggesting efficient dispersion and stabilization of particles.

On the other hand, the comparison of viscosity evolution day by day was shown in Appendix B. The viscosity shear rate graph revealed distinct trends among the different SHMP concentrations. At the onset of hydration (Day 0), the 2wt% sample exhibited the lowest viscosity, followed by the 1wt%

and 0wt% samples. However, by Day 1, the viscosity of the 1wt% and 2wt% samples surpassed that of the 0wt% sample, indicating the initiation of particle dispersion. Subsequently, on Day 7, the viscosity of the 1wt% and 2wt% samples converged, surpassing that of the 0wt% sample, suggesting further dispersion and stabilization. On Day 14, the viscosity of the 1wt% sample exceeded that of the 2wt% sample, indicating potential overdosage of SHMP at 1wt%. However, by Day 30, the viscosity of the 2wt% sample encompassed that of the 1wt% sample, suggesting an optimal concentration for efficient dispersion and stabilization.

The observed trends could be attributed to the dispersing and hydrating effects. The higher the SHMP concentrations, the better the particle dispersion and stabilization, leading to reduced agglomeration and increased viscosity. However, overdosage of SHMP might lead to excessive dispersion, resulting in higher viscosities. Therefore, the optimization of SHMP concentration was essential to achieve the desired viscosity and stability for ceramic processing. Based on the findings, the 2wt% SHMP concentration appeared to offer the most efficient dispersion and stabilization, making it a potential candidate for optimization in ceramic processing applications. However, further experimentation and analysis were needed to confirm these observations and optimize SHMP concentration for specific applications.

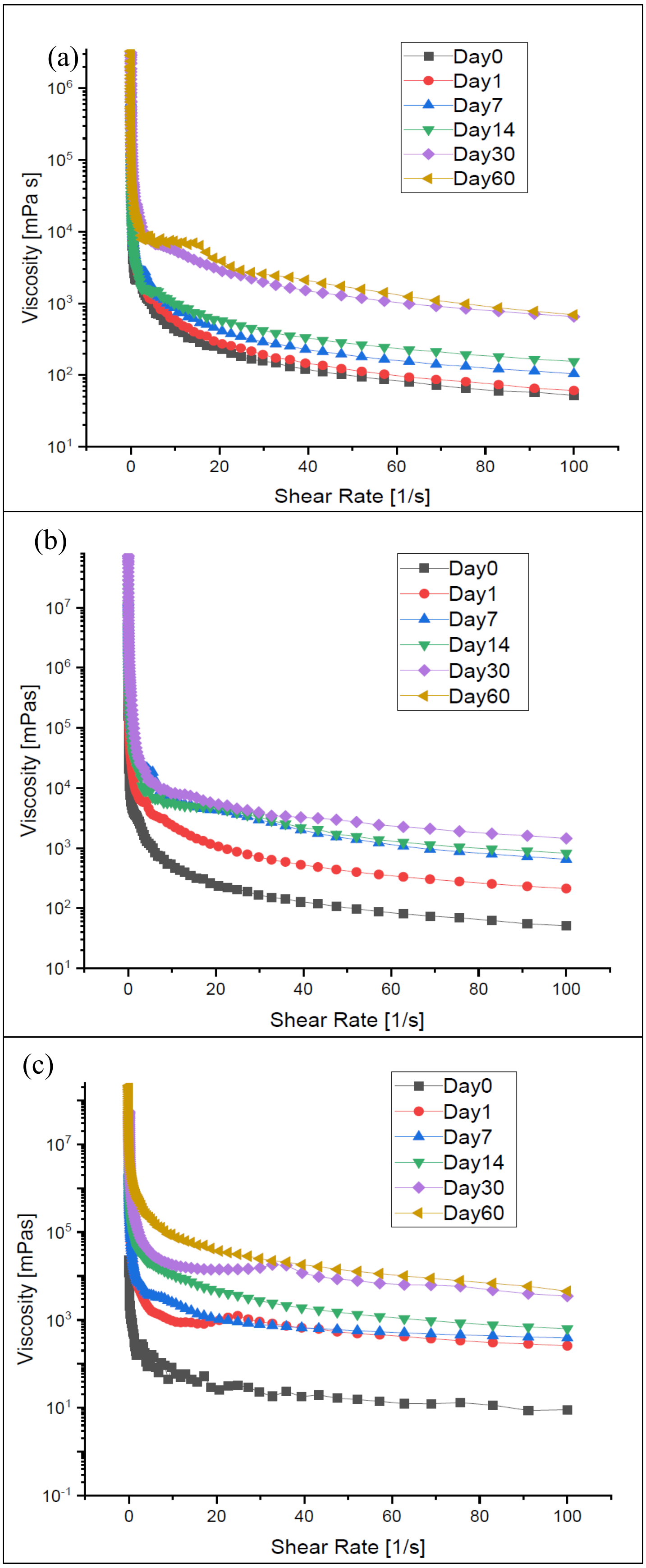


Figure 4.15: Viscosity against Shear rate curve of (a)0wt%, (b)1wt%, (c)2wt%.

4.6.2 Optimization of wt% of SHMP

The optimization will be carried out based on the results obtained from the section 4.6.1. In order to achieve mass production of magnesium orthosilicate, the 0wt% and 1wt% SHMP addition is proven in section 4.5.2 which capable to produce pure magnesium orthosilicate, but it will be advantageous to make a step forward by further increases the water to solid ratio up to 2wt%. Hence, the 2wt% SHMP addition will be investigated to find the optimum water to solid ratio. The viscosity curve of 0wt% in Day 0 will serve as reference for the optimization guideline. When the solid to water ratio increases, the viscosity will increase. Once the viscosity of the sample surpasses the reference line, this marks the maximum water to solid ratio. The optimum wt% will be done through calculations using interpolation method. The reference x axis point taken is at shear rate of 29.8 (1/s) as the viscosity of the samples achieved a stable state. The Figure 4.16 shows the viscosity of test carried out.

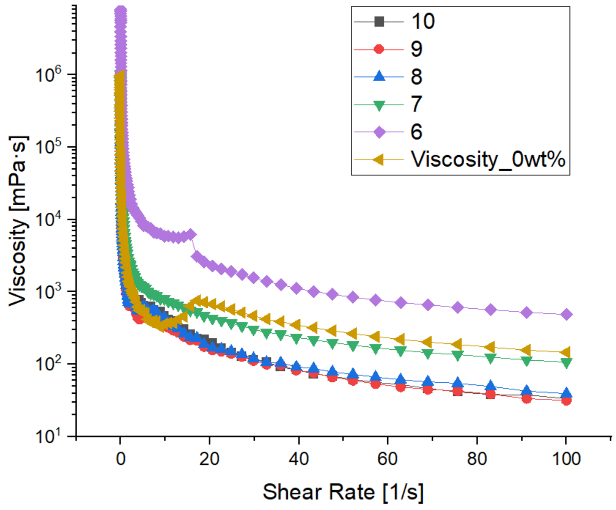


Figure 4.16: Graph of Viscosity against Shear Rate.

Table 4.1: Viscosity of various water to solid ratio at Shear rate of 29.8 [1/s].

Water to Solid Ratio	Viscosity [mPas]
7:1	305.14
6:1	1562

Calculations:

$$\frac{7-6}{305.14-1562}=\frac{x-6}{466.77-1562}$$

$$x=6.871$$

$$\therefore\ Hence, the\ optimum\ water\ to\ solid\ ratio\ is\ 6.871.$$

This optimization approach ensures that the viscosity of the mixture remains within acceptable limits for mass production of magnesium orthosilicate while maximizing the water-to-solid ratio to enhance processing efficiency. The calculated optimum water-to-solid ratio of 6.871 provides a quantitative basis for determining the appropriate SHMP concentration necessary to achieve the desired viscosity and stability for ceramic processing. This result underscores the importance of careful consideration of water to solid ratios in ceramic processing to ensure optimal material properties and production efficiency. Moreover, by leveraging the reference viscosity curve of the 0wt% SHMP sample, which has been proven capable of producing pure magnesium orthosilicate, the optimization process aligns with the objective of achieving high-quality ceramic materials. The calculated optimum water-to-solid ratio serves as a valuable parameter for fine-tuning the SHMP concentration in the production process, ultimately contributing to the successful mass production of magnesium orthosilicate with consistent quality and performance.

4.7 Summary

The study involves the development of effective mechanical activation methods, optimization of calcination duration and the investigate the effects of SHMP on the formation of magnesium orthosilicate.

For section 4.2, the X-ray diffraction (XRD) analysis of magnesium orthosilicate synthesis using high shear mixing and planetary ball milling revealed distinct outcomes. High shear mixing, conducted for durations of 1 and 2 hours, yielded pure magnesium orthosilicate, suggesting efficient synthesis within a shorter timeframe. Conversely, planetary ball milling resulted in the presence of MgO, indicating incomplete reaction between MgO and SiO2. The efficiency of high shear mixing in achieving homogeneity and stability through nanoparticle dispersion was evident, leading to the breakdown of aggregates and precursor bonds. This method demonstrated energy efficiency and the potential for reduced processing times compared to planetary ball milling, which requires significant energy input and cannot achieve homogenous magnesium orthosilicate formation. Therefore, high shear mixing emerges as a more effective mechanical activation method for synthesizing magnesium orthosilicate, offering shorter processing times and lower energy consumption.

Next, the section 4.3 carries out the investigation into magnesium orthosilicate synthesis highlighted calcination temperature dependent reactions, with pure magnesium orthosilicate successfully obtained at higher temperatures of $1200°C$ and $1100°C$, while incomplete reactions were observed at lower temperature of $1000°C\ and\ 900°C$. Further optimization through varying calcination holding times revealed that a 3-hour duration yielded higher crystallinity compared to shorter durations, emphasizing the importance of adequate reaction time. These findings suggest that optimizing conditions, such as calcination temperature and holding time, can enhance the synthesis of pure magnesium orthosilicate, with $1100°C$ for 3 hours identified as optimal for high-quality synthesis.

Furthermore, the section 4.4 will be revealing the effects of SHMP on the hydration evolution of MgO. Without SHMP, MgO starts reacting quickly, but some MgO remains unchanged after 30 days. With a small amount of SHMP (1wt%), the reaction continues steadily for 60 days, but there's still some unchanged MgO. However, if we add a lot of SHMP (2wt%), MgO doesn't react much at all, and there's still a lot of unchanged MgO after 60 days. This happens because SHMP affects how MgO dissolves in water. Additionally, there is stratification phenomenon happened where the water and MgO separate over time, making it harder for MgO to fully react. Looking at pictures of the reactions under a microscope, we see that SHMP helps make the reaction more uniform and controlled, resulting in finer crystals of the reaction product, which is magnesium hydroxide ($Mg(OH)_2$). These findings show that SHMP can change how MgO reacts with water, which could be useful for making materials with specific properties.

Moreover, Section 4.5 reveals the investigation of the synthesis of magnesium orthosilicate with varying amounts of sodium hexametaphosphate (SHMP) as a dispersant reveals important insights into the reaction process and resulting material properties. Without SHMP, pure magnesium orthosilicate is successfully synthesized, facilitated by thorough hydration of MgO in water.

However, adding 1wt% SHMP introduces complexities due to challenges in achieving homogeneous mixing, although pure magnesium orthosilicate is still obtained. Increasing SHMP to 2wt% inhibits $Mg(OH)_2$ formation, impacting reaction kinetics and leading to incomplete conversion to magnesium orthosilicate. Notably, SHMP concentration influences particle size and morphology, with higher concentrations resulting in smaller, more uniform particles. The calcination temperature also affects particle size, with higher temperatures leading to smaller particles. These findings emphasize the critical role of SHMP concentration and processing conditions in controlling the synthesis and properties of magnesium orthosilicate, offering valuable insights for materials engineering applications.

Lastly, the viscosity study in section 4.6 highlights the significant impact of sodium hexametaphosphate (SHMP) concentration on MgO hydration. Higher SHMP concentrations promote better particle dispersion and stabilization, leading to increased viscosity. However, overdosage may result in excessive dispersion and higher viscosities. Optimization using the viscosity curve of 0wt% as a reference yields an optimum water-to-solid ratio of 6.871, guiding SHMP concentration for efficient processing. This approach ensures viscosity remains within acceptable limits while maximizing water-to-solid ratio, crucial for successful mass production of high-quality magnesium orthosilicate.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

In conclusion, the most effective mixing method to synthesis magnesium orthosilicate is using high shear mixer. The high shear mixing is capable to produce pure magnesium orthosilicate after calcination at temperature of 1200^oC. The mixing duration can be optimized from 2 hours to 1 hour to get the same purity and crystallinity of the magnesium orthosilicate powder. The ball mill will be not capable of produce pure magnesium orthosilicate and contains MgO.

Next, the optimization of calcination conditions is carried by testing different temperature and holding time. Pure magnesium orthosilicate was successfully obtained at higher temperatures of $1200°C$ and $1100°C$, while incomplete reactions occurred at lower temperatures. Further optimization through varying calcination holding times demonstrated that a 3-hour duration yielded higher crystallinity compared to shorter durations, highlighting the importance of adequate reaction time. These findings suggest that optimizing conditions, such as calcination temperature and holding time of $1100°C$ for 3 hours identified as optimal for can enhance the synthesis of pure magnesium orthosilicate.

Furthermore, the viscosity study shows the vital role of SHMP concentration in the viscosity evolution during MgO hydration. There will be a steady increase in viscosity without SHMP (0wt%) to the efficient dispersion observed at 2wt% but unable to obtain pure magnesium orthosilicate. This highlights the importance of the balance required for optimal particle dispersion and stabilization. By leveraging the insights gained, particularly from the 0wt% samples proved capable to produce pure magnesium orthosilicate, an optimization approach is proposed. The optimum water-to-solid ratio of 6.871, offers a quantitative framework for fine-tuning SHMP concentration to achieve desired viscosity and stability while maximizing processing efficiency. Ultimately, this optimization process ensures consistent, high-quality magnesium orthosilicate production, underscoring its significance in materials engineering applications.

5.2 Recommendations for future work

5.2.1 Perform Brunauer-Emmett-Teller (BET) and Thermogravimetric Analysis (TGA) analysis.

The BET and TGA analysis will provide a deeper insight into the formation mechanism of magnesium orthosilicate. BET analysis will help determine the specific surface area and porosity of the synthesized magnesium orthosilicate, shedding light on its physical properties. TGA analysis can elucidate the thermal stability and decomposition behaviour of magnesium orthosilicate under different conditions, aiding in understanding its thermal properties and reaction kinetics.

5.2.2 Test higher weight percent (wt%) of SHMP addition.

One of the futures can be done is to explore the possibility of raising the wt% of SHMP addition to aid the mass production of magnesium orthosilicate. By varying the SHMP concentration, the influence of dispersant content on magnesium orthosilicate formation kinetics, particle morphology, and material properties can be systematically studied. This investigation can help identify the optimal SHMP concentration for achieving desired material characteristics and production efficiency.

5.2.3 Optimizing High Sehar Mixing

Further optimization of high shear mixing can be achieved by testing various mixing speeds. Experimenting with different rotational speeds of the mixing apparatus can fine-tune the dispersion and homogenization of precursor materials, leading to improved reaction efficiency and product quality. By systematically varying mixing parameters, such as speed and duration, the impact on magnesium orthosilicate synthesis can be assessed to optimize the mixing process for enhanced performance.

5.2.4 Exploration of alternative precursor

The future work can be done is exploring alternative precursor materials and synthesis routes can broaden the scope of magnesium orthosilicate synthesis. Investigating different sources of magnesium and silicon, as well as alternative methods such as sol-gel synthesis or hydrothermal methods, can offer alternative routes to magnesium orthosilicate production with potentially improved efficiency and tailored properties. By exploring diverse precursor materials and synthesis strategies, the versatility and applicability of magnesium orthosilicate in various applications can be expanded.

REFERENCES

Kukovecz, Á., Kanyó, T., Kónya, Z. and Kiricsi, I., 2005. Long-time low-impact ball milling of multi-wall carbon nanotubes. Carbon, 43(5), pp.994-1000.

Moropoulou, A., Bakolas, A. and Aggelakopoulou, E., 2001. The effects of limestone characteristics and calcination temperature to the reactivity of the quicklime. *Cement and concrete Research*, *31*(4), pp.633-639.

Bafrooei, H.B., Ebadzadeh, T. and Majidian, H., 2014. Microwave synthesis and sintering of forsterite nanopowder produced by high energy ball milling. *Ceramics International*, *40*(2), pp.2869-2876.

Brindley, G.W. and Hayami, R., 1965. Kinetics and mechanism of formation of forsterite (Mg2SiO4) by solid state reaction of MgO and SiO2. *Philosophical Magazine*, *12*(117), pp.505-514.

Carlisle, E.M., 1970. Silicon: a possible factor in bone calcification. *Science*, *167*(3916), pp.279-280.

Liu, C.C. and Maciel, G.E., 1996. The fumed silica surface: A study by NMR. *Journal of the American Chemical Society*, *118*(21), pp.5103-5119.

Cheney, B., 2007. Introduction to scanning electron microscopy. *Materials Engineering department San Jose State University*.

Cheong, D.I., Kim, J. and Kang, S.J.L., 2002. Effects of isothermal annealing on the microstructure and mechanical properties of SiC ceramics hot-pressed with Y2O3 and Al2O3 additions. *Journal of the European Ceramic Society*, *22*(8), pp.1321-1327.

Du, Y.J., Yang, Y.L., Fan, R.D. and Wang, F., 2016. Effects of phosphate dispersants on the liquid limit, sediment volume and apparent viscosity of clayey soil/calcium-bentonite slurry wall backfills. *KSCE Journal of Civil Engineering*, *20*, pp.670-678.

Emadi, R., Tavangarian, F., Zamani, F.R. and Gholamrezaie, A., 2011. INFLUENCES OF FLUORINE AND CHLORINE IONS ON THE FORMATION OF NANOSTRUCTURE FORSTERITE DURING MECHANICAL ACTIVATION OF TALC AND PERICLASE. *Journal of Ceramic Processing Research*, *12*(5), pp.538-543.

Tavangarian, F., Emadi, R. and Shafyei, A., 2010. Influence of mechanical activation and thermal treatment time on nanoparticle forsterite formation mechanism. *Powder Technology*, *198*(3), pp.412-416.

Gillet, P., Richet, P., Guyot, F. and Fiquet, G., 1991. High-temperature thermodynamic properties of forsterite. *Journal of Geophysical Research: Solid Earth*, *96*(B7), pp.11805-11816.

Groenendijk, I., van Delft, M., Versloot, P., van Loon, L.J. and de Groot, L.C., 2022. Impact of magnesium on bone health in older adults: A systematic review and meta-analysis. *Bone*, *154*, p.116233.

de Camargo, I.L., Morais, M.M., Fortulan, C.A. and Branciforti, M.C., 2021. A review on the rheological behavior and formulations of ceramic suspensions for vat photopolymerization. *Ceramics International*, *47*(9), pp.11906-11921.

Jia, Y., Wang, B., Wu, Z., Han, J., Zhang, T., Vandeperre, L.J. and Cheeseman, C.R., 2016. Role of sodium hexametaphosphate in MgO/SiO2 cement pastes. *Cement and Concrete Research*, *89*, pp.63-71.

John, 2022. *Annealing Aluminum: How's it Done.* [Online] Available at: https://mellowpine.com/blog/annealing-aluminum/

Liu, Y., Guo, J., Li, W., Yang, X., Li, W., Zhou, M. and Zhang, J., 2022. Comparison and estimation on deagglomeration performance of batch high shear mixers for nanoparticle suspensions. Chemical Engineering Journal, 429, p.132420.

AmanNejad, M. and Barani, K., 2021. Effects of ball size distribution and mill speed and their interactions on ball milling using DEM. *Mineral Processing and Extractive Metallurgy Review*, *42*(6), pp.374-379.

Farhanchi, M., Neysari, M., Vatankhah Barenji, R.E.Z.A., Heidarzadeh, A. and Taherzadeh Mousavian, R., 2015. Mechanical activation process for self-propagation high-temperature synthesis of ceramic-based composites: modeling and optimizing using response surface method. *Journal of Thermal Analysis and Calorimetry*, *122*, pp.123-133.

Ni, S., Chou, L. and Chang, J., 2007. Preparation and characterization of forsterite (Mg2SiO4) bioceramics. *Ceramics International*, *33*(1), pp.83-88.
Nilles, J.L., Coletti Jr, J.M. and Wilson, C., 1973. Biomechanical evaluation of bone-porous material interfaces. *Journal of biomedical materials research*, *7*(2), pp.231-251.

Ding, P., Orwa, M.G. and Pacek, A.W., 2009. De-agglomeration of hydrophobic and hydrophilic silica nano-powders in a high shear mixer. *Powder Technology*, *195*(3), pp.221-226.

Pacek, A.W., Ding, P. and Utomo, A.T., 2007. Effect of energy density, pH and temperature on de-aggregation in nano-particles/water suspensions in high shear mixer. *Powder technology*, *173*(3), pp.203-210.

Mosaner, P., Bonelli, M. and Miotello, A., 2003. Pulsed laser deposition of diamond-like carbon films: reducing internal stress by thermal annealing. *Applied Surface Science*, *208*, pp.561-565.

Pitawala, H.M.T.G.A., 2019. Mineralogy, petrography, geochemistry and economic potential of carbonate rocks of Sri Lanka. *Journal of the Geological Society of Sri Lanka*, *20*(1).

Ramesh, S., Yaghoubi, A., Lee, K.S., Chin, K.C., Purbolaksono, J., Hamdi, M. and Hassan, M.A., 2013. Nanocrystalline forsterite for biomedical applications: synthesis, microstructure and mechanical properties. *journal of the mechanical behavior of biomedical materials*, *25*, pp.63-69.

Walling, S.A., Kinoshita, H., Bernal, S.A., Collier, N.C. and Provis, J.L., 2015. Structure and properties of binder gels formed in the system Mg (OH) 2–SiO 2–H 2 O for immobilisation of Magnox sludge. *Dalton Transactions*, *44*(17), pp.8126-8137.

Sano, S., Saito, N., Matsuda, S.I., Ohashi, N., Haneda, H., Arita, Y. and Takemoto, M., 2006. Synthesis of high density and transparent forsterite ceramics using nano-sized precursors and their dielectric properties. *Journal of the American Ceramic Society*, *89*(2), pp.568-574.

Scalera, F., Gervaso, F., Sanosh, K.P., Sannino, A. and Licciulli, A.J.C.I., 2013. Influence of the calcination temperature on morphological and mechanical properties of highly porous hydroxyapatite scaffolds. *Ceramics International*, *39*(5), pp.4839-4846.

Seyforth, J.A., 2015. Scanning electron microscopy (SEM): an introduction to the use of SEM for characterising the surface topology and composition of matter with further applications. *Experimental Techniques In Condensed Matter Physics*, *30*(1), pp.779-89.

BAHETI, V., ABBASI, R. and MILITKY, J., 2013. Optimisation of ball milling parameters for refinement of waste jute fibres to nano/micro scale in dry conditions. *Journal of Textile Engineering*, *59*(5), pp.87-92.

Durham, W.B. and Goetze, C., 1977. A comparison of the creep properties of pure forsterite and iron-bearing olivine. *Tectonophysics*, *40*(3-4), pp.T15-T18.

Wang, Z.F., Chen, M.J., Ma, Y., Liu, H., Han, C.H. and Wang, X.T., 2017. Low - temperature molten salt synthesis of forsterite powders with controllable morphology. International Journal of Applied Ceramic Technology, 14(1), pp.3-8.

Zhi, W., Zhanjun, W. and Guodong, S., 2011. Effect of annealing treatment on mechanical properties of a ZrB2–SiC–graphite ceramic. *Materials Science and Engineering: A*, *528*(6), pp.2870-2874.

Jia, Y., Wang, B., Wu, Z., Han, J., Zhang, T., Vandeperre, L.J. and Cheeseman, C.R., 2016. Role of sodium hexametaphosphate in MgO/SiO2 cement pastes. *Cement and Concrete Research*, *89*, pp.63-71.

Živojinović, J., Kosanović, D., Blagojević, V.A., Pavlović, V.P., Tadić, N., Vlahović, B. and Pavlović, V.B., 2022. Dielectric properties of mechanically activated strontium titanate ceramics. *Science of Sintering*, *54*(4).

APPENDICES

Appendix A: SEM images of Magneisum Orthosilicate powders after calcinated at 1200^oC

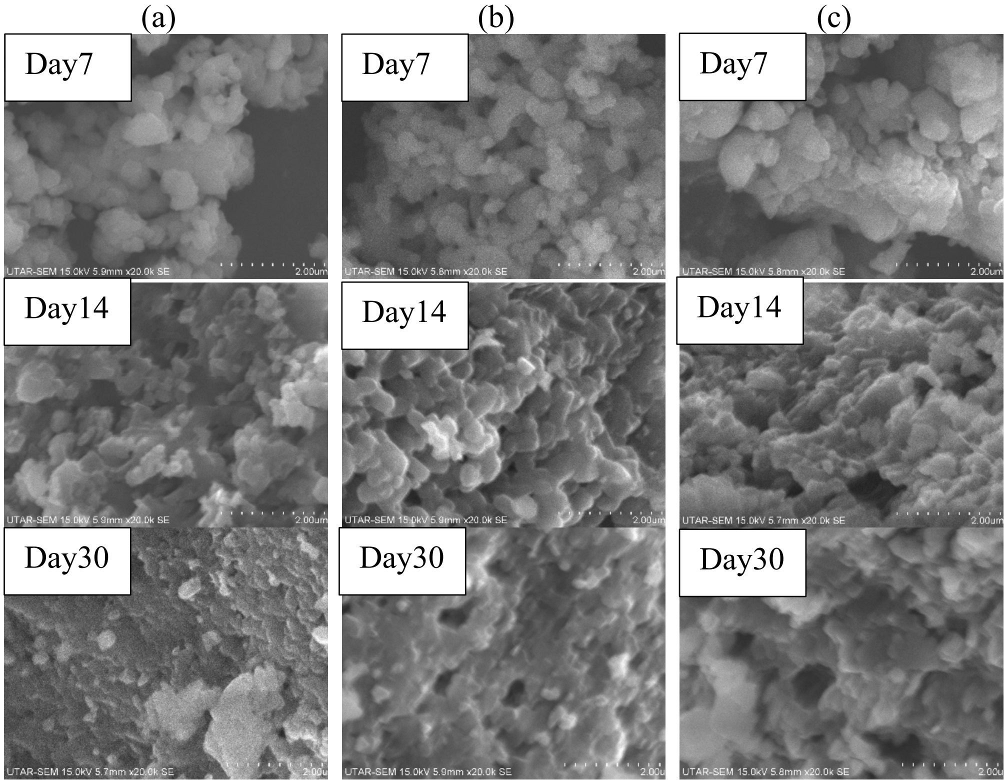


Figure A.5.1: SEM image of calcinated Magnesium Orthosilicate powder with (a)0wt%, (b)1wt%, and (c) 2wt% of SHMP addition.

Appendix B: Graphs of Hydration of MgO after various time intervals.

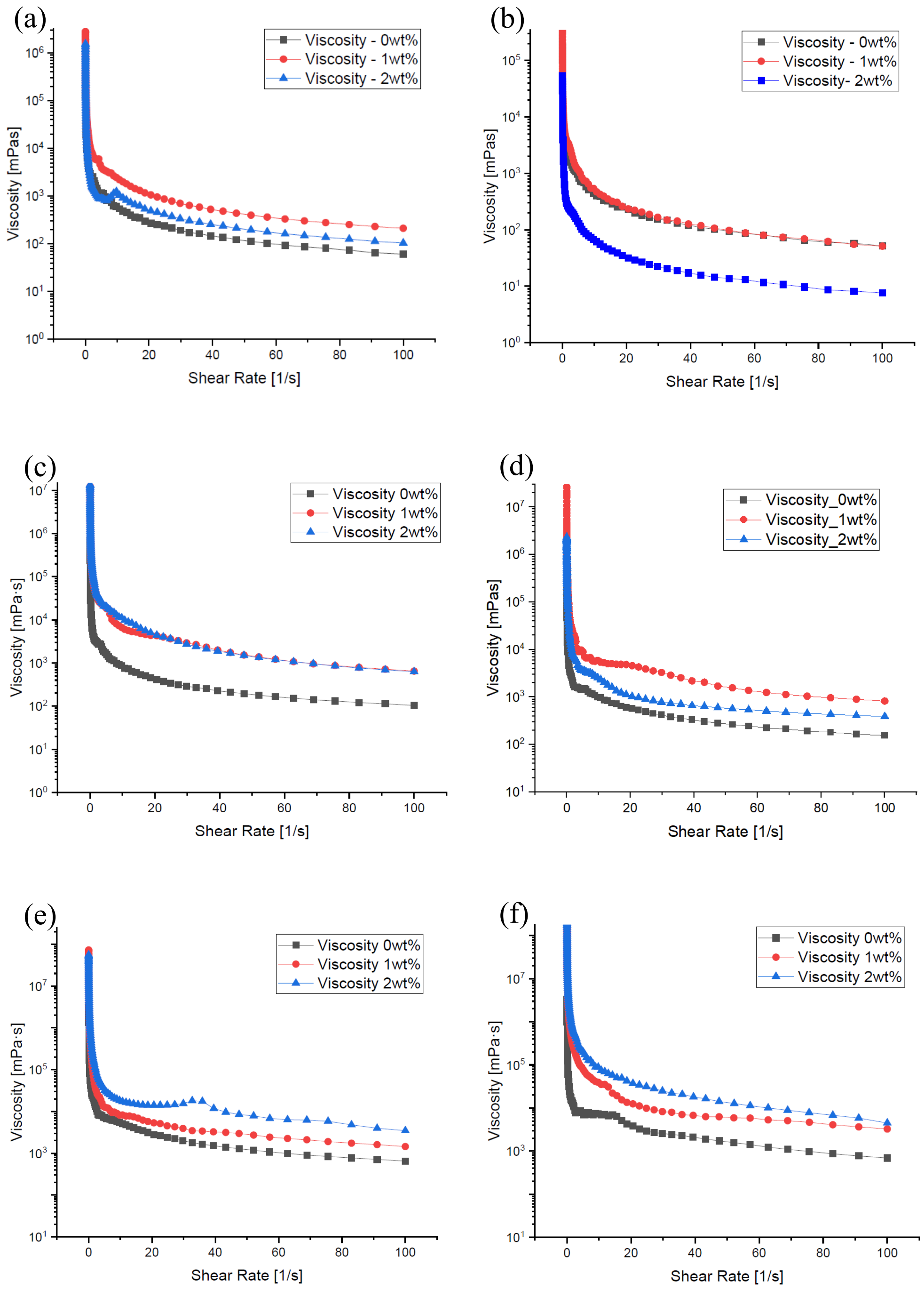
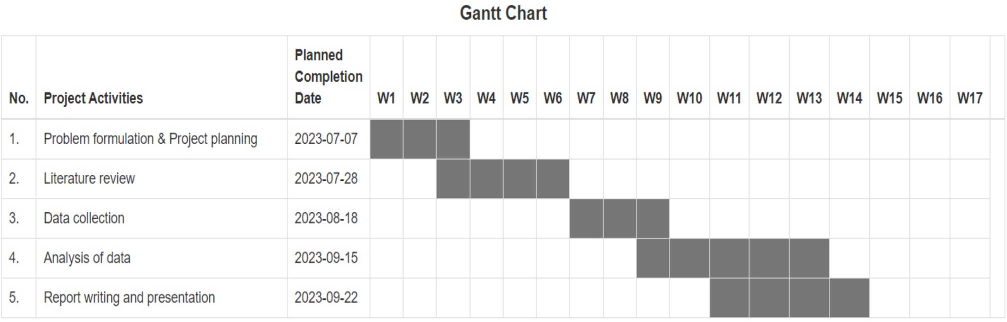


Figure B.1: Graphs of Hydration of MgO after (a) Day1, (b) Day1, (c) Day7, (d) Day14, (e) Day30, (f) Day60.

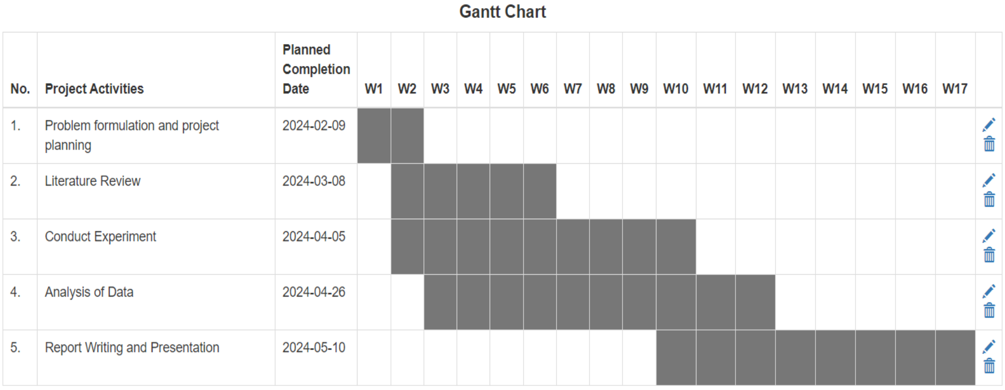
Appendix C: Gannt chart for FYP1 and FYP2.



Gantt Chart

No.	Project Activities	Planned Completion Date	W1	W2	W3	W4	W5	W6	W7	W8	W9	W10	W11	W12	W13	W14	W15	W16	W17
1.	Problem formulation & Project planning	2023-07-07	■	■	■														
2.	Literature review	2023-07-28			■	■	■	■											
3.	Data collection	2023-08-18							■	■	■								
4.	Analysis of data	2023-09-15									■	■	■	■	■				
5.	Report writing and presentation	2023-09-22											■	■	■	■			

Figure C.1: Gannt Chart for FYP1.



Gantt Chart

No.	Project Activities	Planned Completion Date	W1	W2	W3	W4	W5	W6	W7	W8	W9	W10	W11	W12	W13	W14	W15	W16	W17	
1.	Problem formulation and project planning	2024-02-09	■	■																✎ 🗑
2.	Literature Review	2024-03-08		■	■	■	■	■												✎ 🗑
3.	Conduct Experiment	2024-04-05		■	■	■	■	■	■	■	■	■								✎ 🗑
4.	Analysis of Data	2024-04-26			■	■	■	■	■	■	■	■	■	■						✎ 🗑
5.	Report Writing and Presentation	2024-05-10										■	■	■	■	■	■	■	■	✎ 🗑

Figure C.2: Gannt Chart for FYP2.