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Study of the Crystal Structure of CaO/SiO_2, MgO/SiO_2, and CaO–MgO/SiO_2 Catalysts

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ABSTRACT

This study aims to analyze the crystal structure characteristics and phase composition of the CaO/SiO_2, MgO/SiO_2, and CaO-MgO/SiO_2 catalysts, and to synthesize SiO_2 from Ende Flores Natural Zeolite using the hydrothermal and sol-gel methods. CaO was synthesized from Kupang NTT hard water lime deposits, refluxed with HCl, and titrated with Na_2CO_3, then calcined at 1000 ºC to obtain CaO. MgO was synthesized from $MgCO_3$ calcined at 800 °C. The synthesis of each type of catalyst, CaO/SiO_2, MgO/SiO_2, and CaO-MgO/SiO_2, was carried out using the impregnation method. The XRD characterization results show that, in SiO_2, the tridymite phase with a triclinic crystal structure is formed. CaO forms the Lime phase with a cubic crystal structure, while the MgO catalyst shows the Periclase phase and also has a cubic crystal structure. The CaO/SiO_2 catalyst forms a single phase, namely Larnite (Ca_2SiO_4), with a monoclinic crystal structure. The MgO/SiO_2 catalyst formed three phases, namely the dominant Periclase phase (MgO) with a cubic crystal structure, the Olivine phase (Mg_2SiO_4) with an orthorhombic crystal structure, and the Tridymite phase (SiO_2) with a cubic crystal structure. The CaO-MgO/SiO_2 catalyst forms two phases, namely the dominant phase Olivine (Mg_2SiO_4) with an orthorhombic crystal structure and the Portlandite phase (CaH_2O_2) with a trigonal crystal structure.

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INTRODUCTION

Catalysts play a crucial role in chemical reactions, as they are compounds capable of accelerating reaction rates without undergoing permanent chemical changes [1]. Catalysts function by providing active sites that can adsorb reactants and reduce activation energy, thereby enabling reactions to proceed more rapidly. Due to their essential function, the application of catalysts has become a fundamental requirement in various industrial processes. In general, catalysts are classified into two categories: homogeneous and heterogeneous catalysts. Homogeneous catalysts offer the advantage of effective interaction with reactants; however, they present significant challenges in product separation [2]. Consequently, heterogeneous catalysts have emerged as a more effective alternative because they can be easily separated from the reaction products and reused [3].

CaO and MgO-based catalysts are among the most widely developed heterogeneous basic catalysts due to their abundant availability, low cost, and relatively high catalytic activity. CaO exhibits strong basicity, making it highly effective for transesterification reactions, whereas MgO demonstrates superior thermal stability despite having lower basic strength than CaO. However, the direct application of CaO and MgO still faces a major limitation, namely the leaching of Ca^{2+}

and Mg^{2+} ions from the solid phase into the liquid phase during the reaction process [4]. This phenomenon is caused by interactions between the catalyst and alcohols, such as methanol, which can partially dissolve the active phase. As a result, the number of active sites decreases, catalyst deactivation occurs, and biodiesel yield is reduced [5]. Furthermore, leaching may transform the catalytic system into a semi-homogeneous system, thereby diminishing the inherent advantages of heterogeneous catalysts.

To address this limitation, one widely explored approach is the impregnation of catalysts onto supporting materials [4]. Silica (SiO_2) is one of the most commonly used supports because of its high thermal stability, large surface area, and porous structure, which enables uniform dispersion of the active phase. In addition, the presence of silanol groups on the SiO_2 surface facilitates interactions between the support and the catalyst, thereby strengthening metal–oxide bonding and reducing the likelihood of leaching [5]. Thus, the role of SiO_2 extends beyond merely serving as a physical support; it also enhances the chemical stability and active-site distribution of the catalyst.

Although numerous studies have reported the application of CaO/SiO_2 and MgO/SiO_2 catalysts, comprehensive comparative studies involving these systems alongside the $CaO–MgO/SiO_2$ catalyst system remain limited. Several studies have indicated that the combination of CaO and MgO may improve catalytic performance through a synergistic effect, integrating the high basicity of CaO with the thermal stability of MgO [4]. Moreover, CaO–MgO-based catalysts have been reported to exhibit superior catalytic activity compared with single-component catalysts due to interactions between the active phases that enhance reaction efficiency [5]. However, the relationships among crystal structure, phase distribution, and catalyst stability within this system remain insufficiently understood, highlighting the need for further investigation.

Characterization of the crystal structure of catalysts is an important aspect in identifying crystalline phases, composition, and lattice parameters associated with the physical and chemical properties of catalysts. This analysis is commonly conducted using X-ray diffraction (XRD) [3]. Through crystal structure characterization, the relationships among atomic arrangement, phase distribution, and crystallite size with catalytic activity and stability can be elucidated [6]. Such information is essential for optimizing catalyst performance, particularly in improving stability and minimizing leaching. Therefore, this study aims to investigate the crystal structures of CaO/SiO_2, MgO/SiO_2, and $CaO–MgO/SiO_2$ catalysts. One of the objectives of this research is to provide insights into the crystal structural characteristics of these materials.

RESEARCH METHODS

Time and Location of the Study

This study was conducted from February to June 2025 at the Chemistry Education Laboratory, Faculty of Teacher Training and Education, Universitas Nusa Cendana. The characterization analysis using X-ray diffraction (XRD) was carried out at the Center for Minerals and Advanced Materials, Universitas Negeri Malang.

Research Instruments and Materials

The equipment used in this study included glassware, a thermometer, a 200-mesh sieve, a porcelain mortar, an oven, stainless steel plates, a desiccator, a reflux flask, an Ohaus balance, an oven column, a magnetic stirrer, filter paper, a digital balance (Denver), a water pump, a calcination and reduction reactor, an analytical balance, a Buchner funnel, a stopwatch, a pressure cooker, a smart mixer, a hot plate, aluminum foil, screw clamps, titration apparatus, and an X-ray diffraction (XRD) instrument. The XRD instrument consisted of three main components: an X-ray source, a sample holder/goniometer, and an XRD detector.

Experimental Design

The diffractograms obtained from the XRD analysis were processed using Match! software version 4.3 to examine the crystal structure of the catalyst materials. The analysis included determining the crystal systems, lattice parameters, and phase composition of each catalyst material: CaO, MgO, CaO/SiO_2, MgO/SiO_2, and CaO–MgO/SiO_2.

Experimental Procedure

Treatment of Natural Zeolite from Ende, Flores (ZAEF)

Natural zeolite from Ende, Flores, was sorted, and light green-colored zeolite samples were selected. The selected zeolite was washed, crushed into fine particles, passed through a 200-mesh sieve, and weighed to obtain 500 g of sample. The zeolite was then immersed in deionized water and stirred with a glass rod for 30 minutes at room temperature, followed by standing for 2 hours. Subsequently, the filtrate was separated from the sediment.

The zeolite was then subjected to hydrothermal treatment. ZAEF was placed in a pressure cooker and mixed with deionized water. The zeolite suspension was heated at 100 °C for approximately 3 hours and then allowed to stand for 24 hours. The hydrothermally treated ZAEF was filtered and transferred into a plastic container. Deionized water was added until the container was half full, and the mixture was stirred with a glass rod for 1 hour. After stirring, the ZAEF was allowed to stand for 24 hours. This cleaning procedure was repeated using the same treatment until the ZAEF became clean and exhibited a bright green color. Finally, the treated ZAEF was dried in an oven at 120 °C for 6 hours to obtain purified natural zeolite from Ende, Flores.

Synthesis of SiO_2

A total of 250 g of purified ZAEF was refluxed with 750 mL of 8 M NaOH solution for 24 hours, followed by filtration to obtain a sodium silicate solution. The filtrate was then titrated with 2 M HCl to pH 7 under continuous stirring using a magnetic stirrer until a white precipitate formed. The resulting white gel was aged at room temperature for 24 hours, then filtered and washed four times with 500 mL of deionized water to remove chloride salts, as confirmed by testing with 1% $AgNO_3$ solution.

The neutral precipitate was separated by filtration using Whatman No. 42 filter paper. The resulting solid was dried at 110 °C for 12 hours until a constant mass was achieved. After cooling, the solid was weighed and ground into fine powder. The silica powder was subsequently calcined at 1000 °C for 6 hours. The calcined silica was then cooled, weighed, and finely ground using a porcelain mortar. The prepared silica was used as the support material for CaO and MgO catalysts.

Synthesis of CaO

Hard-water lime originating from Kupang, East Nusa Tenggara, was collected, boiled, and washed with deionized water under stirring using a magnetic stirrer. The material was then dried in an oven until completely dry. The cleaned lime was sieved through a 140–200 mesh sieve. A total of 100 g of the sieved material was weighed using a digital balance and refluxed with 500 mL of 3 M HCl solution while continuously stirred with a magnetic stirrer until complete dissolution occurred.

The resulting solution was filtered and subsequently titrated with 1.2 M Na_2CO_3 solution until the reaction was complete, resulting in the formation of $CaCO_3$ precipitate. The precipitate was separated from the solution and washed with deionized water until free from Cl^- ions. The precipitate was then dried in an oven at 60 °C for 6 hours. The dried $CaCO_3$ solid was calcined at 1000 °C for 6 hours to obtain a CaO catalyst. The prepared CaO catalyst was weighed and stored in a tightly sealed container until further use.

Synthesis of MgO

A total of 200 g of $MgCO_3$ was weighed and calcined at 800 °C for 6 hours. After cooling, the resulting MgO was weighed, placed in a sealed container, and stored for further use.

Synthesis of CaO/SiO_2

The CaO/SiO_2-15% catalyst was prepared by gradually adding 21.25 g of CaO into 250 mL of deionized water to form a $Ca(OH)_2$ suspension. The catalyst composition consisted of 85% CaO and 15% SiO_2. Subsequently, 3.75 g of SiO_2 was added to the suspension, and the mixture was continuously stirred using a magnetic stirrer at 400 rpm for 4 hours.

The suspension was then subjected to ultrasonic treatment for 3 hours, with intermittent manual stirring every 10 minutes. After ultrasonic treatment, the mixture was allowed to stand for 24 hours to facilitate the deposition of $Ca(OH)_2$ onto the SiO_2 support. The water solvent was removed by drying the suspension in an oven at 110 °C for 24 hours until completely dry. The resulting powder was then calcined at 900 °C for 6 hours. After cooling, the prepared catalyst was stored in a tightly sealed container for further use.

Synthesis of MgO/SiO_2

The MgO/SiO_2 catalyst was prepared by gradually adding 21.25 g of MgO into 250 mL of deionized water to form an $Mg(OH)_2$ suspension. The catalyst composition consisted of 85% MgO and 15% SiO_2. Subsequently, 3.75 g of SiO_2 was added to the suspension, and the mixture was continuously stirred using a magnetic stirrer at 400 rpm for 4 hours.

The suspension was then subjected to ultrasonic treatment for 3 hours, with intermittent manual stirring every 10 minutes. After ultrasonic treatment, the mixture was allowed to stand for 24 hours to facilitate the deposition of $Mg(OH)_2$ onto the SiO_2 support. The water solvent was subsequently removed by drying the suspension in an oven at 110 °C for 24 hours until completely dry. The resulting powder was then calcined at 900 °C for 6 hours. After cooling, the prepared catalyst was stored in a tightly sealed container for further use.

Synthesis of $CaO–MgO/SiO_2$

The $CaO–MgO/SiO_2$ catalyst was prepared by gradually adding 10.625 g of MgO and 10.625 g of CaO into 250 mL of deionized water to form $Mg(OH)_2$ and $Ca(OH)_2$ suspensions. The catalyst composition consisted of 42.5% MgO, 42.5% CaO, and 15% SiO_2. Subsequently, 3.75 g of SiO_2 was added to the mixed suspension, and the mixture was continuously stirred using a magnetic stirrer at 400 rpm for 4 hours.

The suspension was then subjected to ultrasonic treatment for 3 hours, with intermittent manual stirring every 10 minutes. After ultrasonic treatment, the mixture was allowed to stand for 24 hours to facilitate the deposition of the hydroxide precipitates onto the SiO_2 support. The water solvent was removed by drying the suspension in an oven at 110 °C for 24 hours until completely dry. The resulting powder was then calcined at 900 °C for 6 hours. After cooling, the prepared catalyst was stored in a tightly sealed container and was ready for characterization.

RESULTS AND DISCUSSION

Catalyst Characterization Using X-Ray Diffraction (XRD)

Catalyst characterization using X-ray diffraction (XRD) was conducted to determine the crystal structure of the catalysts, including crystalline phases and phase composition, crystal system, and lattice parameters. XRD analysis was performed on catalyst samples with different SiO_2 compositions. The analysis was carried out by placing the catalyst samples in the sample holder and irradiating them with X-rays over a 2θ scanning range of 10°–90°, using a copper (Cu) X-ray source with a $K\alpha_1$ wavelength of 1.54 Å [7]. The resulting data were presented as diffractograms showing the relationship between the diffraction angle (2θ) and X-ray intensity.

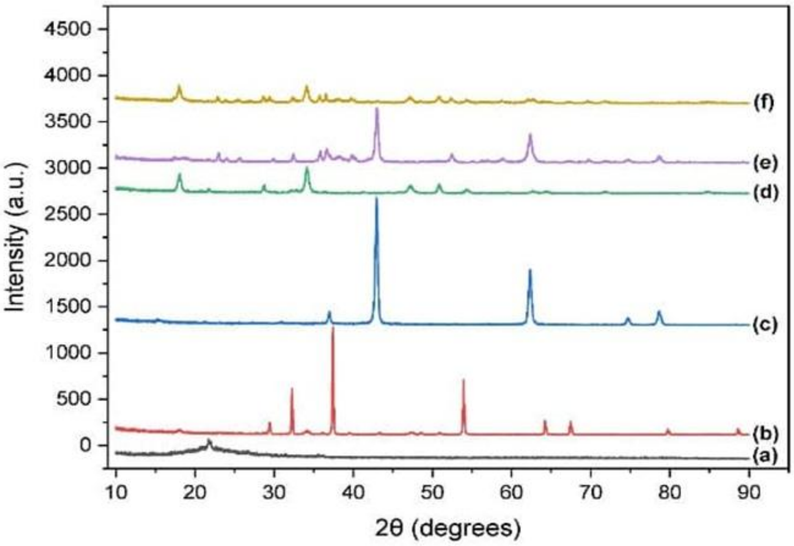

Figure 1. Diffractograms of (a) SiO_2, (b) CaO, (c) MgO, (d) CaO/SiO_2, (e) MgO/SiO_2, and (f) $CaO–MgO/SiO_2$.

SiO_2

The XRD diffractogram of the SiO_2 catalyst is presented in Figure 2. The X-ray diffraction (XRD) analysis revealed that the mineral phase present in the SiO_2 sample was identified as tridymite. This phase corresponds to silicon dioxide (SiO_2), commonly known as silica, as referenced in the ICSD database. Diffraction peaks were detected at 2θ angles of 21.76° and 21.82°, corresponding to the crystal plane orientations of (040) and (220), respectively. Based on the analysis performed using Match! Software version 4, the tridymite phase was identified as having a triclinic crystal system with lattice parameters of a = 9.9320 Å, b = 17.2160 Å, and c = 81.8640 Å, with lattice angles of $\alpha = \beta = \gamma = 90°$. The phase exhibited a calculated density of 2.28100 g/cm³.

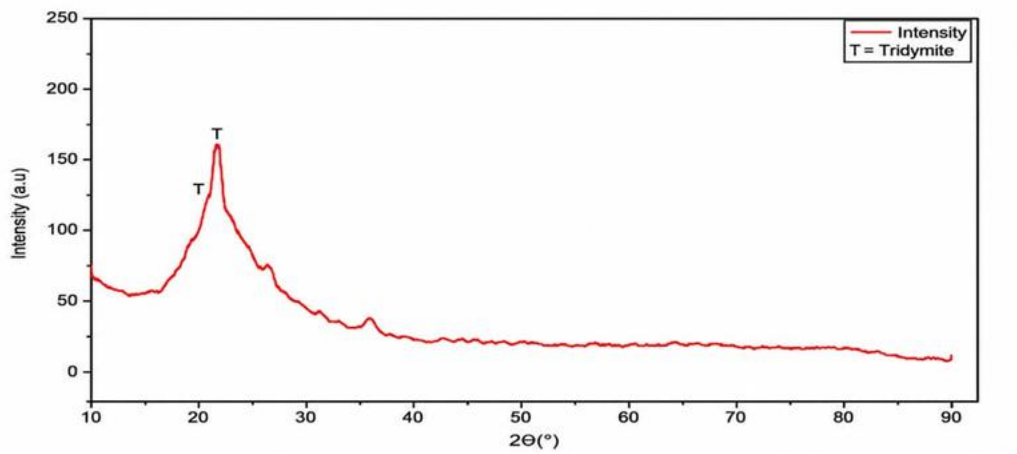

Figure 2. XRD diffractogram of SiO_2.

The formation of the tridymite phase in the SiO_2 sample indicates that the silica material underwent a structural transformation from an amorphous phase to a more ordered crystalline phase. Tridymite generally forms under high-temperature conditions, suggesting that the synthesis process or thermal treatment applied was sufficient to promote the recrystallization of the silica structure.

These findings are consistent with previous studies reporting that the formation of the tridymite phase is strongly influenced by high temperature, holding time, and the initial crystallinity of the material [8]. Those studies demonstrated that increasing synthesis temperature directly enhances tridymite crystal growth, as identified through XRD analysis. In addition, other studies have reported that tridymite is a high-temperature stable polymorph of SiO_2, commonly observed in materials subjected to extreme thermal treatment. This has been described in studies on silica-based refractory materials, where the presence of tridymite was detected after heating at temperatures ranging from 900 to 1500 °C using XRD analysis [9].

The formation of the tridymite phase has important implications for the catalytic properties of the material, particularly in terms of improved thermal stability and structural strength. Crystalline phases such as tridymite generally exhibit higher stability than amorphous phases, making the catalyst more resistant to high temperatures during catalytic reactions. However, increased crystallinity may also result in a reduction in specific surface area, as the structure becomes more ordered and densely packed, potentially decreasing the number of available active sites.

Based on the diffraction pattern obtained, the characteristic SiO_2 peaks were detected at 2θ values of 21.76° and 21.82°, corresponding to the (040) and (220) crystal planes, respectively. The very small angular difference between these two peaks suggests a possible peak shift, which may indicate slight changes in lattice parameters or the presence of internal strain within the tridymite crystal structure. This shift may have been induced by the synthesis process or thermal treatment, which could have triggered structural reorganization within the silica crystal framework, resulting in minor changes in interplanar spacing.

CaO

The XRD diffractogram of the CaO catalyst is presented in Figure 3. The X-ray diffraction (XRD) analysis revealed that the mineral phase present in the CaO sample was identified as the lime phase. According to the ICSD database, this phase corresponds to calcium oxide (CaO). Diffraction peaks were observed at 2θ values of 32.24°, 32.32°, 37.39°, 37.49°, and 53.89°, corresponding to the crystal plane orientations of (111), (200), (202), (311), and (222), respectively. Based on the analysis performed using Match! Software version 4, the lime phase was identified as having a cubic crystal system with lattice parameters of a = b = c = 4.8110 Å and lattice angles of α = β = γ = 90°. The phase exhibited a calculated density of 2.92700 g/cm³.

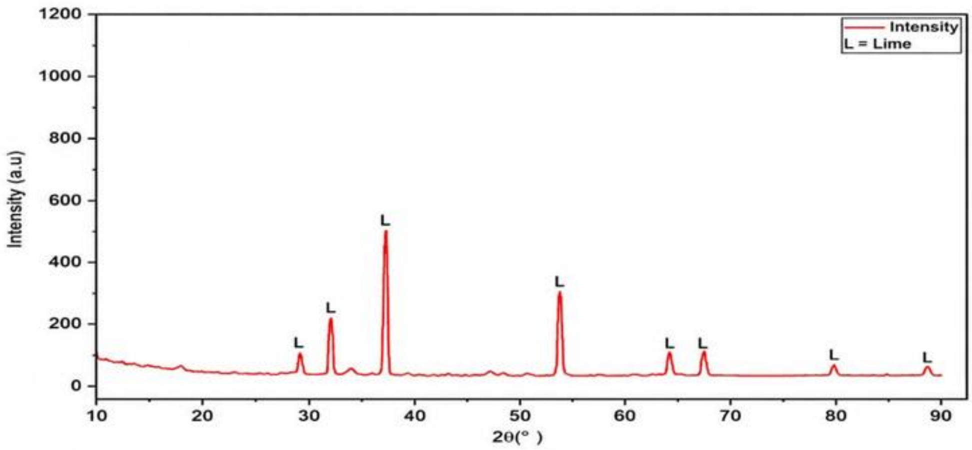

Figure 3. XRD diffractogram of CaO.

The identification of the lime phase (CaO) in the sample indicates that the material successfully underwent an effective calcination process, during which precursor compounds such as $CaCO_3$ were decomposed into CaO. The appearance of sharp diffraction peaks suggests that the resulting CaO possesses a high degree of crystallinity with a stable cubic crystal structure, indicating that the thermal treatment applied was sufficient to optimally form the catalytically active phase.

These findings are consistent with previous studies reporting that calcined CaO exhibits characteristic diffraction patterns associated with a cubic crystal structure, with crystallinity increasing as calcination temperature rises [10]. In addition, other studies have demonstrated that the crystalline characteristics of CaO, including crystal size and diffraction peak intensity, are strongly influenced by thermal treatment conditions and play a significant role in determining the material properties [11].

The formation of the CaO phase has important implications for the catalytic properties of the material, particularly in terms of enhanced basicity and catalytic activity. CaO is a heterogeneous basic catalyst containing active O^{2-} ion sites that facilitate catalytic reactions. A well-defined crystalline structure contributes to improved thermal stability; however, excessive crystal growth may reduce the specific surface area, thereby decreasing the number of accessible active sites.

Based on the diffraction pattern, characteristic CaO peaks were observed at 2θ values of 32.24° and 32.32°, as well as 37.39° and 37.49°, corresponding to the (111), (200), (202), and (311) crystal planes, respectively. The close proximity of these diffraction peaks may indicate subtle variations in lattice parameters or the presence of internal strain within the CaO crystal structure. Such variations may arise from non-uniform calcination conditions, which can lead to slight lattice distortions within the crystalline framework.

MgO

The XRD diffractogram of the MgO catalyst is presented in Figure 4. The X-ray diffraction (XRD) analysis revealed that the mineral phase present in the MgO sample was identified as the periclase phase. According to the ICSD database, this phase corresponds to magnesium oxide (MgO). Diffraction peaks were detected at 2θ values of 36.91°, 37.00°, 42.89°, 43.00°, and 62.27°, corresponding to the crystal plane orientations of (111), (200), (202), (311), and (222), respectively.

Based on the analysis performed using Match! Software version 4, the periclase phase was identified as having a cubic crystal system with lattice parameters of a = b = c = 4.2160 Å and lattice angles of $\alpha = \beta = \gamma = 90°$. The phase exhibited a calculated density of 3.57200 g/cm³.

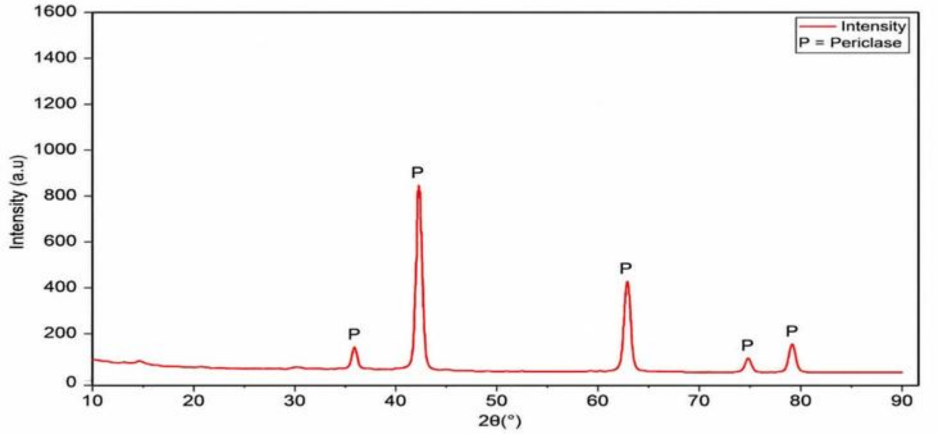

Figure 4. XRD diffractogram of MgO.

The formation of the periclase phase (MgO) indicates that the material underwent an effective calcination or thermal treatment process, resulting in the formation of a stable crystalline MgO structure. This phase represents the most thermodynamically stable crystalline form of magnesium oxide at elevated temperatures, and its presence indicates the successful transformation of the precursor into a pure oxide phase [12]. These findings are consistent with previous studies reporting that MgO typically crystallizes in a cubic periclase structure, characterized by prominent diffraction peaks associated with the (111), (200), and (220) crystal planes, which commonly appear at 2θ values in the ranges of approximately 36°–43° and 62° [13].

The formation of the periclase phase has important implications for the catalytic properties of the material, particularly in terms of enhanced basicity, as MgO is widely recognized as a strong heterogeneous basic catalyst. The stable cubic crystal structure also contributes to improved thermal stability and provides active surface sites that play a significant role in catalytic reactions, particularly those involving base-catalyzed mechanisms such as transesterification [14].

Based on the diffraction pattern, characteristic MgO (periclase) peaks were observed at 2θ values of 36.91° and 37.00°, as well as 42.89° and 43.00°, corresponding to the (111), (200), (202), and (311) crystal planes, respectively. The close proximity of these diffraction peaks may suggest subtle variations in lattice parameters or the presence of internal strain within the MgO crystal structure. Such variations may arise from non-uniform calcination conditions or the presence of crystal defects, which could lead to slight lattice distortions in the formed periclase phase.

CaO/SiO₂

The XRD diffractogram of the CaO/SiO₂ catalyst is presented in Figure 5. The X-ray diffraction (XRD) analysis revealed that the mineral phase present in the CaO/SiO₂ sample consisted of a single phase, identified as larnite. According to the ICSD database, this phase corresponds to calcium silicate (Ca_2SiO_4). Diffraction peaks were detected at 2θ values of 18.03°, 31.92°, 32.01°, 33.20°, and 34.20°, corresponding to the crystal plane orientations of (011), (10-1), (002), (101), and (110), respectively. Based on the analysis performed using Match! software version 4, the larnite phase was identified as having a monoclinic crystal system with lattice parameters of a = 5.50770 Å, b = 6.75050 Å, and c = 9.34080 Å, with lattice angles of $\alpha = \gamma = 90°$ and $\beta = 94.596°$. The phase exhibited a calculated density of 3.30500 g/cm³.

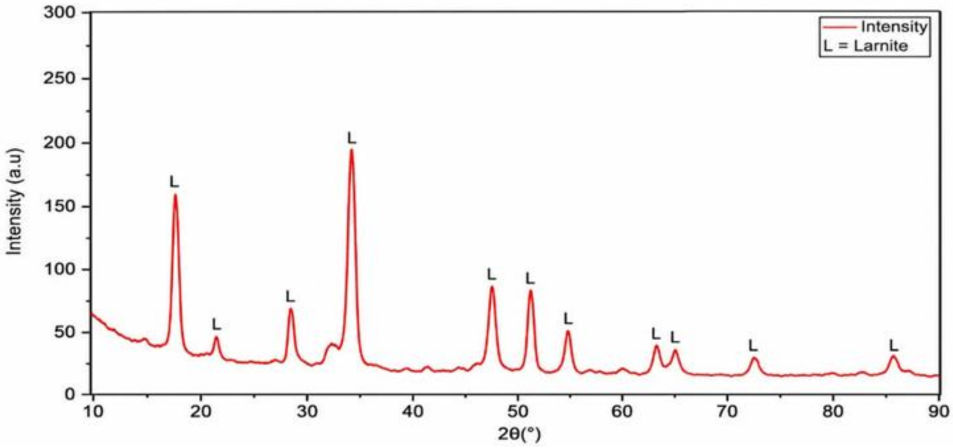

Figure 5. XRD diffractogram of CaO/SiO$_2$.

The formation of the larnite phase (Ca$_2$SiO$_4$) indicates that chemical interactions occurred between CaO and SiO$_2$ during the synthesis or calcination process, resulting in the formation of a thermally stable calcium silicate compound. This phase is commonly formed at elevated temperatures through a solid-state reaction between CaO and SiO$_2$, indicating the successful synthesis of a homogeneous composite material [15]. These findings are consistent with previous reports indicating that Ca$_2$SiO$_4$ (larnite) exhibits a monoclinic crystal structure with characteristic diffraction peaks within the 2θ range of approximately 18°–35° [16].

The formation of the larnite phase has important implications for the catalytic properties of the material, particularly through the combination of the basic properties of CaO and the supporting characteristics of SiO$_2$, resulting in improved thermal stability and a more uniform distribution of active sites. Ca$_2$SiO$_4$ has also been reported to exhibit favorable catalytic activity in base-catalyzed reactions, such as transesterification, due to the presence of Ca^{2+} ions that contribute to catalytic activity [17].

Based on the diffraction pattern, characteristic Ca$_2$SiO$_4$ (larnite) peaks were observed at 2θ values of 31.92° and 32.01°, as well as 33.20° and 34.20°, corresponding to the (10-1), (002), (101), and (110) crystal planes, respectively. The close proximity of these diffraction peaks may indicate subtle variations in lattice parameters or the presence of internal strain within the Ca$_2$SiO$_4$ crystal structure. Such variations may arise from interactions between CaO and SiO$_2$ during the calcination process, which may not have occurred entirely homogeneously, thereby leading to slight lattice distortions in the formed larnite phase.

MgO/SiO$_2$

The XRD diffractogram of the MgO/SiO$_2$ catalyst is presented in Figure 6. The X-ray diffraction (XRD) analysis revealed that the mineral phases identified in the MgO/SiO$_2$ sample consisted of three major phases, namely periclase, olivine, and tridymite. The phase composition analysis indicated that periclase accounted for 50.6% of the sample, followed by olivine at 48.7% and tridymite at 0.8%.

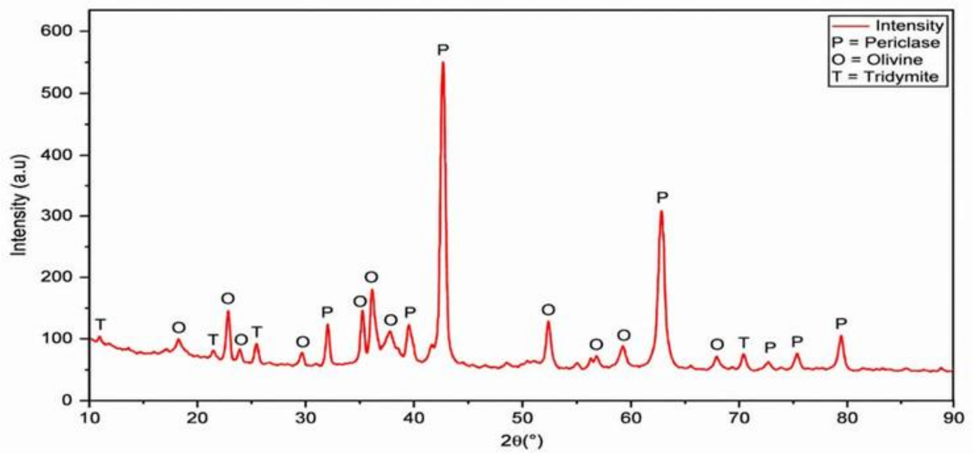

Figure 6. XRD diffractogram of MgO/SiO_2.

The periclase phase, identified in the ICSD database as magnesium oxide (MgO), was detected at 2θ values of 36.74°, 42.91°, 43.02°, 62.27°, and 62.24°, corresponding to the crystal plane orientations of (111), (200), (202), (311), and (222), respectively. Based on the analysis performed using Match! software version 4, the periclase phase was identified as having a cubic crystal system with lattice parameters of $a = b = c = 4.2160$ Å and lattice angles of $\alpha = \beta = \gamma = 90°$, with a calculated density of 3.57200 g/cm³.

The olivine phase, identified in the ICSD database as magnesium silicate (Mg_2SiO_4), was observed at 2θ values of 17.45°, 22.94°, 23.96°, 25.57°, and 25.63°, corresponding to the crystal plane orientations of (020), (110), (021), (101), and (111), respectively. Based on the analysis using Match! software version 4, the olivine phase exhibited an orthorhombic crystal system with lattice parameters of $a = 4.7400$ Å, $b = 10.1985$ Å, and $c = 5.9792$ Å, with lattice angles of $\alpha = \beta = \gamma = 90°$, and a calculated density of 3.22700 g/cm³.

The tridymite phase, identified in the ICSD database as silicon dioxide (SiO_2), commonly known as silica, was detected at 2θ values of 10.17°, 21.67°, 23.00°, 23.96°, and 24.02°, corresponding to the crystal plane orientations of (211), (202), (321), (400), and (402), respectively. Based on the analysis performed using Match! software version 4, the tridymite phase was identified as having a cubic crystal system with lattice parameters of $a = b = c = 20.97788$ Å and lattice angles of $\alpha = \beta = \gamma = 90°$, with a calculated density of 1.03700 g/cm³.

The dominance of the periclase and olivine phases suggests that the interaction between MgO and SiO_2 proceeded effectively, resulting in the formation of magnesium silicate (Mg_2SiO_4), while residual unreacted MgO remained in the periclase phase. The presence of the tridymite phase indicates that a small fraction of free SiO_2 underwent recrystallization during the calcination process.

The formation of these three phases indicates that relatively complex interactions occurred between MgO and SiO_2 during the synthesis process. The periclase phase suggests that a portion of MgO remained in the form of free oxide, whereas the formation of the olivine phase (Mg_2SiO_4) confirms the occurrence of a high-temperature solid-state reaction between MgO and SiO_2, resulting in the formation of thermally stable magnesium silicate. The presence of the tridymite phase indicates that part of the SiO_2 transformed from an amorphous phase to a crystalline phase due to thermal treatment [18]. These findings are consistent with previous reports indicating that MgO can react with SiO_2 at elevated temperatures to form Mg_2SiO_4 (olivine), while SiO_2 may transform into crystalline phases such as tridymite under similar conditions.

The formation of these three phases has significant implications for the catalytic properties of the material. The periclase phase (MgO) serves as an important active basic site in catalytic reactions. The olivine phase (Mg_2SiO_4) contributes to the thermal stability and mechanical strength of the material, although its basicity is generally lower than that of pure MgO. Meanwhile, the tridymite phase (SiO_2) functions as a support material that may enhance surface area and promote a more uniform distribution of active sites [18].

Based on the diffraction pattern, characteristic MgO (periclase) peaks were observed at 2θ values of 42.91° and 43.02°, as well as 62.24° and 62.27°, corresponding to the (200), (202), (311), and (222) crystal planes, respectively. Similarly, the Mg_2SiO_4 (olivine) phase exhibited closely spaced peaks at 2θ values of 25.57° and 25.63°, corresponding to the (101) and (111) crystal planes. The SiO_2 (tridymite) phase also showed adjacent peaks at 2θ values of 23.96° and 24.02°, corresponding to the (400) and (402) crystal planes. The proximity of these diffraction peaks may suggest subtle variations in lattice parameters or the presence of internal strain within the crystal structures. Such variations may result from interactions between MgO and SiO_2 during synthesis and calcination that may not have occurred entirely homogeneously, thereby leading to slight lattice distortions in the formed periclase, olivine, and tridymite phases.

CaO-MgO/SiO_2

The X-ray diffraction (XRD) analysis revealed that the mineral phases identified in the CaO–MgO/SiO_2 sample consisted of two phases, namely olivine and portlandite, with phase compositions of 60.7% and 39.3%, respectively.

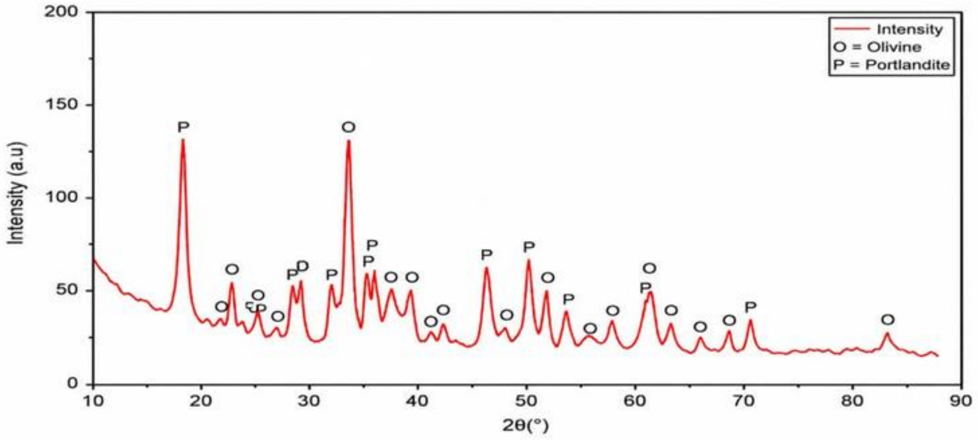

Figure 7. XRD diffractogram of CaO–MgO/SiO_2.

The olivine phase, identified in the ICSD database as magnesium silicate (Mg_2SiO_4), was detected at 2θ values of 22.90°, 22.96°, 29.53°, 32.33°, and 32.41°, corresponding to the crystal plane orientations of (020), (110), (021), (101), and (111), respectively. Based on the analysis performed using Match! software version 4, the olivine phase was identified as having an orthorhombic crystal system with lattice parameters of a = 4.74900 Å, b = 10.19850 Å, and c = 5.97920 Å, with lattice angles of $\alpha = \beta = \gamma = 90°$. The calculated crystal density was 3.22700 g/cm^3.

The portlandite phase, identified in the ICSD database as calcium hydroxide ($Ca(OH)_2$), was observed at 2θ values of 18.06°, 28.68°, 34.08°, 34.16°, and 36.63°, corresponding to the crystal plane orientations of (001), (100), (101), (002), and (102), respectively. Based on the analysis performed using Match! software version 4, the portlandite phase exhibited a trigonal crystal system with lattice parameters of a = b = 3.59250 Å and c = 4.90500 Å. The lattice angles were α =

β = 90°, while γ was consistent with the trigonal crystal symmetry. The calculated crystal density was 2.24400 g/cm^3.

The dominance of the olivine phase suggests that a solid-state reaction occurred between MgO and SiO_2, resulting in the formation of thermally stable magnesium silicate. Meanwhile, the presence of the portlandite phase indicates that residual CaO likely reacted with moisture to form $Ca(OH)_2$ during the cooling process or after calcination.

The formation of the olivine phase confirms that interactions between MgO and SiO_2 occurred during the synthesis and calcination processes, leading to the formation of a thermally stable magnesium silicate compound. In contrast, the formation of the portlandite phase indicates partial hydration of CaO into $Ca(OH)_2$ due to exposure to water vapor or ambient moisture during synthesis or sample storage. The presence of these two phases suggests that the synthesis process resulted not merely in a physical mixture, but also in chemical transformations among the constituent components of the material. These findings are consistent with previous reports indicating that the olivine phase commonly forms in MgO–SiO_2 systems following high-temperature thermal treatment, whereas portlandite frequently appears as a result of CaO hydration [19].

Based on the diffraction pattern, the olivine phase exhibited closely spaced diffraction peaks at 2θ values of 22.90° and 22.96°, as well as 32.33° and 32.41°, corresponding to the (020), (110), (101), and (111) crystal planes, respectively. Meanwhile, the portlandite phase showed adjacent peaks at 2θ values of 34.08° and 34.16°, corresponding to the (101) and (002) crystal planes. The close proximity of these diffraction peaks may indicate subtle variations in lattice parameters or the presence of internal strain within the crystal structures [20]. Such variations may arise from interactions among CaO, MgO, and SiO_2 during synthesis and calcination that may not have occurred entirely homogeneously, thereby leading to slight lattice distortions in the formed olivine and portlandite phases.

Table 1. XRD analysis data of SiO_2, CaO, MgO, CaO/SiO_2, MgO/SiO_2, and CaO–MgO/SiO_2 samples.

Sample	Phase	Crystal Structure	Lattice Parameters	Phase Composition
SiO_2	Tridymite	Triclinic	a=9,9320 Å b=17,21600 Å c=81,86400 Å	100%
CaO	Lime	Cubic	a=b=c= 4,8110 Å	100%
MgO	Periclase	Cubic	a=b=c= 4,21600 Å	100%
CaO/SiO_2	Larnite	Monoclinic	a=5,50770 Å b=6,75050 Å c=9,34080 Å	100%
MgO/SiO_2	Periclase	Cubic	a=b=c= 4,21600 Å	50,6%
	Olivine	Orthorhombic	a=4,74000 Å b=10,19850Å c=5,97920 Å	48,7%
	Tridymite	Cubic	a=b=c= 20,97788 Å	0,8%

CaO-MgO/SiO_2	Olivine	Orthorhombic	a= 4,74900 Å b=10,19850 Å c= 5,97920 Å	60,7%
	Portlandite	Trigonal	a= b=3,59250 Å c= 4,90500 Å	39,3%

CONCLUSION

Based on the X-ray diffraction (XRD) characterization results, the SiO_2, CaO, MgO, CaO/SiO_2, MgO/SiO_2, and CaO–MgO/SiO_2 samples successfully formed distinct crystalline phases depending on their composition and synthesis conditions. The SiO_2 sample formed the tridymite phase, CaO formed the lime phase, and MgO formed the periclase phase, indicating the successful calcination process and the formation of stable crystalline structures. In the composite materials, the larnite phase was identified in CaO/SiO_2, while olivine, periclase, tridymite, and portlandite phases were detected in MgO/SiO_2 and CaO–MgO/SiO_2, confirming the occurrence of chemical interactions among the oxide components during the synthesis process. The formed phases significantly influenced the catalytic properties of the materials, particularly in enhancing thermal stability, basicity, and the distribution of active sites. In addition, the presence of closely spaced diffraction peaks suggests subtle variations in lattice parameters and possible internal crystal strain induced by the synthesis and calcination processes. Overall, the combination of CaO, MgO, and SiO_2 successfully produced heterogeneous catalytic materials with crystalline characteristics that demonstrate potential for application in base-catalyzed reactions.

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