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Valorization of rice husk ash-derived silica as a support material for CaO catalysts: Implications for catalyst characteristics

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Abstract

Rice husk ash (RHA) is a renewable and sustainable source of silica with large surface area and porosity, making it a well-suited support material for catalysts. Likewise, the calcium oxide (CaO)-rich marble waste powder can be employed as a low-cost heterogeneous catalyst for various applications, including 1,4-butanediol dehydration, syngas production, and transesterification. However, pure CaO often experiences leaching and deactivation during catalytic reactions. Incorporating CaO catalyst onto silica derived from RHA offers a promising strategy to improve its stability, particle dispersion, porosity, surface area, and tolerance to free fatty acids. This study aimed to develop a CaO catalyst derived from marble waste powder combined with sulfonated silica synthesized from RHA to enhance particle distribution, reduce crystallinity, and improve surface area. The experimental procedure involved the chemical and thermal activation of SiO_2 from RHA, sulfonation of SiO_2, thermal activation of CaO from marble waste powder, and impregnation of CaO/SiO_2 catalysts with varying CaO-to-SiO_2 ratios (25:75, 50:50, and 75:25 wt.%). The raw materials (SiO_2 and CaO) were characterized using XRF, while the resulting CaO/SiO_2 catalysts characteristics were analyzed by SEM, EDX, XRD, and BET to determine their morphological and structural properties. The results showed that the purity of SiO_2 and CaO obtained through chemical and thermal activation was 93.67% and 99.13%, respectively. The sulfonation process on SiO_2 successfully added $–SO_3$ groups at 36.5%, which supported the formation of acid sites on the catalyst. Characterization showed that the surface morphology was composed of particles measuring 2–8 µm with a dominant amorphous structure. The incorporation of SiO_2 gave rise to new crystal peaks, but reduced crystal intensity, especially at the 50%:50% composition. The composition of CaO/SiO_2 at 75%:25% possessed the best physical properties with a surface area of 22.24 m^2/g, a pore volume of 65.29 mm^3/g, and a pore diameter of 11.74 nm, indicating high potential as a heterogeneous catalyst for various applications.

Keywords: CaO/SiO_2 catalyst; marble waste powder ; rice husk ash; wet impregnation

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INTRODUCTION

Rice husk (RH), a by-product of the rice milling industry, had gained considerable attention as a supplementary material for waste valorization and value addition in industrial and environmental applications. South and Southeast Asia, which together accounted for approximately 90% of global rice production, generated vast quantities of RH, making it an abundant and renewable resource (USDA, 2025). RH primarily consisted of cellulose, lignin, and approximately 20% silica. When subjected to controlled combustion, it yielded high-purity amorphous or crystalline silica (90–97%) depending on the burning conditions. The silica produced can be used as an economical source of silicon oxide and other silica-based materials (Adam *et al.,* 2006). Silica extracted from RH had been widely applied in the production of soluble silicates, reinforcing fillers for rubber and silicone materials, and as catalyst supports synthesized through sol–gel, wet precipitation, ion-exchange, and impregnation techniques (Amutha *et al.,* 2010; Nasir *et al.*, 2021).

Traditionally, amorphous silica had been synthesized from chemical precursors such as tetramethyl orthosilicate (TMOS), tetraethyl orthosilicate (TEOS), and polyethoxydisiloxane (PEDS). However, the high cost and environmental issues associated with these synthetic sources prompted researchers to explore more sustainable alternatives, such as RHA (Mohamed *et al.,* 2015). In recent years, rice husk (RH) has emerged as an economical and eco-friendly source of amorphous silica, containing approximately 20–22% silica with minimal metallic impurities such as K_2O, Na_2O, and Fe_2O_3 (Nasir *et al.*, 2021; Hamidu *et al.*, 2025). The high silica content, amorphous nature, and large surface area of RHA make it an excellent support material for catalysts, offering superior reactivity, selectivity, and stability. Additionally, its porous structure enhances reactant diffusion, facilitates catalyst regeneration, and minimizes corrosion within reactor systems, further highlighting RHA's potential as a sustainable material for catalytic applications (Nasir *et al.*, 2021).

On the other hands, the excellent catalytic activity and low production cost of calcium oxide (CaO) catalysts have attracted considerable attention among heterogeneous base catalysts (Almasi *et al.*, 2024). As a heterogeneous base catalyst, CaO has demonstrated significant activity in vapor-phase dehydration of 1,4-butanediol (Yadagiri *et al.*, 2017), syngas production (Irfan *et al.*, 2021), and transesterification reactions, due to its strong basicity, mild reaction conditions, and relatively low environmental impact (Wang *et al.*, 2019). Moreover, renewable mineral sources offer an abundant and sustainable supply of CaO (Basumatary *et al.*, 2023). Various raw materials for the synthesis of CaO catalysts have been investigated, including oyster shells (Chen *et al.*, 2016; Syazwani *et al.*, 2017), eggshells (Ali *et al.*, 2023; Basumatary *et al.*, 2023), marble waste powder (Alhanif *et al.*, 2018), and animal bones (Suwannasom *et al.*, 2016; Adepoju *et al.*, 2021).

Marble waste powder, which is primarily composed of dolomite $CaMg(CO_3)_2$ and calcite ($CaCO_3$) (Tamim *et al.*, 2024), has recently attracted growing attention as a low-cost and sustainable precursor for CaO heterogeneous catalysts. Upon calcination, marble waste powder can be converted into highly reactive CaO with strong basicity and good catalytic activity. Previous studies have reported that the calcination of marble waste powder produces CaO with relatively high purity, reaching 55.42% (El-Sayed *et al.*, 2018), and a considerable surface area of 31.39 m^2/g (Nawar *et al.*, 2019), indicating its suitability as a potential catalyst precursor.

However, pure CaO catalysts exhibit instability and frequently suffer from leaching problems during the reaction (Wang *et al.*, 2019). Contact of CaO with water and CO_2 from the surrounding air decreases catalytic activity through hydration or carbonation reactions, forming $Ca(OH)_2$ and $CaCO_3$ (Kouzu and Hidaka, 2012; Chen *et al.*, 2016). In addition, partial leaching of Ca^{2+} ions from the CaO surface frequently occurs during transesterification, necessitating additional separation processes and producing substantial amounts of wastewater (Witoon *et al.*, 2014). Previous studies have also reported that pure CaO is partially soluble in methanol, releasing Ca^{2+} ions into the polar phase (Granados *et al.*, 2009; Marinković *et al.*, 2016). Moreover, the presence of FFA can promote soap formation through their interaction with the CaO catalyst during transesterification (Azzahro and Broto, 2022).

Currently, various CaO-based composite catalysts impregnated with metal oxides such as CaO/MgO (Yan *et al.*, 2008), CaO/ZnO (Toledo Arana *et al.*, 2019), CaO/CeO_2 (Kingkam *et al.*, 2024), and CaO/Al_2O_3 (Kesserwan *et al.*, 2020) have been widely synthesized to enhance the stability of CaO catalysts during reaction. These catalyst composites can inhibit the leaching of Ca^{2+} ions through electrostatic interactions between CaO and inorganic oxides (Wang *et al.*, 2019). However, the preparation of these catalyst composites is often complex and time-consuming, while the metal oxide precursors are relatively costly (Maleki *et al.*, 2017). Consequently, many non-metal-reinforced CaO/SiO_2 composite catalysts have been developed for practical

applications and have become the focus of recent studies. Witoon *et al.* (2014) successfully synthesized CaO-containing porous materials using bimodal meso–macroporous silica as a support material. Shan *et al.* (2017) also reported that a CaO catalyst supported on natural diatomite (SiO_2) exhibited excellent stability due to the uniform dispersion of Ca compounds and the formation of stable Ca–O–Si bonds. The presence of Ca–O–Si bonds enhanced the stability of CaO/SiO_2 composite catalysts while reducing the leaching of Ca^{2+} ions. A small degree of catalyst deactivation was observed for CaO supported on peat biochar, primarily due to the leaching of Ca^{2+} ions into the product phase (Wang *et al.*, 2019). These findings suggest that SiO_2 is a more effective support material for catalyst composite synthesis owing to its favorable physicochemical properties.

In this study, CaO catalysts derived from marble waste powder were impregnated with SiO_2 extracted from RHA. The SiO_2 served as a support material to enhance particle dispersion, reduce crystallinity, and increase the surface area of the CaO catalyst. Furthermore, the SiO_2 was sulfonated with $–SO_3H$ functional groups to introduce additional acid sites, thereby improving the catalyst's tolerance to free fatty acids (FFA). The physicochemical characteristics of the pretreated raw materials and the resulting catalysts were comprehensively analyzed using SEM, EDX, XRD, and BET techniques to evaluate the influence of composition on catalyst properties.

MATERIALS AND METHODS

Materials

RHA was obtained from brick kiln residue in Lampung, Indonesia, and marble waste powder was obtained from a chemical store in Jakarta, Indonesia. NaOH (99% purity, Merck, Germany) was used as the extracting agent for silica (SiO_2) from RHA and CaO from marble waste powder. HCl (37% purity, Merck, Germany) and H_2SO_4 (95-97% purity, Smart lab) used for chemical activation and sulfonation were purchased from chemical stores.

CaO Preparation from Marble Waste Powder

CaO, as a catalyst raw material obtained from marble waste powder , was pretreated and thermally activated prior to use. The marble waste powder was cleaned and washed with distilled water, then filtered and oven-dried at 105°C for 2 hours. The dried marble waste powder was subsequently calcined at 850°C for 2 hours to produce oxides such as CaO and MgO (El-Naggar *et al.*, 2024). The oxide composition was analyzed using X-ray fluorescence (XRF) to determine the CaO content after calcination.

Preparation and Sulfonation of SiO_2 from RHA

In the initial stage, pretreatment involved separating RHA from impurities and sieving it to obtain a particle size of 100 mesh. Chemical activation was performed by washing with 5 M HCl, followed by neutralization to pH 7 and drying at 150°C for 4 hours until a constant ash weight was achieved. Thermal activation was then conducted at 800°C for 6 hours. Sulfonation of SiO_2 was carried out to introduce $–SO_3H$ functional groups in several steps (Ajeel *et al.*, 2020). A total of 30 g of pretreated SiO_2 was mixed with 200 mL of 2 N NaOH solution, stirred, and heated at 80°C for 4 hours. After cooling to room temperature, the mixture was filtered and neutralized with 5 M H_2SO_4 to pH 7, producing a white gel. The gel was dried at 60°C for 12 hours and subsequently calcined in a furnace at 800°C for 6 hours. The SiO_2 powder obtained before and after sulfonation was analyzed using XRF to determine the SiO_2 and SO_3 contents.

Synthesis and Characterization of CaO/SiO_2 Catalyst

The CaO/SiO_2 heterogeneous catalyst was synthesized using the wet impregnation method (Ho *et al.*, 2022). For this process, 30 g of calcined CaO was dispersed in 200 mL of deionized water and heated at 70°C in a closed system under magnetic stirring at 700 rpm. Sulfonated SiO_2 was then gradually added to the CaO dispersion at 25:75, 50:50, and 75:25 %wt. ratios, followed by stirring for 4 hours until a homogeneous mixture was achieved. The $CaO:SiO_2$ ratio was determined based on the weights of the pretreated CaO powder and sulfonated SiO_2, given the relatively high purity of the raw materials. The mixture was then allowed to stand for 18 hours, after which the water was removed by oven-drying at 105°C for 24 hours. The resulting dried catalyst was subsequently calcined in a furnace at 850°C for 2 hours.

The morphology, shape, and elemental composition of the CaO/SiO_2 catalyst were characterized using scanning electron microscopy (SEM, JEOL JSM-6510LA, Japan) equipped with energy-dispersive X-ray spectroscopy (EDX, FEI Quanta 400F, 15 kV). The specific surface area, pore volume, and pore size were determined through Brunauer–Emmett–Teller (BET) analysis using N_2 adsorption–desorption isotherms on a Micromeritics ASAP 2020 analyzer. In addition, the crystal structure of the CaO/SiO_2 catalyst was analyzed by X-ray diffraction (XRD, Shimadzu, Japan).

RESULTS AND DISCUSSION

Chemical Analysis of RHA and Marble Waste Powder After Pretreatment and Sulfonation

Silica (SiO_2) was an inorganic oxide compound composed of silicon and oxygen atoms arranged in tetrahedral structural units. These units bonded to each other, forming a three-dimensional network that constituted the SiO_2 matrix. The material could exist in either an amorphous form or as a crystalline phase (quartz), the latter being more thermodynamically stable, with pore sizes ranging from 5 to 3000 Å (Nzereogu *et al.*, 2023). SiO_2 could be extracted from

various natural sources, including quartz sand, plant leaves, and agricultural wastes (Luthfiah *et al.*, 2021). XRF analysis of rice husk ash (RHA) after chemical and thermal activation (pretreatment) revealed a dominant SiO_2 component (93.67%) compared to other oxides such as K_2O, P_2O_5, CaO, and Fe_2O_3 (Table 1). Acid activation of RHA using HCl reduced impurities in the form of metal oxides, particularly Na_2O, K_2O, and CaO (Wogo *et al.*, 2011). Acid treatment also minimized the formation of undesirable hexagonal SiO_2 crystals by decreasing the surface area and interactions with other compounds (Başgöz and Güler, 2020). Subsequent washing with deionized water further removed residual Cl^- contaminants from HCl (Fernandes *et al.*, 2017). The SiO_2 content obtained in this study exceeded the values reported in previous investigations on RHA, which recorded values of 76.6% and 82.26%, respectively (Raharja *et al.*, 2013; Susanto *et al.*, 2021). Variations in SiO_2 content among different RHA samples were largely influenced by factors such as plant species, climatic conditions, fertilizer type, soil chemistry, and geographic region (Suryana *et al.*, 2018). In addition, the SiO_2 content obtained from RHA was higher than that derived from fly ash (49%) (Wahyuono *et al.*, 2024), bagasse charcoal (68.88%) (Adli *et al.,* 2018), and coconut husk (92.97%) (Nurhayati *et al.*, 2025). However, it was still lower than the purity of commercial SiO_2 (99%) (Elizondo-Villarreal *et al.*, 2024).

Table 1. The results of XRF analysis of SiO_2 and CaO from RHA and marble waste powder

Oxide compounds	Composition (%)		
	RHA after pre-treatment	Sulfonated RHA	Marble waste powder after calcinated
SiO_2	93.67	60.29	0.37
P_2O_5	0.75	0.74	-
SO_3	0.28	36.50	-
K_2O	3.58	2.00	-
CaO	0.64	0.36	99.13
TiO_2	0.21	*Trace*	-
MnO	0.18	*Trace*	*Trace*
Fe_2O_3	0.58	*Trace*	*Trace*
Al_2O_3	-	-	0.38

Extraction of SiO_2 from RHA through alkali treatment (NaOH) followed by sulfonation via strong acid titration (H_2SO_4) yielded SiO_2 and SO_3 contents of 60.29% and 36.50%, respectively (Table 1). The higher SO_3 content compared with the non-sulfonated sample indicated that the incorporation of $–SO_3H$ groups was successful. According to previous studies, SiO_2 functionalized with $–SO_3H$ groups exhibited enhanced surface acidity and catalytic activity as a solid acid catalyst, while also demonstrating reusability without a significant decline in performance (Sherry and Sullivan, 2011; Shah *et al.*, 2014). The increased surface acidity was particularly advantageous when sulfonated SiO_2 was impregnated with a base catalyst, as it minimized the risk of saponification reactions caused by the FFA content in the feedstock (Melero *et al.*, 2009).

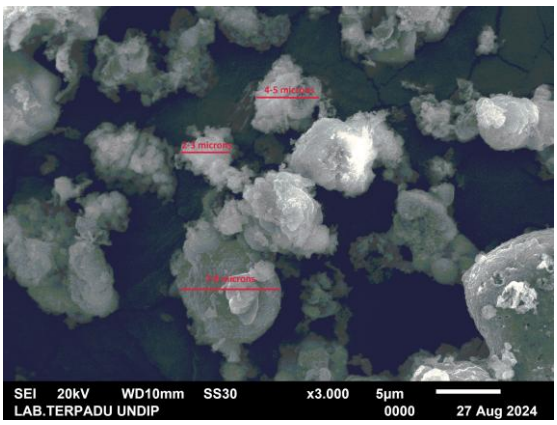
Based on Table 1, the results of the XRF analysis of marble waste powder calcined at 850°C for 2 hours showed a CaO composition of 99.13%. This value was considerably higher than that reported in previous research, which obtained a CaO content of 80.13% from marble sludge (El-Naggar *et al.*, 2024). The higher purity in this study could be attributed to the more efficient calcination process in powdered form compared to sludge, as well as differences in the origin of the raw materials. Moreover, the low impurity content in the initial feedstock also contributed to the higher CaO purity obtained. The CaO content obtained in this study was higher than that reported for other raw materials such as oyster shells (90.45%) (Lin *et al.*, 2020) and bovine bones (57.3%) (Prasetyo *et al.*, 2024), and was comparable to CaO sources such as eggshells (94.5% and 95.3%) (Erchamo *et al.*, 2021; Owoeye *et al.*, 2024) and limestone (95.94% and 97.98%) (Hwidi *et al.*, 2018; Elfina *et al.*, 2024).

CaO acts as a basic catalyst in the transesterification of vegetable oils with alcohols (Basumatary *et al.*, 2023). In catalytic systems, high–melting-point oxides such as CaO function as active components. The XRF analysis further revealed the presence of minor amounts of SiO_2 and Al_2O_3, at 0.37% and 0.38%, respectively. SiO_2 serves as a support that can significantly enhance the dispersion of CaO particles, thereby increase the effective surface area and minimize particle agglomeration (Wang *et al.*, 2024). This structural improvement leads to higher catalytic activity and better stability under reaction conditions. Similarly, Al_2O_3 has been shown to act as an effective support for CaO due to its amphoteric nature and thermal robustness. The presence of Al_2O_3 improves the mechanical strength and thermal resistance of CaO (Teixeira *et al.*, 2022), and may also contribute to the formation of bifunctional acid–base sites (Elias *et al.*, 2020).

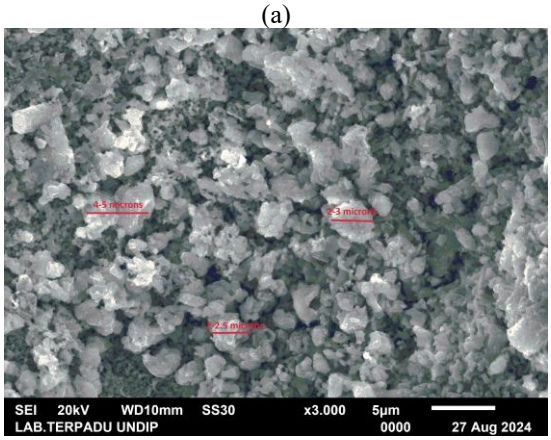
Surface Morphology of CaO/SiO_2 Catalyst

The CaO/SiO_2 catalyst was synthesized by combining SiO_2 derived from sulfonated RHA with CaO from marble waste powder at ratios of 25%CaO/75%SiO_2, 50%CaO/50%SiO_2, and 75%CaO/25%SiO_2. The catalyst mixtures were dried at 105°C for 24 hours and subsequently calcined at 850°C for 2 hours. The surface morphology of the catalysts was characterized using SEM analysis, as shown in Figure 1. Figure 1(a) presents the morphology of the catalyst containing 25%CaO, which exhibited particle sizes ranging from 2 to 8 µm with a predominantly spherical appearance. Large pores were observed between the particles, which could potentially lead to a reduced surface area. The

catalyst with this composition was dominated by large, irregular, and hollow crystals. The presence of SiO_2 in the catalyst was expected to act as a dispersion site for CaO; however, the high proportion of SiO_2 resulted in uneven dispersion and the formation of large aggregates (Ndak *et al.*, 2021). The agglomeration of SiO_2 particles hindered the optimal dispersion of CaO. This observation was consistent with the findings of Nurhayati *et al.* (2025), who reported that SiO_2 exhibited a porous and aggregated structure, with particles adhering to one another to form large micrometer-sized clusters.



(a)



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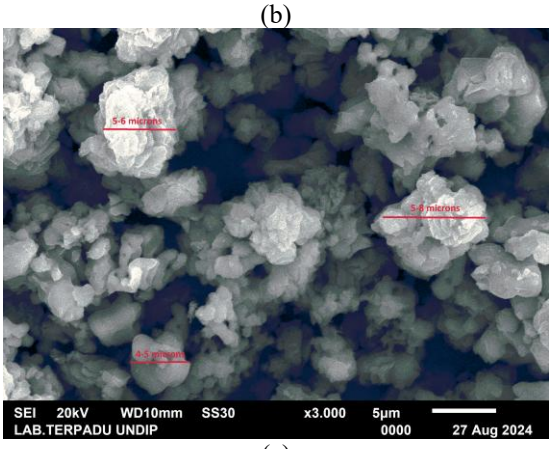

(c)

Figure 1. Surface morphology of CaO/SiO_2 catalyst, a) 25% CaO, b) 50% CaO, and c) 75% CaO with 3000x magnification

These clusters displayed variations in both shape and size, reflecting a broad particle size distribution and natural agglomeration during synthesis (Setyawan *et al.*, 2021).

Figures 1(b) and 1(c) present the morphology of CaO/SiO_2 catalysts with 50% and 75% CaO compositions, respectively. In both catalysts, a more uniform variation in particle size was observed, consisting of smaller particles measuring 2–5 µm and larger agglomerates reaching up to 8 µm. The presence of smaller particles was expected to enlarge the surface area and number of active sites, thereby enhancing catalytic activity. Thus, both CaO compositions had the potential to exhibit strong catalytic performance. In Figure 1(b), the catalyst with 50%CaO displayed a more uniform particle size distribution compared with the 75%CaO catalyst. However, particle accumulation was evident, which could reduce porosity and specific surface area. Previous studies have shown that smaller particle sizes and more uniform particle distributions increase the catalyst surface area (Zhang *et al.*, 2022). Furthermore, the incorporation of SiO_2 in an appropriate proportion within the CaO catalyst increased the total pore volume, which in turn enhanced the number of active sites available for interaction with reactants (Wongjaikham *et al.*, 2023).

Oxide Composition of CaO/SiO_2 Catalyst

The oxide composition of the CaO/SiO_2 catalysts was determined using EDX analysis, as presented in Figure 2. The catalyst with 25% CaO addition contained 26.58% CaO, 24.93% SiO_2, and 13.09% SO_3. The catalyst with 50% CaO addition consisted of 39.28% CaO, 19.48% SiO_2, and 10.62% SO_3, while the catalyst with 75% CaO addition contained 73.13% CaO, 8.22% SiO_2, and 2.85% SO_3. These results demonstrated that increasing the CaO proportion during catalyst preparation was consistently accompanied by higher CaO content, while the relative proportions of SiO_2 and SO_3 decreased. However, the actual oxide compositions obtained from EDX analysis differed from the theoretical mixing ratios. For instance, the 50% CaO addition during mixing resulted in a measured CaO content of 39.28%. This discrepancy could be attributed to the presence of impurities such as P_2O_5 and K_2O in both raw materials (CaO and sulfonated SiO_2), as confirmed by the XRF results in Table 1.

Catalyst impregnation was intended to enhance the stability and reactivity of CaO by increasing its surface area and thermal stability through the incorporation of SiO_2 (Sofyan *et al.*, 2024). The increase in CaO content accompanied by the decrease in SiO_2 indicated the successful impregnation of CaO onto SiO_2, which was influenced by the effectiveness of the impregnation process (Haryono *et al.*, 2023). An increase in CaO content within the CaO/SiO_2 catalyst corresponded to higher basicity,

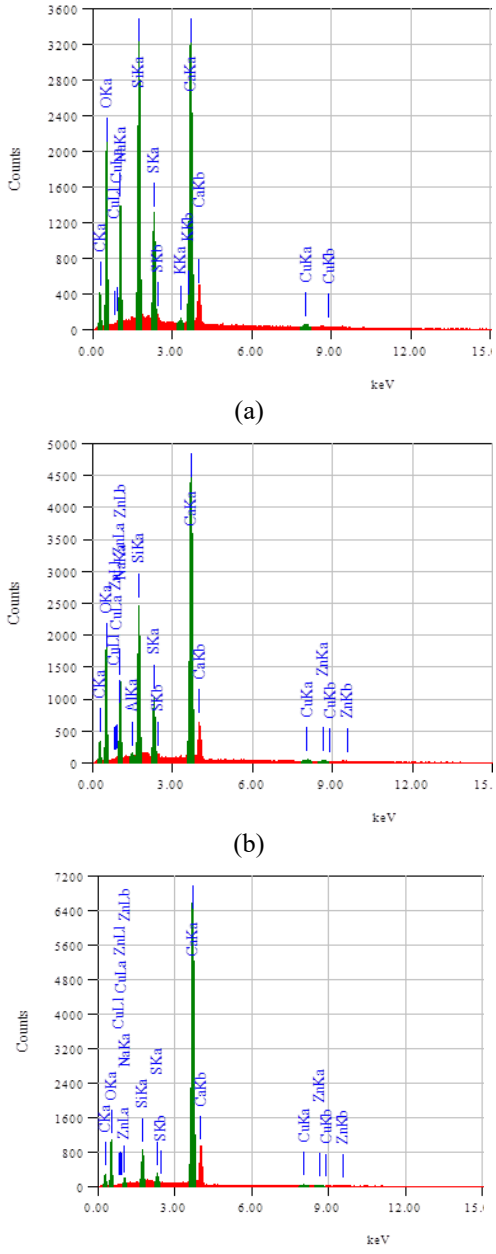

Figure 2. Oxide composition of CaO/SiO_2 catalysts with different CaO loadings, a) 25% CaO, b) 50% CaO, and c) 75% CaO

thereby improving catalytic activity in the transesterification process. Conversely, the presence of SiO_2 as a catalyst support enhanced the mechanical strength of the catalyst, making it more resistant to high reaction temperatures. Furthermore, the presence of SO_3, derived from sulfonated $–SO_3H$ groups, acted as Brønsted acid sites, thereby increasing surface acidity and reducing sensitivity to FFA (Wang *et al.*, 2025)

Crystallinity of CaO/SiO_2 Catalyst

XRD analysis was performed to evaluate the crystallinity of the CaO/SiO_2 catalysts. The XRD pattern of the catalyst with 25% CaO composition exhibited sharp diffraction peaks within the 2θ range of 20 – 33° (Figure 3a). Nandanwar *et al.* (2015) reported that the presence of crystalline inorganic SiO_2 could enhance the overall crystallinity of a material when impregnated. Increasing the CaO composition eliminated the crystal peaks observed in the 20 – 30° range, while new peaks emerged in the catalysts with 50% and 75% CaO compositions at 2θ values of 42 – 55° (Figures 3b and 3c). The XRD pattern of the 75% CaO catalyst (Figure 3c) revealed a reduction in crystallinity and the presence of an amorphous structure with only a few sharp peaks, consistent with observations reported in previous studies (Garmsiri *et al.*, 2018).

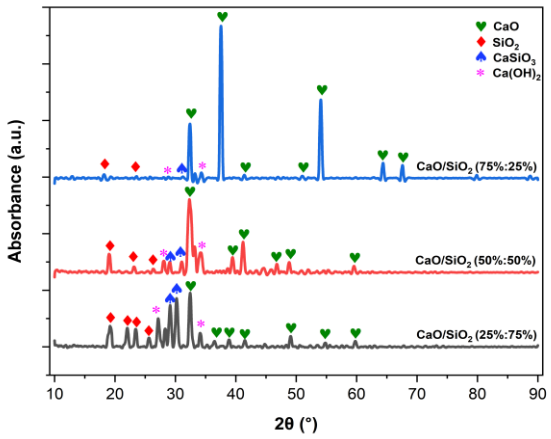

Figure 3. Crystallinity of CaO/SiO_2 catalyst with varying compositions

The appearance of a new diffraction peak at 31 – 32°, 36 – 42°, and 50 – 70° in the catalyst with 75% CaO composition indicated that the calcination process successfully decomposed calcite ($CaCO_3$) into CaO (Nurhayati *et al.*, 2025). Peaks corresponding to portlandite ($Ca(OH)_2$) were still detected in the 2θ ranges of 27 – 28° and 34 – 35° for all CaO/SiO_2 compositions. This was attributed to the reaction of CaO with water vapor or exposure to air during catalyst synthesis, given the hygroscopic nature of CaO (Xin *et al.*, 2021; Nurhayati *et al.*, 2025). In addition, a $CaSiO_3$ peak was observed in the 28 – 30°, which may have overlapped with the $Ca(OH)_2$ peak. This result was consistent with the findings of Nurhayati *et al.* (2025), who identified calcium silicate ($CaSiO_3$) in the same region. The presence of $CaSiO_3$ confirmed that CaO reacted with SiO_2 during the impregnation process.

Physical Characteristics of CaO/SiO_2 Catalyst

The physical characteristics of the CaO/SiO_2 catalysts, including surface area, pore volume, and pore diameter, were analyzed using BET and the results are summarized in Table 2. The catalyst containing 25% CaO exhibited a surface area of 1.999 m^2/g. Increasing the CaO content to 50% and 75% enhanced the surface area to 3.257 and 22.244 m^2/g, respectively.

Table 2. BET results of CaO/SiO_2 catalyst with varying compositions

Catalyst	Surface area (m^2/g)	Pore volume (mm^3/g)	Pore diameter (nm)
CaO/SiO_2 (25%/75%)	1.999	25.98	51.98
CaO/SiO_2 (50%/50%)	3.257	44.95	55.20
CaO/SiO_2 (75%/25%)	22.244	65.29	11.74

This result was consistent with the findings of Manurung *et al.* (2023), who reported that the CaO/SiO_2 catalyst synthesized from chicken eggshells and palm ash, with a composition of 20 %wt. palm ash and calcination at 800 °C, exhibited the highest catalyst surface area of 21.483 m^2/g. The impregnation of CaO onto the SiO_2 support increased the catalyst surface area compared to the commercial CaO (3 m^2/g) without a SiO_2 support, as previously reported by Putra *et al.* (2017). This enhancement is expected because pure SiO_2 possesses a relatively large surface area (39.7 m^2/g) (Putra *et al.*, 2017). However, an excessive incorporation of SiO_2 led to a reduction in the catalyst surface area due to particle agglomeration. Moreover, the surface area of the CaO/SiO_2 catalyst synthesized from RHA and marble waste powder at a CaO composition of 75%wt was higher than that of commercial CaO and SiO_2 (13.3 m^2/g). However, at a CaO composition of 50%wt, the surface area was lower than that of the commercially synthesized catalyst (19.0 m^2/g) (Zdujić *et al.*, 2019).

On the other hand, the enlarge catalyst surface area was also accompanied by an increase in pore volume and a reduction of pore diameter. A higher pore volume indicated greater void space within particle's structure, which could serve as major additional active sites. Meanwhile, the decrease in pore diameter suggested a denser particle packing, contributing to the enhancement of surface area. Conversely, a high proportion of SiO_2 promoted the agglomeration of SiO_2 particles, thereby limiting the effective dispersion of CaO within the catalyst structure (Nurhayati *et al.*, 2025).

Further observations indicated that the pore diameter of the CaO/SiO_2 catalyst at compositions ranging from 25% to 75% CaO was within the range of 11.74 – 55.20 nm. This pore diameter was larger than the molecular size of the designated reactants, which are usually fatty acids, such as palmitic acid (0.37 nm), stearic acid (0.25 nm), oleic acid (0.72 nm), and linoleic acid (1.13 nm) (Shuit *et al.*, 2015; Ikeda *et al.*, 2021). The relatively larger average pore diameter of the catalyst, combined with the interconnected pore structure, reduced diffusion barriers for reactant molecules. Consequently, the reactants could more readily penetrate the catalyst structure, the products could diffuse outward with ease, and a greater proportion of the active sites could be utilized during the transesterification reaction (Tang *et al.*, 2019).

CONCLUSION

Based on the research findings, calcined RHA and marble waste powder after pretreatment contained primarily 93.67% SiO_2 and 99.13% CaO, respectively. The sulfonation of SiO_2 successfully produced a material with 60.29% SiO_2 and 36.50% SO_3. The morphology of the CaO/SiO_2 catalyst with a composition of 25% CaO exhibited predominantly spherical particles ranging from 2 to 8 µm in size, uniformly loaded by large pores and a dominant presence of large crystals. In contrast, catalysts with 50% and 75% CaO compositions demonstrated a more uniform particle distribution. The successful incorporation of CaO was confirmed by the actual compositions of CaO, SiO_2, and SO_3, which closely matched the calculated values at the mixing stage. Moreover, the incorporation of CaO reduced the crystallinity, as evidenced by the disappearance of several crystal peaks within the 2θ ranges of 31 – 32°, 36 – 42°, and 50 – 70°. Furthermore, the inclusion of CaO increased the catalyst surface area to 22.24 m^2/g and the pore volume to 65.29 mm^3/g, although it was accompanied by a reduction in pore diameter to 11.74 nm.

CONFLICT OF INTEREST

Financial contributions and any potential conflict of interest must be clearly acknowledged under the heading 'Conflict of Interest'. Authors must list the source(s) of funding for the study. This should be done for each author.

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