# Transesterification Of Soy Oil For The Production Of FAME Catalyzed By A Ni/Cao Blended Oxide

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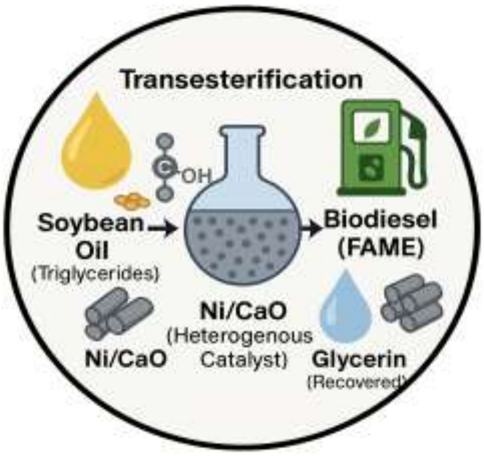
***Abstract:***
*Biodiesel was prepared from soy oil and a mixture of NiO and CaO oxide. Employing calcium (Ca) as a support and Ni as a driver ingredient, the oxide is created by mechanically mixing it. It was investigated how the Ni/CaO catalyst affected the process of turning soy oil into FAME. A variety of catalyst characterization techniques, including BET, FTIR, and more were used to describe the catalyst material. Using a variable weight percentage of Ni, the Ni/CaO catalyst exhibits a 20 wt% transformation rate with soy oil, with NiO being found to be the most effective. $CH_3OH$/oil M ratio 15:01, the temperature of the reaction 64°C, and the amount of catalyst 5wt% were the optimal reaction parameters for FAME conversion. This mixed catalyst is more useful and can produce biodiesel.*

***Keywords:*** *FAME, Mixed Oxide, Catalyst, Soy oil, Base*



## INTRODUCTION

The need for energy is increasing gradually in tandem with the world's population. Although fossil fuels are still the most common source of energy, their supply is strongly correlated with changes in the world economy. Because of the discharge of toxic pollutants such as carbon dioxide ($CO_2$), sulfur oxides ($SO_x$), and nitrogen oxides ($NO_x$), the prolonged and excessive use of these fuels presents serious environmental problems. The environment is harmed by these contaminants. Fossil fuels are also becoming less and less dependable due to problems like fluctuating crude oil prices, excessive consumption, and depleting reserves. These worries highlight the increasing necessity of looking for and funding alternate energy sources. Among the many alternative fuels that are accessible, biodiesel is one of the most promising. It was created by a chemical process known as transesterification, in which methanol and oil react. Edible oils including coconut, palm, peanut, and rapeseed, as well as non-edible oils, can be used as feedstock for the manufacturing of biodiesel. The high cost of producing biodiesel is a major barrier. There are two types of catalysts used to enhance the chemical process: homogeneous and heterogeneous. Despite their high efficiency, homogeneous catalyst's main drawback is how challenging it is to separate them from the reaction's end product. In order to get around this limitation, heterogeneous catalysts are recommended because they are simple to separate and repurpose, which makes the procedure more useful and economical. Using vegetable oil as the raw material, Kim et al. [1] employed a solid base heterogeneous catalyst made of $Na/NaOH/AlO_3$ to get a high biodiesel yield of 94%. By using a $EuO_3/AlO_3$ catalyst, Li et al. [3] were able to convert soybean oil at 70°C for eight hours at a rate of 63.2%. According to Nakagaki et al. [7], anhydrous $MoNa_2O_4$ was an effective solid catalyst that produced more than 95% biodiesel from soybean oil. As a solid base catalyst, Guo et al. and associate used $Na_2SiO_3$, which demonstrated potential for large-scale industrial applications in addition to producing outstanding yields [10]. Alkaline earth metals are also frequently used in the development of different blended base catalysts due to their good catalytic characteristics. Studies [4,5,18] have shown that calcium oxide (CaO) works well as a catalyst to produce biodiesel from soybean oil, with yields exceeding 90% in as little as two to three hours. Other functional metals may have been added with CaO to create hybrid oxide catalysts, which further enhance its catalytic activity. A 12:1 methanol/oil molar ratio over a 6-hour reaction time has demonstrated rates of transformation above 92% for calcium methoxide CaO/KF, [$Ca(OCH_2CH_3)_2$], $CaO/SnO_2$, and Li/CaO [6,9,12,13]. Zhang et al. and colleague [16] used discarded deposits of flake from boiler walls as Ca supported catalyst in a unique way, and the FAME synthesis process worked well. With a catalyst dose of 3–5%, Liu et al. [2] investigated the usage of strontium-based catalysts and produced FAME yields that exceeded 95%. In approximately 0.5 hours, Tantirungrotechai et al. [14] achieved a 93% yield by enhancing MgO with strontium. Using a 14:01 methanol/oil mole ratio and 6 weight percent catalyst loading, Li et al. [11] created a $KOH/Nd_2O_3$ oxide-based catalyst that produced a 92.41% yield, with enhanced performance at larger ratios. In contrast, a mesoporous SBA-15 catalyst examined by Zuo et al. [15] at 190°C produced a lesser yield of 85%, showing decreased catalytic efficiency. In the present investigation, soy oil was transesterified using a Ni/CaO heterogeneous solid base catalyst. Numerous characterization techniques, including SEM, XRD, BET, FTIR, and TEM were used to assess the catalyst, and the impact of important reaction parameters was carefully examined.

## Experimental

### 2.1 Soy Oil's Physicochemical Properties

A regional market provided the soybean oil, which was then examined using accepted methods. Acid value, Density, and free fatty acid content were among the metrics that were assessed. The measured AV and SV readings were used to calculate the oil's average MW. The solution of alkaline was used in a quantitative procedure to determine the acid value.

### 2.2 Catalyst Development

A mechanical mixing technique was used to create the Ni/CaO basic oxide catalyst. First, distilled water was used to dissolve starting solutions of calcium nitrate and nitrate of nickel that contained 20-weight percent nickel. To get rid of any last traces of moisture, these solutions were vigorously stirred and completely mixed before being dried overnight at 110°C. After drying, the mixture was then heated for five hours in air at 800°C in a muffle furnace. The resultant substance was cooled, then milled into an acceptable powder, and given the designation Ni/CaO.

### 2.3 Catalysts characterization

Using a Philips X'pert MPD, powder XRD was carried out to examine the catalysts' crystallographic and structural features. A Perkin Elmer instrument was used to perform infrared spectrum analysis in order to evaluate the impact of chemical groups on the fundamental catalyst. Using nitrogen adsorption-desorption equilibrium conditions and the BET technique, the area of the surface was determined using a Micromeritics ASAP 2010. With a JEOL JSM 7100F, SEM and EDX were used to analyze the surface morphology and elemental composition. Furthermore, a JEOL JSM 2100 TEM was used to view the intricate surface structure. Every characterization process was carried out at CSMCRI.

### 2.4Experimental Setup and Procedure

The study was carried out at LEC, Morbi's CRE Laboratory. Using a heating mantle, a 50 mL two-neck RBF with a condensation device was heated in a water bath for the reaction. Ten grams of soybean oil were used in the reaction, which varied in the temperature range 40 to 70°C, the amount of catalyst ranged from 1 to 6%, the reaction period from 2 to 6 hours, and the alcohol level (6–21%). Whatman filter media was used to filter out the catalyst once the reaction was finished. After that, the reaction mixture was put in a funnel that separates and left there at night to undergo phase separation. The performance of the catalyst was examined in various operating scenarios.

## FINDINGS AND DISCUSSION

### 3.1 Soybean Oil's Physicochemical Properties

Several qualities of soybean oil were examined. A density of 0.91 g/mL was recorded. It was estimated that the saponification value was 192.2 mg of KOH per gram of oil. The FFA content was calculated to be 1.122% by weight, while the acid value was found to be 2.244 mg KOH/g. The average molecular weight of soybean oil was estimated to be around 886.2 g/mol based on these factors.

### 3.2 Catalyst characterization

The complete thermal breakdown of calcium carbonate into calcium oxide takes place between 800 and 900 °C, according to Shajaratun et al. [20]. Meanwhile, between 275°C and 500°C, nickel hydroxide is converted to nickel oxide, according to Bera et al. and associates[21].

The X-ray diffraction result of the 20% Ni/CaO catalyst are shown in Figure 1. According to the standard JCPDS File, the calcined Ni/CaO catalyst's XRD spectrum showed diffraction peaks that matched those of CaO at 2θ values of 32.47, 37.62, 54.16, 64.46, and 67.67. According to the JCPDS File, additional peaks that corresponded to NiO were found at 37.62, 43.50, 63.06, and 75.55.

Based on certain XRD peaks, the Debye-Scherrer equation was used to estimate the catalyst's crystallite size; the findings are shown in Table 1.

Table 1 shows the 20% Ni/CaO catalyst's crystallite sizes and BET area.

| **Name of Catalyst** | **Crystallite Size(nm)** | | $S_{BET}$ **(m$^2$/g)** |
|---|---|---|---|
| | CaO | NiO | |
| NiO/CaO | 23.92 | 20.27 | 2.33±0.074 |

The crystallite size of the CaO phase in the Ni/CaO composite catalyst was 23.92 nm, whereas the NiO phase was bigger at 20.27 nm. As anticipated, a decrease in crystall

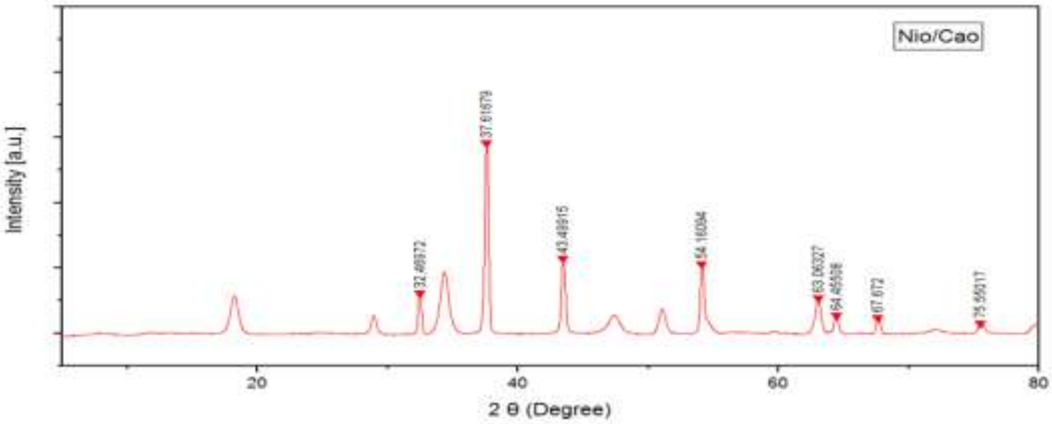
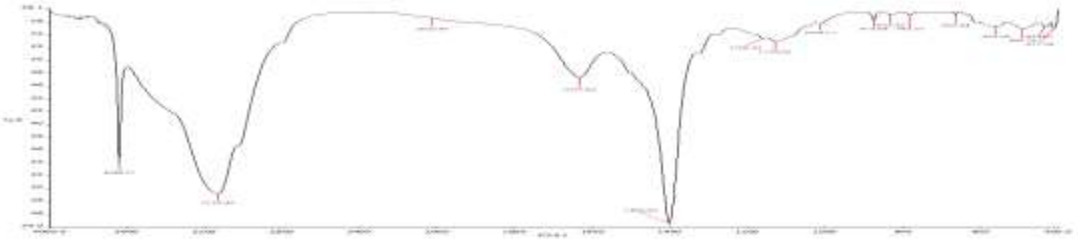


**Fig.** 1. Chart of XRD for Ni/CaO catalystite size correlated with an increase in FWHM. High-temperature calcination and NiO integration into the calcium oxide matrix caused sintering, which changed the mixed oxide system's particle shape.The NiO/CaO FTIR spectra are displayed in Figure 2. The presence of $CaCO_3$ was shown by an absorption band in the calcined catalysts' spectra located at 1402 cm⁻¹. The hydroxyl groups from structural water adsorbed on the catalyst interface are also represented by a wide band in the 3139–3644 cm⁻¹ region. These absorption bands imply that carbon dioxide and water were adsorbed onto the Calcium oxide surface during the air calcination process, which partially formed carbonate and hydroxyl species.

**Fig.** 2. FTIR of NiO/CaO catalyst



The vibratory stretching of the NiO bond was identified as the cause of the prominent absorption wave at 562 cm⁻¹ in the Ni/CaO catalyst's FTIR spectra. The CaO and Ca–O–Ni connections are represented by peaks that emerge at 875 cm⁻¹ and 614 cm⁻¹, accordingly [4]. A crowning at 713 cm⁻¹ was also found, and it was linked to CaO vibrations that were impacted by the existence of NiO in the catalyst structure [19].

Table 1 provides a summary of the exterior area of NiO-CaO mixed catalysts. These results demonstrate that surface area decreases with increasing crystallite size, which was in line with the XRD findings. The fine particles probably sinter due to the elevated calcination temperature of 800 °C, which led to their agglomeration and, ultimately, a decrease in the catalyst's surface area.

The SEM pictures showing the Ni/CaO catalyst's morphology are shown in Figure 4. The SEM data demonstrates a dual particle size pattern in Figure 4. Much smaller CaO particles were dispersed throughout the surfaces of the larger, erratic grain-like structures, which were designated as NiO crystals. This finding is consistent with the dimension of the crystallite data obtained from the XRD examination. It implies that the mixing of NiO results in a decrease in the size of CaO particles. Particle agglomeration brought on by high-temperature calcination is most likely the cause of the clustering observed in the pictures.

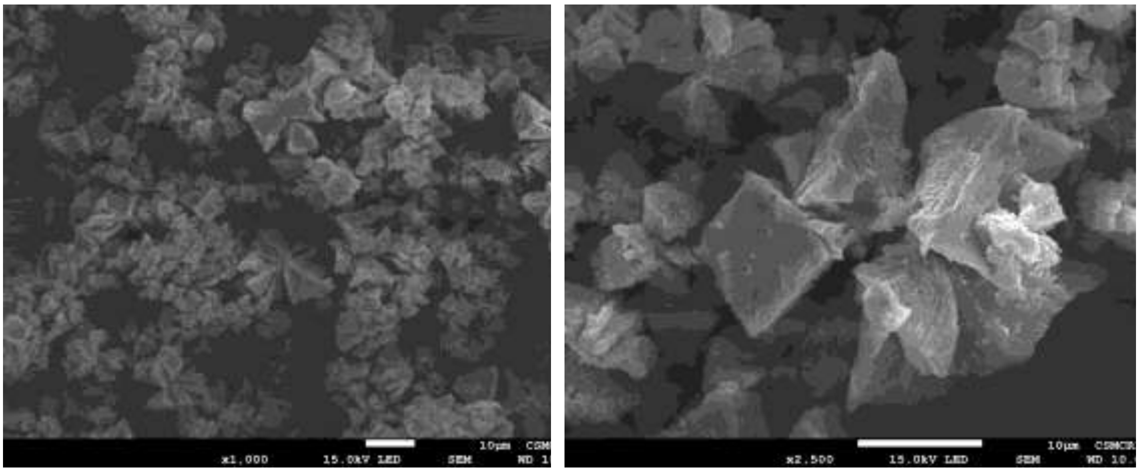


**Fig.** 3. SEM image of NiO/CaO catalyst

As seen in Figure 4, EDX analysis verified that the catalyst included both calcium and Ni. Nickel particles are evenly distributed throughout the calcium oxide surface, as seen in the TEM image. The image's darker portions show where NiO has been placed upon the CaO surface, suggesting that there is good nickel oxide contact and distribution across the support.

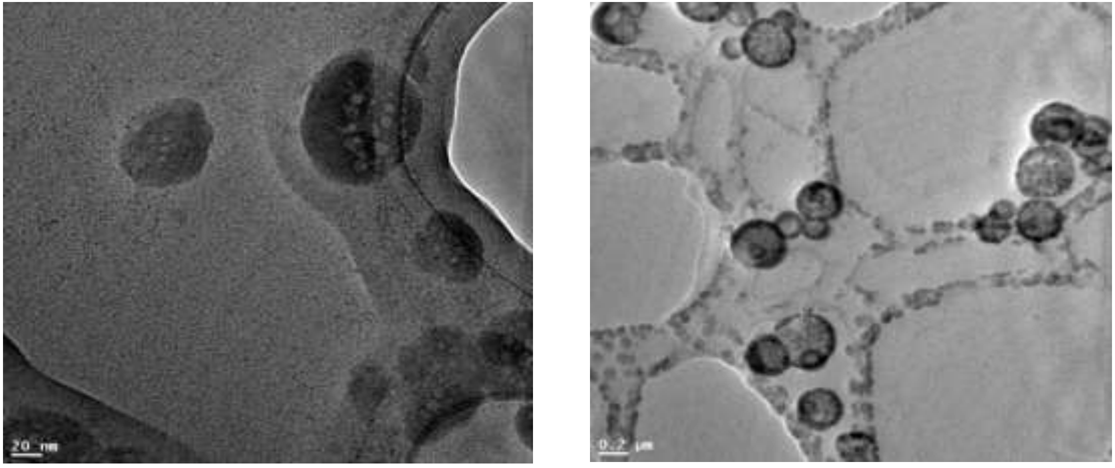


**Fig** 4. TEM image of Ni/CaO catalyst

### 3.3 Optimal Reaction conditions

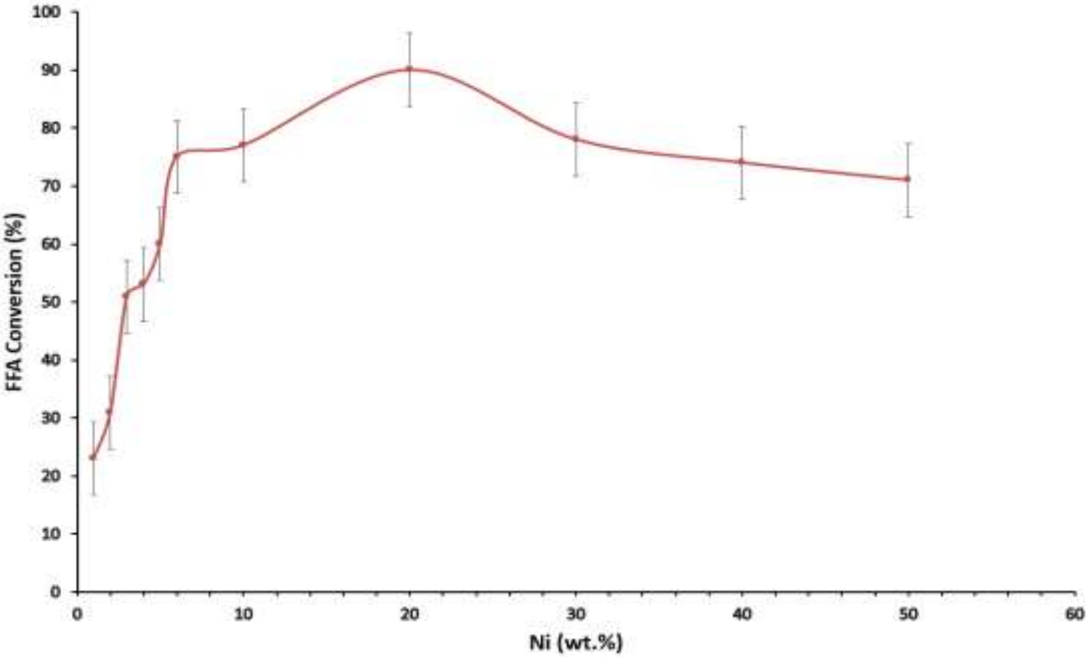
#### 3.3.1 The activity of catalysis for Ni/CaO blended oxide

Adding different concentrations of nickel (Ni) to a calcium oxide (CaO) substrate resulted in a variety of blended oxide catalysts. The efficiency of these catalysts in encouraging the transesterification of soy oil into FAME was then evaluated. Conditions such as 10 g of soybean oil, 64 °C, a methanol/oil molar ratio of 12:01, and a catalyst dosage of 5 weight percent were used to perform the reactions. As shown in Fig. 5 [17], the catalyst with 20% Ni by weight exhibited the highest efficiency, achieving FAME conversion rates exceeding 90%. Keeping the 20% Ni fixed the other process parameters are optimized.

**Fig** 5. Optimized Ni percentage at stated condition

#### 3.3.2 Reaction time

It was investigated how reaction time affected the conversion of free fatty acids (FFAs). The prolonged contact time between the catalyst and reactants was responsible for the better FFA conversion, according to the results. After five hours of reaction, the maximum conversion, around 92%, was reached. If less time was provided the reaction may incomplete on the other side if more time initiate the reverse the reaction. These results imply that, as shown in Fig. 6, reaction time is an important factor in the generation of biodiesel in air conditions. **Fig 6**. Effect of process time on FAME amount at conditions such as $CH_3OH$/oil ratio 12:01, 64°C temperature, and 5wt% Catalyst amount **3.3.3 $CH_3OH$/Soy oil ratio** Three moles of methanol are needed for every mole of soy oil, based on the transesterification reaction's stoichiometry. Utilizing too much methanol aids in shifting the balance in favor of the synthesis of methyl esters because the reaction is reversible. FAME conversion significantly improved with an increase in the methanol-to-oil molar ratio (Figure 7). The maximum conversion rate of 94% was attained at a molar ratio of 15:0;1. Nevertheless, conversion efficiency decreased when the methanol ratio increased more.



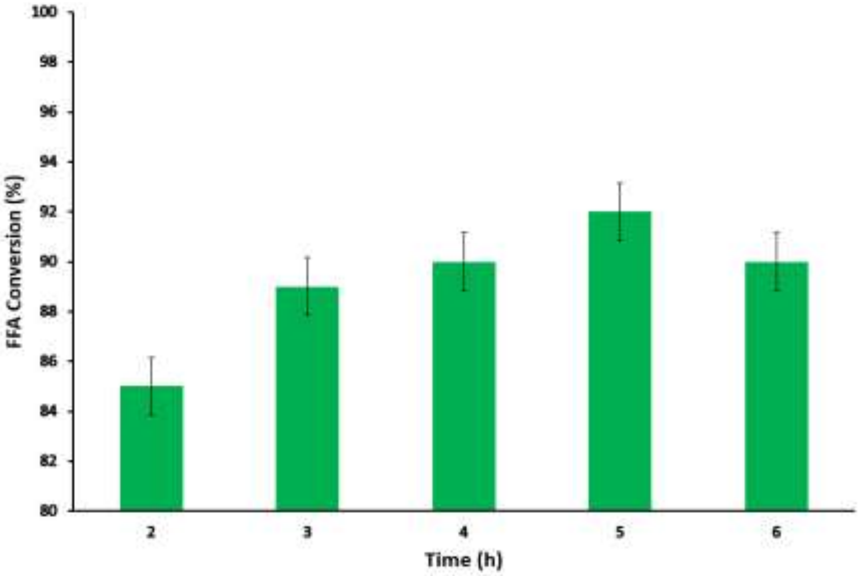


**Fig 7**. Effect of molar ratio on FAME amount at conditions such as
64°C temperature, 5wt% Catalyst amount, and 5 h time

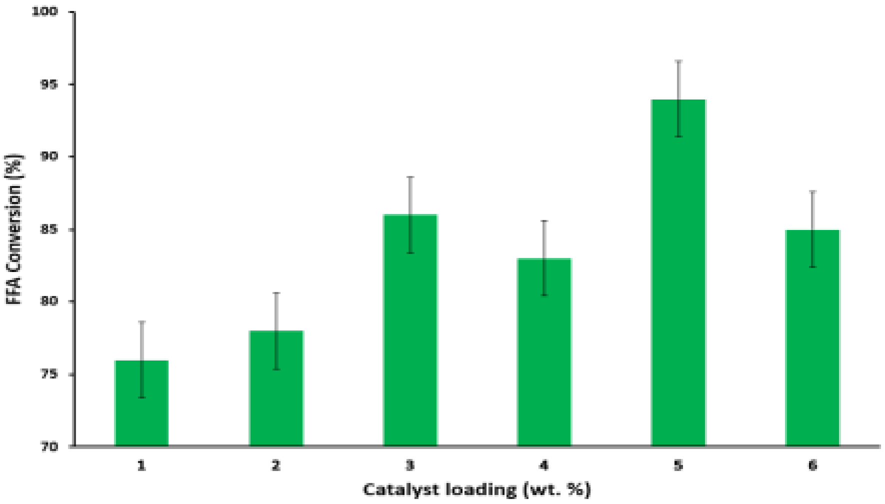
### 3.3.4 Catalyst-loading

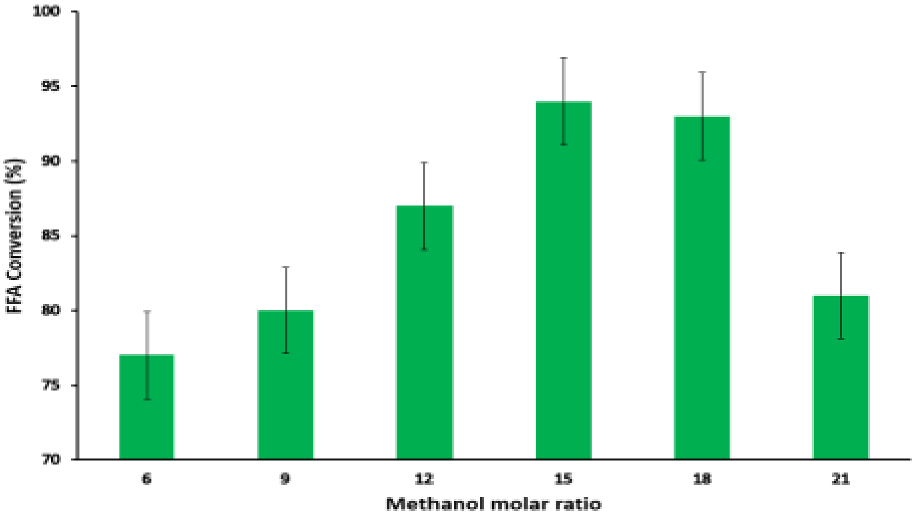
20% Ni/CaO catalyst concentration, with a range of 1 to 6 weight percent based on oil weight, was examined. Because there were fewer functional catalytic sites available, FAME conversion was decreased with a lower catalyst amount. Biodiesel conversion improved with increasing catalyst loading, but too much was unfeasible from an economic standpoint and started the reverse reaction. Figure 8 shows that the conversion reached over 90% at an ideal loading of 5 weight percent. Nevertheless, conversion decreased with increases above 5 weight percent, presumably as a result of limited reactant mass transfer within the reaction mixture. These findings suggest that catalyst loading is essential to the transesterification process's effectiveness.

**Fig. 8**. Effect of catalytic quantity on FAME amount at conditions such as
64°C temperature, 5wt% Catalyst amount, and 12:01 molar ratio

## CONCLUSION

A nickel-incorporated calcium-supported hybrid oxide catalyst 20% Ni/CaO that was made by mechanical mixing showed outstanding catalytic performance in this investigation when it came to producing biodiesel from soybean oil at 64°C. Transesterification reactions were used to test different nickel loadings and assess their catalytic activity. Under best circumstances, which included a catalyst loading of 5 weight percent, a reaction period of 5 hours, and a methanol/oil M ratio of 15:01, the highest FAME production of 93.8% was achieved. This catalyst's encouraging activity points to the possibility of lowering the costs associated with producing both catalyst and biodiesel. Thus, employing the right quantity of catalysts not only improves process efficiency but also facilitates commercial FAME synthesis that is both economical and environmentally friendly.





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