EFFECTS OF CALCINATION TEMPERATURE ON THE CATALYTIC ACTIVITY OF CAO SYNTHESIZED BY ALKALINE PRECIPITATION IN THE TRANSESTERIFICATION REACTION

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Abstract

Environmental issues and the scarcity of fossil fuel have motivated the development of research on alternative fuels such as biodiesel and ethanol. These have the advantage of being renewable and less harmful to the environment. Biodiesel has been widely studied due to its properties similar to fossil diesel. It is obtained through the transesterification reaction using vegetable oil or animal fat, alcohol and catalyst. The aim of this work is to synthesize calcium oxide catalyst and verify its catalytic activity in the transesterification reaction. Alkaline precipitation from an aqueous solution of calcium chloride was used to prepare crystalline calcium oxide. Samples calcined at 750, 850, and 950 °C were tested in three reaction cycles for the synthesis of fatty acid methyl esters. All the obtained ceramic powders showed catalytic activity, and methyl ester yields reached 65-91% in all three syntheses. In terms of reusability, the catalyst calcined at 850 °C showed methyl ester yields higher than 85%, proving advantageous over the other samples. These results were associated with the loss of active species by leaching and with the dissolution of CaO. Kinematic viscosity values in the range of 1.9-6.0 mm^2/s at 40 °C were obtained, except for methyl ester produced with CaO calcined at 750 °C. The catalytic activity analysis confirmed the superiority of CaO calcined at 850 °C to produce methyl esters.

Introduction:-

The entire world has observed a variety of weather events that demonstrate that something is wrong today. Scientists explain that the global average temperature is 1 °C higher than it was 200 years ago, owing to higher concentrations of greenhouse gases in the atmosphere (Climate change evidence and causes 2020), mainly carbon dioxide (CO_2) produced by fossil fuel combustion. Therefore, an urgent reduction in the consumption of fossil fuels is needed.

The use of renewable energy resources has been resorted to for the mitigation of greenhouse gas emissions (Silalahi et al. 2021). Presently, bioethanol and biodiesel stand out as renewable resources with high potential to be used as a substitute for fossil fuels. According to Renewables 2020 (2020), both the production and consumption of biofuels have increased over the ten-year period 2009-2019. Biodiesel is a fuel obtained through transesterification reactions in which vegetable oil or animal fat in the presence of an alcohol (usually methanol) and a catalyst are transformed into alkyl esters (Knothe et al. 2004). It offers advantages over petrol diesel, such as non-toxicity, greenhouse gas reduction, pollutant reduction (carbon monoxide, particulate matters, hydrocarbons, and smoke), lower sulphur and aromatic content, high lubrication properties, high flash point, biodegradability, sustainability and growth of rural manufacturing jobs (Suresh et al. 2018; Gebremariam and Marchetti 2018; Demirbas 2009; Kumar and Saluja 2020. Moreover, blends of biodiesel and petrol diesel can be prepared and used in engines without any modifications (Suresh et al. 2018), and various resources are available for biodiesel production (Gebremariam and Marchetti 2018). Disadvantages of using biodiesel include higher NO_x emissions (Demirbas 2009; Kumar and Saluja 2020) and high feedstock costs (Kumar and Saluja 2020).

Environmental and social issues alone would suffice to justify investments in biodiesel production. Still, the depletion of natural fossil resources reinforces the demand for alternative fuels (Suresh et al. 2018). Based on such facts, governments around the world have implemented energy security policies and incentives for the biodiesel market (OECD/FAO 2020). In Brazil, an increase in biodiesel production is anticipated mainly because of the RenovaBio program by which the government is committed, among other responsibilities, to reducing greenhouse gas emissions. In 2025, 781,560 m^3 of biodiesel were produced throughout Brazil (National Agency of Petrol, Natural Gas and Biofuels 2025). Vegetable oil, animal fat, and cooking oil waste are raw materials used to synthetize biodiesel. Sunflower, rapeseed, soybean, peanut, and palm are examples of edible feedstocks, while linseed, neem, jatropha, and karanja are examples of non-edible feedstocks (Ambat et al. 2018). Soybean oil is a major feedstock used to obtain biodiesel in many countries, such as the United States, Brazil, Argentina, and Paraguay (OECD/FAO 2020).

Transesterification of oils and fats is the most well-known process for biodiesel production (Jamil et al. 2018). In this case, triglycerides react with alcohol (methanol or ethanol) in the presence of a catalyst which can be homogeneous (sulfuric acid, sodium hydroxide, and potassium hydroxide) or heterogeneous (Na_2SiO_3, CaO, and CaO-ZrO_2). Furthermore, there is the possibility of biocatalytic transesterification using an immobilized lipase enzyme. More recently, alternative catalysts, ionic liquids ([Ch][OH] and [Ch][OMe]) (Jamil et al. 2018) and biomass (waste shell and ash) (Abdullah et al. 2017) have been developed to mitigate disadvantages concerning the aforementioned substances and materials.

Calcium oxide (pure, supported or mixed with other metallic oxides) can be used to produce biodiesel (Mazaheri et al. 2021). Commercial powder, nanoparticles and natural sources are other ways to obtain a pure CaO catalyst. Bimetallic oxides composed of CaO and other metals increase the basic properties of the catalyst and improve its stability. The efficiency of CaO-based catalysts in transesterification reactions depends on parameters such as catalyst reusability, calcination temperature, catalyst amount, alcohol:oil molar ratio, basic sites, water content, support, and free fatty acid content.

In view of the above, this study drew on the precipitation process to obtain calcium oxide at different calcination temperatures for the synthesis of biodiesel from commercial soybean oil and methanol. The effects of calcination temperature and catalyst reuse on biodiesel quality were investigated.

Materials and Methods:-

Synthesis and characterization of calcium oxide ceramic powder

Boiled and distilled water was used to prepare NaOH and $CaCl_2$ solutions. The alkaline solution was added to the salt solution, and the mixture was continuously stirred at 80 ºC. The precipitate obtained was filtered, washed with both distilled water and ethanol, and dried in an oven at 80 ºC. Three samples of ceramic powder were prepared at different calcination temperatures, namely 750, 850 and 950 ºC, to obtain crystalline materials and different microstructures that could produce some effect on the catalytic activity in the transesterification reaction. The heating rate was 10 ºC/min, and all samples were maintained in a muffle furnace for 4 h under air atmosphere. Subsequently, the materials were crushed and stored in plastic bags.

For its characterization, the material was analyzed by powder X-ray diffraction on a Shimadzu RXD6000 diffractometer using Cu-Kα radiation (40 kV and 30 mA) in the 2θ range between 10 and 70º and a step size of 0.02º to determine the crystalline phases present.

Synthesis and characterization of fatty acid methyl esters (FAME)

Commercial soybean oil and methanol, at a 1:6 molar ratio of oil to methanol, and a calcium oxide catalyst at 1.0% (w/w) were used to synthetize FAME (i.e., biodiesel). The reactor consisted of a three-neck glass flask coupled to a thermometer, a condenser, and a mechanical stirring rod that was submerged in a water bath. The reaction mixture was maintained under stirring for 4 h at 65 ºC. After cooling, the mixture was centrifuged, and the liquid part was transferred to a decanting flask for phase separation. The lower phase (glycerin) was collected, and the upper phase was washed with distilled water and then dried in an oven at 80 ºC for 2 h. Magnesium sulfate was added to this material, which was filtered after agitation and stored in a glass bottle.

After the first synthesis, another two were performed reusing the catalyst (the solid part), which was collected after centrifugation of the reaction mixture from the previous synthesis. Therefore, three samples of methyl ester were prepared for each sample of ceramic powder obtained at the temperatures under study.

For the characterization of the material, the FAME content was determined, and viscosity measurements were made. The FAME content was obtained as per the European standard (EN 14103), using an HP7820A gas chromatograph equipped with a flame ionization detector, and a Supelcowax10 capillary column with a film thickness of 0.20 µm, L x I.D. 15 m x 0.20 mm. Hydrogen and methyl heptadecanoate were used as a carrier gas and as an internal standard, respectively. The process yield (Y) was calculated using Equation 1 (Yee et al. 2011).

$$Y = \frac{\sum weight\ of\ methyl\ esters\ (g)}{\sum weight\ of\ oil\ used\ (g)} \times 100\% \qquad \text{Equation 1}$$

Viscosity measurements were taken in a Cannon-Fenske capillary viscometer, in water bath with temperature setting and a timer. Three flow times at 40 ºC were measured for each FAME sample.

Results and Discussion:-

Figure 1 shows X-ray diffraction patterns of the ceramic powder samples obtained at different calcination temperatures.

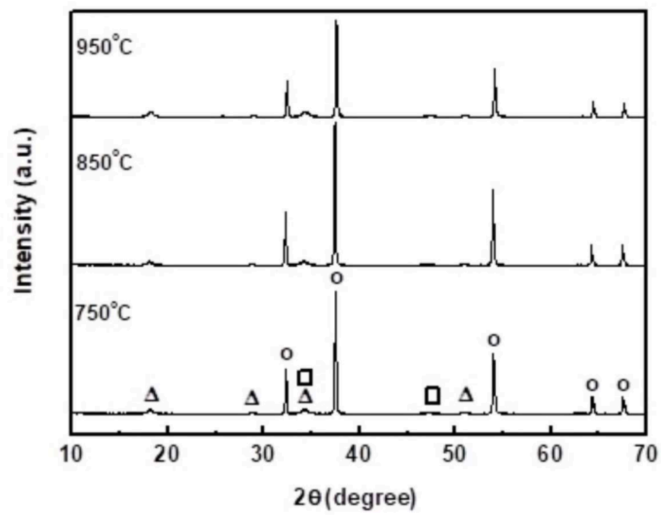

Figure 1:- XRD patterns of the ceramic powders calcined at different temperatures.

o - CaO; **Δ** - $Ca(OH)_2$; □ - $CaCO_3$

CaO diffraction peaks (JCPDS file 77-2376) were identified in all patterns, as can be seen in the diffractograms (Alonso et al. 2010), thus confirming the synthesis of this crystalline oxide. Furthermore, the results indicated the presence of calcium hydroxide and calcium carbonate in the material at the three calcination temperatures (750, 850, and 950 ºC). 2θ diffraction peaks attributed to the hydroxide phase (JCPDS file 84-1264) and the carbonate phase (JCPDS file 76-606) were weak and wide, probably revealing the material's low crystallinity. Granados et al. (2007) proved that CaO exposed to room air is partially hydrated and transformed into the corresponding hydroxide, and reported that after ten days of such exposure, calcium hydroxide and calcium carbonate diffraction peaks were observed instead of calcium oxide diffraction peaks. In the present study, the samples were exposed to room air for a few minutes during the procedures of crushing, measuring the mass, and placing the powder into the three-neck glass flask, which allowed for hydration and carbonation of the samples and, consequently, formation of hydroxide and carbonate.

The analysis of the results regarding the synthesis of methyl esters considered two aspects: the FAME yield (Figure 2), which refers to the catalytic activity of calcium oxide; and the process yield (Figure 3), which is associated with the initial amount of oil and the amount of FAME obtained in the process according to Equation 1.

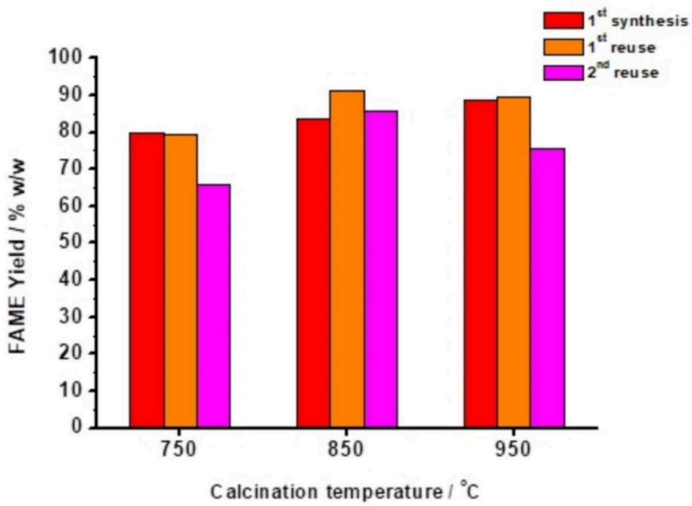

Figure 2:- Catalytic activity of CaO calcined at different temperatures for the 1st synthesis and the two reuses of the catalyst.

Catalytic activity was observed for the CaO calcined at the three temperatures for the three syntheses performed (1st synthesis and the two reuses) (Figure 2), with FAME yields reaching 65-91% (w/w). Commonly, calcium oxide is obtained by thermal decomposition of calcium compounds, such as hydroxides and carbonates. The temperature at which oxide is formed will depend on such precursor compounds, as well as on the heating rate and the atmosphere (Marinković et al. 2016). Additionally, precursor salts used to prepare calcium hydroxide influence the catalytic activity of calcium oxide obtained in a transesterification reaction (Alonso et al. 2010). When an aqueous solution of calcium nitrate was used in the alkaline precipitation, the synthesized CaO was almost inactive in producing FAME, while the use of an aqueous solution of calcium acetate provided the catalyst synthesis with a high rate of FAME formation.

In the present study, the results indicated that the CaO produced from calcium chloride as the precursor salt for calcium hydroxide precipitation showed catalytic activity in the transesterification reaction. Other important aspects to consider were the heating rate (10 °C/min) and the atmosphere (air) used to obtain the catalyst, which interfere with the calcination temperature. Alonso et al. (2010) investigated the thermal decomposition of $Ca(OH)_2$ in a 20 vol.% O_2/Ar atmosphere at a heating rate of 10 K min^{-1} and at up to 800 °C using temperature-programmed evolved gas analysis – mass spectrometry (EGA-MS) and observed that the formation of synthetized CaO from nitrate and acetate precursor salts occurred at 432 °C and 452 °C, respectively. Thus, to synthetize alkyl esters, they used ceramic powders calcined at 800 °C, for 1 h, in a 20 vol.% O_2/Ar atmosphere, and at 10 K min^{-1}. In the present study, under the above mentioned conditions, the lowest calcination temperature at which calcium oxide was obtained was 750 °C for 3 h, confirming the feasibility of the synthesis process using calcium chloride as a precursor salt that produces a catalytically active material in the formation of FAME.

The comparison of the three samples evinced that catalytic activity depends on calcination temperature (Figure 2). XRD patterns indicated the same crystalline phases for the three samples, demonstrating that properties other than crystallinity probably contribute to the catalysis of the calcium oxide synthesized under the aforementioned conditions. The calcium oxide calcined at 850 ºC and 950 ºC showed catalytic activity superior to that obtained at 750 ºC. Changes in particle size, porosity, and specific surface area occur when samples are subjected to heating (Kingery et al. 1975) and can affect the catalytic properties of the material prepared (Badnore et al. 2018). Although such structural information was not investigated in the present study, appropriate surface area conditions apparently were not achieved at 750 ºC. Meng et al. (2013) observed that catalytic activity significantly depends on the calcination temperature of Ca/Al composite oxide. Among their samples calcined at 120, 400, 600, 800, and 1000 ºC, the highest yield of FAME was obtained for the sample calcined at 600 ºC, which had the highest surface area, namely 27.36 m^2/g.

In terms of the reusability of the catalysts, the calcium oxide obtained at 850 ºC demonstrated an advantage over the other samples because the FAME yields were greater than 85% in this study. The reuse of the catalyst causes the loss of active species by the leaching process and the dissolution of CaO in the reaction media, consequently decreasing the activity of the catalyst in the transesterification reaction (Granados et al. 2007). The solubility of calcium oxide is relatively low in methanol, 0.04 mg mL^{-1} at 333 K (Granados et al. 2009), and this is due to the formation of calcium methoxide, $Ca(OCH_3)_2$. As the reaction proceeds, the glycerol produced in the medium contributes to the formation of another species, calcium diglyceroxide, $Ca(C_3H_7O_3)_2$, which is as catalytically active as calcium metoxide. In this way, these species are involved in the homogenous catalysis to produce FAME.

Loss of catalytic activity in the reuse of nanocrystalline CaO in a transesterification reaction was observed for a catalyst prepared using thermal decomposition of calcium carbonate at 1000 ºC for 2 h and, for three consecutive reaction cycles, the conversion values decreased from 89.79% to 75.21% (w/w) (Badnore et al. 2018). According to said authors, the adsorption of CO_2 and moisture on the surface of CaO during reaction cycles caused a drop in the catalytic activity. Although these results agree with those obtained for the samples calcined at 850 and 950 ºC in the present work, the main reason for the observed lower catalyst activity, presumably, was the dissolution of CaO in the medium and the loss while transferring the material to the subsequent synthesis. Simbi et al. (2022) investigated the reusability of the bi-functional CaO/Al_2O_3 catalyst in the transesterification reaction to produce FAME from waste cooking sunflower oil and methanol, and they observed loss of catalytic activity after five cycles of reuse. Even with CaO associated with another oxide, processes such as leaching and calcium diglyceroxide formation were also used to justify these results.

The results indicated that the formation of metoxide and diglyceroxide species through calcium dissolution may be related to the lower catalytic activity of the three samples in the second reuse. In these cases, a higher amount of material was lost in the two previous syntheses. However, such species may have contributed to maintaining the catalytic activity (1st synthesis and 1st reuse) of the catalysts obtained at 750 ºC and 950 ºC or to increase it, as observed in the catalyst obtained at 850 ºC, which showed the lowest FAME yield variation among the three samples investigated. The reusability results are likely associated with the morphological properties of the synthesized catalyst (Oueda et al. 2017). In catalyst reuse, specific surface area, pore volume and average pore diameter can be modified due to interactions of calcium oxide with different molecules of the reaction medium and, consequently, the catalytic activity will change as well. Thus, the treatment at 850 ºC may have led the sample to a microstructure that favored a higher FAME yield and leaching in smaller proportion when compared to the other samples treated at 750 ºC and 950 ºC.

Figure 3 displays the process yield results determined by Equation 1, which considers the amounts of oil and FAME produced in the synthesis. In this regard, factors related to the catalyst and to the various steps taken to produce FAME, from synthesis conditions to material purification, were considered when calculating the process yield. Among the factors related to the steps of FAME production are: loss of catalyst in transferring the material to the subsequent synthesis, catalyst dragged by the glycerin formed, emulsion formation, washing with water of the produced FAME, and drying with magnesium sulfate. Process yields (% w/w) in the range of 33% (950 ºC) and 60% (750 ºC), both for the 1st reuse, were obtained. In this vein, knowing the possible causes that contributed to the decrease in the process yield is of paramount importance so that measures to improve the process can be planned.

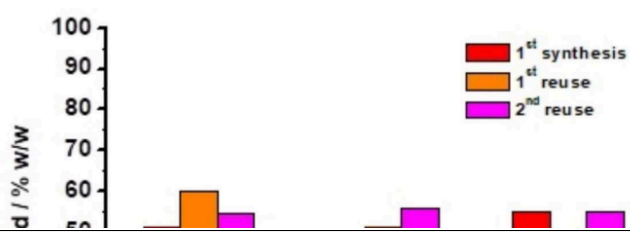

Figure 3:- Yield in the production of FAME considering the initial amount of oil and the amount of FAME obtained at the end of the process for CaO calcined at different temperatures for the 1st synthesis and the two reuses of the catalyst.

Viscosity results are shown in Figure 4. Badnore et al. (2018) worked with refined soybean oil and found a kinematic viscosity value equal to 27.06 mm^2/s at 40 °C. After the transesterification reaction, they observed a value of 3.91 mm^2/s (40 °C) for the synthesis with nanocrystalline CaO. The American standard (ASTM D6751) establishes that the kinematic viscosity of a produced biodiesel must be in the range of 1.9-6.0 mm^2/s at 40 °C. The values obtained in the present study are in line with this standard, except for the FAME produced in the reaction catalyzed by the sample calcined at 750 °C in the 2nd reuse. These viscosity results agree with the FAME yield results since the most viscous sample (6.5 mm^2/s) had the lowest FAME yield (65.9%). The higher viscosity value observed is probably due to triglyicerides not converted partially or completely by the catalyst.

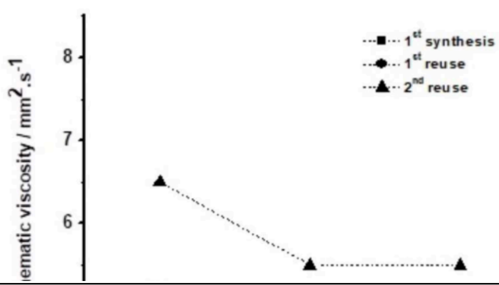

Figure 4:- Kinematic viscosity at 40 ºC of FAME samples produced with CaO calcined at different temperatures for the 1st synthesis and the two reuses of the catalyst.

The results obtained in this work and those found in the literature demonstrate the potential of calcium oxide to be used as a catalyst in transesterification reactions. Nevertheless, there are some aspects that still need to be investigated to make it attractive for such an application. According to the present study, calcium oxide can also be synthesized from the alkaline precipitation of an aqueous solution of calcium chloride, and the ceramic catalyst produced must be exposed to the atmosphere for as short a time as possible so that calcium hydroxide and calcium carbonate are not formed. Crystallinity as well as H_2O and CO_2 contamination were observed in the samples calcined at the three temperatures under study, and because all samples presented catalytic activity, they could be used to produce FAME.

The results concerning the transesterification reaction indicated that the group of three syntheses using calcium oxide obtained at 850 ºC showed a superior FAME yield with the highest catalytic activities in the two reuses. On the other hand, the process yield for this group was not considerably better than the yield from the other groups studied. Still, the steps of synthesis and purification of FAME can be planned according to the factors mentioned above to obtain superior results in this yield as well.

The potential of calcium oxide-based catalysts in transesterification reaction is well known as are the challenges to improve the efficiency of these materials in order to make them viable for the industrial production of biodiesel. Further research is required to develop CaO catalysts supported on inorganic (alumina, silica, and zeolites) (Mazaheri et al. 2021) or organic (biomass waste) (Tang et al. 2018) matrices to produce materials with high catalytic activity in transesterification reactions and lower the cost of biodiesel production.

Conclusion:-

Calcium oxide was obtained from the alkaline precipitation of an aqueous solution of calcium chloride. The material showed crystallinity when calcined at 750, 850, and 950 ºC in addition to catalytic activity in the transesterification reaction using commercial soybean oil and methanol at the 1:6 molar ratio of oil to alcohol. The catalyst was tested in two reuses in the transesterification reaction. The samples calcined at the three temperatures under study led to FAME yields in the range of 65-91% (w/w) for the 1st synthesis and the two reuses. The process yield results revealed values between 33% and 60% (w/w), indicating that measures need to be adopted to reduce catalyst and FAME losses during the process. Therefore, the analysis considering the results of the catalyst synthesis, the FAME yield and the process yield demonstrated the advantage of CaO calcined at 850 ºC in the transesterification reaction.

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