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Biodiesel Production Mediated by Eggshell Catalyst : A Review of the Literature

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Abstract -*An increase in demand and the constant growth of crude oil prices globally and the massive consumption of petroleum reserves resulted in creating alternate and more eco-friendly ways to solve this problem. Biodiesel production has been the most viable and promising approach to replace petroleum-based fuel. Biodiesel is preferred due to its high lubricity, biodegradability, high combustion efficiency and low toxicity. In recent years, there have been various efforts made to make biodiesel a commercial fuel. This paper reviews on enhancement and commercial improvement of biodiesel production by using non-conventional heterogeneous catalysts like eggshell in transesterification reaction. Heterogeneous base catalyst solves various problems caused by homogeneous catalysts as they are*

(a) Non-corrosive

(b) Recyclable

(c) Non-toxic

(d) Improve the separation and purification steps.

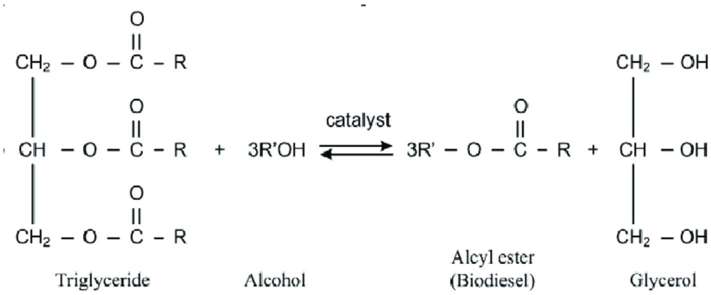
The viability of catalysts, several effects on catalyst and characteristics of catalysts are also concentrated in this article. The consumption of eggshell as a catalyst for biodiesel production provides a cost-effective and eco-friendly way of reusing solid eggshell waste and also reduces the price of biodiesel, making it competitive with petroleum diesel.

Key Words: **eggshell catalyst, biodiesel production, heterogeneous base catalyst, reusability, sustainability**

1. INTRODUCTION

Because of the energy and global warming crisis, development of renewable energy has been focused worldwide. Biodiesel is quite possibly the most likely alternative energy since it is sustainable and harmless to the ecosystem [1]. Furthermore, fossil fuels are unfriendly to the environment because they contribute to poisonous gas emissions from car and industrial internal combustion engines, which contribute to global warming, acid rain and depletion of ozone layer [2]. Biodiesel (fatty acids methyl, FAME) can be derived from the transesterification of triglycerides (the main component of vegetable oils or animal fats) with a short chain alcohol (mainly methanol). It has become popular as a possible alternative to fossil fuels. The main advantages of this fuel are that its properties and performance are similar to conventional diesel fuels Currently, homogeneous process catalyzed by sodium or potassium hydroxide is a common and efficient method for biodiesel production though the removal of these catalysts is costly, time consuming and generates large amount of waste water [3]. To overcome these impediments, heterogeneous catalyst explored as an alternative to the conventional homogeneous catalysts. Zeolites and metal catalysts are examples of heterogeneous solid acid and base catalysts [4].

Transesterification is the process in which the vegetable oil/animal fat is subjected to chemical reaction with alcohol and using solid base catalysts to produce glycerol and ester. The simplified form of this chemical reaction is given as:



Where R1, R2, R3 are fatty acid chains.

Oxides of magnesium and calcium oxide have been tried as solid base catalyst owing to their easy availability, low cost and non-corrosive nature. The research was also reported about the good performance of eggshells among the catalyst selection, in transesterification as comparison to golden apple snail and Meretrix Venus shells, chicken egg shell has the highest calcium content and the largest surface area. The research was carried out with used eggshells to undergo calculations-hydration-dehydration treatment to obtain calcium oxide catalyst. Eggshell calcined above 800 °C was found to be the most active with a biodiesel yield range of 97-99 %. This was attributed to the formation of crystalline CaO as the active phase used for the transesterification [13]. A research on emulsification of biodiesel with water to investigate its influence on the overall diesel engine was reported [6]

This study addresses the following objectives: one is to synthesize a potential catalyst from eggshell waste and to identify most effective calcination conditions for the calcined eggshells derived catalyst. The second objective is to determine the effect of the methanol to oil ratio, the weight of the catalyst and the reaction temperature using the most effective calcined eggshell conditions derived catalyst to form biodiesel [8].

2. BIODIESEL PRODUCTION PROCESS

Achanni et.al worked on the application of eggshells waste as a heterogeneous catalyst for biodiesel production. Duck and chicken waste eggshell were used as raw material for biodiesel production and eggshells were calcined at 600-900°C for 4h to convert calcium carbonate to calcium oxide. To research the physical and chemical structure of the catalyst, XRD, XRF, SEM, and BET methods were used to characterize its physical and chemical properties. The authors studied in detail the chemical activity of the eggshell catalyst in transesterification reaction and the various effects like reaction time, reaction temperature, methanol/oil ratio and reusability of the catalyst. The authors studied the optimum conditions to yield biodiesel from palm oil and the conversion rate was about 92 % and 94% for duck and chicken eggshell under specific conditions of 65°C reaction temperature, 4h reaction time, methanol to oil molar ratio 9 and catalyst loading 20wt% with 1 atm pressure. Experimental results conclude catalyst reusability was up to 4 times (80.6) and after that loss of its catalytic activity takes place and fuel properties of the biodiesel obtain meet all standard conditions that make it feasible for consumption. Cao catalyst from eggshell was stable and had impressive catalytic activity during the reaction which makes it a potential alternate catalyst for the making of low-cost Biodiesel.

Nasar Mansir et.al reviewed Bifunctional catalyst derived from waste eggshell for biodiesel production from high FFA waste cooking oil. The authors discussed the global energy crisis and depletion of reserved fossil fuels and growing demand for energy consumption resulted in a search for alternate fuel like Biodiesel which is more sustainable, biodegradable, less toxic and eco-friendly than conventional fossil fuels, but making of biodiesel from the conventional homogeneous catalyst is not feasible due to its separation problem which makes it costly, toxic and non-biodegradable. So, the authors discussed various solution to counter this problem and create an effective biofuel. They studied the use of waste cooking oil (WCO) can reduce half the cost of fatty acid methyl ester (FAME) production and use of heterogeneous catalyst from eggshell can reduce the ecological problem and its cost-effective can solve the various problem faced by the conventional homogeneous catalyst. The authors also covered a detailed study of biodiesel manufacturing from various types of catalyst like homogeneous, heterogeneous, solid base and modified eggshell catalyst and also discussed alternate techniques for biodiesel manufacturing from methods like conventional reflux and autoclave reactor.Commercialization of biodiesel production from heterogeneous catalysts can solve the global energy need and industrial production at a low cost.

Ali A. Jazie et.al studied Eggshell as an environmentally friendly catalyst for rapeseed oil transesterification. The authors investigated the viability of calcined egg shell as a catalyst for rapeseed oil transesterification reaction for biodiesel production and after calcination, surface area of the catalyst is increased, thus leading to improved catalytic activity. The maximum biodiesel yield obtained was about 96% under given optimum condition (i.e., at 900°C for 2h with a 9:1 molar ratio of methanol to oil and 3 wt.%) and biodiesel yield was optimized using response surface methodology (RSM). By performing multiple regression analysis, a quadratic polynomial equation was formed for biodiesel. Catalyst characterization was done by X-ray diffraction (XRD) and Fourier transform spectrometry (FTIR). Experimental results demonstrate the reusability of the catalyst is about 14 times with no apparent loss of activity and after that it loses activity gradually and this property will reduce the cost of catalyst so making it feasible to be used in large scale biodiesel production.

Supriya B.Chavan et.al reviewed the fuel properties of biodiesel made from jatropha curcas oil and waste eggshell were investigated. The authors extracted calcium oxide (Cao) from eggshell to use as a catalyst, and Jatropha curcas oil was used as a raw material for biodiesel production. The eggshell was calcined at 900°C for 2.5 hours to obtain the highest quality calcium carbonate. Catalyst characterization was done by XRD, FTIR and DTA-TGA methods. The maximum biodiesel yield obtained was about 90% under given optimum condition (i.e., at 60°C for 2.5h with a 9:1 molar ratio of methanol to oil and 2 wt.%) and the parameters of yield biodiesel like acid value, density, kinematics viscosity, flash point, cloud point, fire point, pour point, cetane number and calorific value were of ASTM D6751 standard and also carbon (%), water (%), nitrogen (%) and hydrogen (%) were also evaluated and were within the limit of ASTM standard. Catalyst reusability was approximately 6 times without significant loss of operation, indicating that the biodiesel generated is cost-effective and of high quality for commercial biodiesel production.

Solid oxide derived from waste shells was studied as a biodiesel catalyst by Jutika Boro et.al. Mullusk and waste eggshell are the richest sources of calcium carbonate, and when these shells are calcined at optimal conditions, calcium carbonate is formed, and this solid oxide can be used as an alternative catalyst to conventional homogeneous catalyst for biodiesel manufacturing. Solid oxide catalyst is not only cost-effective but also eco-friendly and biodiesel production heavily depends on factors like temperature, time, the catalyst used and the molar ratio of methanol to oil. The authors studied the characteristics of various shells in transesterification reaction and catalysts were highly stable and active. Re-consumption of these waste materials reduces cost and also is sustainable for commercial production of biodiesel.

Annam Renita et.al carried out this experiment analysis to study production of non-traditional heterogeneous base catalyst made from waste egg shells, as well as a contrast and kinetic analysis of conventional homogeneous and heterogeneous base catalysts for biodiesel production from vegetable oil. For the preparation of renewable catalyst, the eggshell was calcined at a high temperature ranging from 700 to 1000°C for 2-4 hours. The calcined eggshell and commercial catalyst were trans-esterified with palm oil, sunflower oil, coconut oil for manufacturing of biodiesel and properties such as density, kinematic viscosity, flash point, acid value, calorific value and reusability of produced biodiesel are compared for both renewable and commercial catalyst. A study found that the yield of biodiesel produced from egg shell catalyst is comparable to commercial catalyst with a yield of 95% and eggshell catalyst was used four times before the decline in its activity. So, it was concluded that biodiesel production from eggshells is a feasible, cost effective and renewable alternative.

Ziku Wei et.al carried out this analysis to study variation in properties and characterization of eggshell catalysts and their impact on biodiesel production. The feasibility of the eggshell catalyst was studied in the triglyceride transesterification process for production of biodiesel. The effect of different calcination temperatures on eggshell catalyst behavior and reusability was investigated. From SEM images, it was discovered that that below 700°C the size and shape of the particle was similar to a normal eggshell and above 800°C the size of the particle decreased and the shape became more regular. The study also discovered that at higher calcined temperatures, the yield of eggshell catalyst is high, with a yield of 97%-99% above 800°C and 30% below 600°C.They also found the yield of biodiesel increases with an increase in methanol/oil ratio and reaction time. It was found that eggshell derived catalyst can be repeated used 13 times with no apparent loss. So, it was concluded that the eggshell catalyst is an effective, cost-competitive and eco-friendly alternative to commercial catalysts

Y.C.Wong et.al reviewed the methodology of producing biodiesel using eggshell derived catalyst by employing used cooking oil. They calcined the eggshells which were later used as a catalyst for the transesterification process. The eggshell catalyst was subjected to different operating conditions and parameters like by means of a muffle furnace, a tube furnace with and without the supply of Nitrogen gas. They also remarked on the consequences of methanol to oil ratio for the formation of Biodiesel. Due to the formation of calcium methioxide, the biodiesel yield increased as the methanol volume increased. Until a certain limit, the reaction was triggered due to calcium methioxide for methanol to oil ratio. They observed the changes in the biodiesel formation by varying the catalyst concentration, which also accelerated the volume of biodiesel up to a certain extent. They also concluded the effect of temperature on the biodiesel yield, which favored the production at higher temperatures, if the temperature rises even higher, results in solubility of methanol into triglycerides and not influencing the biodiesel yield above 65-degree Celsius. The reaction temperature profile properties were tabulated for the formation of biodiesel using the eggshell catalyst.

Ayoola A. Ayodeji et al. investigated the production of biodiesel using conventional CaO catalyst and catalyst derived from calcined and uncalcined waste eggshells, which were then used for the transesterification of soya bean oil. The factors to be processed were methanol to oil ratio, catalyst concentration and time required for the reaction. X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF) spectroscopy were used to analyze the elemental composition of the catalysts. The XRF analysis was based on the principle of emission of excited elements present in the catalyst when subjected to high energy X-Rays. The properties of soya bean biodiesel products such as density, pour point, flash point, viscosity, water content and acid value were determined. They observed that there was a resemblance in the properties of conventional biodiesel to that of biodiesel produced for eggshell catalyst.

A review on the possibilities of production of biodiesel using the waste cooking oil with calcined eggshell catalyst with the addition of emulsifying process and its effect on diesel engine performance was performed by Yie Hua Tan et.al. To improve the biodiesel properties for use in diesel engines, they used a biodiesel-H2O emulsion technique with two phase emulsion (water in oil) and three phase emulsion (oil in water in oil). The two-phase consisting of ethanol-biodiesel water and micro emulsion had a longer ignition delay than that of the neat biodiesel. They discovered that three phase emulsion has a higher volumetric fraction than two phase emulsion. When compared to pure biodiesel, the biodiesel emulsion method produced a higher heating value without taking water content into account. The Biodiesel-H2O emulsion was then implemented in the diesel engine process and was then compared with the results of pure biodiesel and its performances. The Biiodiesel-H2O emulsion inhibited characteristics like enhancement of ignition quality, improving combustion of biodiesel fuel, and higher brake specific consumption. The emulsification method has proved to be beneficial for reducing Nitrogen Oxide (NOx) emission and upgrading the combustion efficiency of biodiesel. They reduced the NOx, Particulate Matter (PM), toxic and smoke emission levels than other biodiesel types.

N P Asri et.al worked on the production of biodiesel, where the main purpose was to utilize CaO contained in eggshells into a solid catalyst instead of homogeneous catalyst from used cooking oil. The eggshells had small porosity, high CaCO3 content, which provides opportunities for development of heterogeneous catalysts. The used cooking oil (UCO) was analyzed with an FFA content of 4.4946% by the AOCS method. UCO was then pre-treated, filtered and heated at 100-105ºC for removal of water content. To reduce the FFA content in the UCO, it was treated with an adsorption process using coconut choir powder (CCP). The best percentage of CCP to use was 7%. The eggshells were crushed, soaked and rinsed with warm water and dried at 110 ºC for 24h, which were then powdered and sieved on 100 mesh and retained on 200 mesh. Their eggshells were calcined at 900 ºC for 4h in a muffle furnace. The eggshell catalyst (ES-CaO) was obtained and held in a desiccator with silica gel to prevent water and CO2 from contacting the catalyst. The transesterification was taken out in a batch type reactor fitted with a reflux condenser. The process was carried out at 65 ºC with a UCO: methanol ratio of 1:15, with an ES-CaO amount of 3-7% for 2-7 hours at constant speed. The mixtures were filtered at room temperature to isolate the catalyst. In a separating funnel, the liquid phase was segregated. BET and XRD analysis were used to examine the biodiesel collected. BET analysis gave results as the surface area of calcined eggshells was 125.927, which was greater than that of crab shell, mud dam shell and commercial CaO. XRD analysis showed that calcined eggshells at 900 ºC converted CaCO3 into CaO, where the active ingredient is CaO.

The results obtained were that the factors affecting the biodiesel production were the FFA content and moisture content in the UCO. The water content accelerates the hydrolysis reaction and reduces ester formation and therefore the water content should not be more than 0.5% in the triglyceride. The FFA content affects the production as more FFA will result in soap formation. The FFA content was reduced to 0.2079% using CCP. The catalyst amount varied from 3-7% at 65 ºC to 1h at 1:15 oil to methanol ratio. The amount of biodiesel produced increased as the catalyst was loaded. 6% was considered to be the optimum catalyst loading and was used for further research. The reaction temperature was also taken into consideration, by changing it from 1 to 7 h. Mass transfer was slow at 1h and equilibrium condition was obtained at 7h giving 75.92% of biodiesel yield.

N. Sh. El-Gendy et.al used heterogeneous solid catalysts as reusable, less corrosive, safer, more environmentally friendly properties as an alternative to the homogeneous catalysts in the manufacturing of biodiesel. In organic solvents, CaO in eggshells has a low solubility, but it has fair activity under mild conditions. The study used three waste cooking oils: waste frying oil (WFO), waste sunflower oil (WSFO), and waste corn oil (WFCO) and were centrifuged and filtered at 105 ºC at 2h. the eggshell catalyst was calcined at 800 ºC for 2h. the biodiesel obtained was analyzed under gas chromatography analysis. The properties like density, kinematic viscosity, acid number and saponification value were evaluated.

The type of oil used in the process had little impact on the yield, but the methanol to oil ratio, catalyst heating, and reaction time had the greatest impact. The catalyst loading had a negative effect on the yield, while the methanol to oil ratio directly affected the yield. Biodiesel produced from WFO had higher fatty and methyl esters (FAME) yield than the other waste oils. The iodine value shows the unsaturation degree, the degree of unsaturation affects the fuel oxidation tendency, which then influences the flow properties. The cetane number rises with chain length and falls with unsaturation.

When compared to the petrol diesel, the advantages were that biodiesel obtained from eggshell catalyst were free of Sulphur which doesn't corrode the mechanical parts. they also have a higher flashpoint, which makes it less flammable and safe for transportation and viscosity was competitive. They concluded that CaO obtained from waste eggshells gave higher biodiesel yield.

Sulaiman et al investigated calcined solid coconut waste and eggshells to develop a heterogeneous catalyst. The homogeneous catalyst has poor thermal stability, reduces the catalytic efficiency, increases viscosity and results in gel formation. Due to the stated properties, there was a shift towards heterogeneous catalysts which improve reaction during catalytic activity. The coconut to eggshell waste was taken to be about 5:1%. The FAME yield obtained was about 81% at 65 ºC for 3h with a methanol to oil ratio of 24:1. Both wastes were kept in the oven at 110 ºC overnight before being calcined at 900 ºC for 4 hours. SEM, XRD, and FTIR analysis were used to characterize the catalyst. The reaction was carried out in a shake flask for 3h at 500 rpm and was further carried out at 6000 rpm for 10 min. The upper layer obtained was biodiesel.

The response surface methodology and central composite design were used to investigate the interactions between coconut and eggshell waste and to predict the best conditions for FAME yield. Analysis of variance was used to find out the results. The maximum FAME yield was 81% at 5 wt.% of coconut waste and 1 wt.% of eggshell.

Anthill eggshell Ni-Co mixed oxides-based catalyst was prepared from co precipitation by Yusuff A. S. et al. the eggshell catalyst was obtained by calcination at 110 ºC for 2h. Co (NO3)2.6H2O and Ni (NO3)2.6H2O were weighed and mixed in 69.7 wt.%, 17.4 wt.%, 8.6 wt.% and 4.3 wt.% proportion of eggshell, anthill, cobalt nitrate hexahydrate, nickel nitrate hexahydrate, respectively and fed into a beaker. Suspension was formed by adding water to the mixture. The pH of the resulting slurry was changed to 8.0 by adding 0.1 M Na2CO3 solution and then aged for 2 hours with stirring in a fume hood at 80˚ c the solution was then filtered through filter papers and oven dried for 12 hours at

110° c. The resulting dried mixture was therefore calcined in a muffle furnace under static air conditions at a temperature of 1000 ºC for 4 h.

The transesterification reaction was taken out in a batch reactor fitted with condenser and thermometer. The conditions were 3-7 wt. % of catalyst loading, reaction temperature of 50-70 ºC, reaction time of 2-4h and methanol to oil ratio of 6:1 – 12:1

FTIR analysis was done to find functional groups and SEM analysis for investigate the surface topography and morphology. The raw catalyst had irregular and undefined particles with surface covered by adsorbed gases and volatile matters. The calcined catalyst was found out to be a porous material with thermally activated carbonate.

The outcomes were collected and recorded and the effect of reaction temperature on output is negligible. High reaction temperature enhances mass transfer of reactants and favors high reaction rate and high conversion. At 70 °C, the highest biodiesel yield was obtained because at 80 °C, liquid methanol preferentially forms glycerol over biodiesel. The highest biodiesel yield was obtained after 2 hours of reaction time at 70° c Excess loading resulted in an undesired saponification reaction, so the optimal catalyst loading was 3 wt.%. The effect of methanol to oil ratio on biodiesel had a linear relationship. Too much quantity of methanol could come in between the separation of products, affecting the yield. The optimum methanol to oil ratio was discovered to be 12:1. The yield of biodiesel was evaluated as 88.42% at the above stated optimum conditions.

Ngoya Tshizanga et.al researched the processing of biodiesel as a feedstock using calcined eggshell ash as a heterogeneous base catalyst. The amount of FFA is waste cooking oil was found out to be 9%. The rate of reaction of base catalyst incorporated biodiesel is greater than that of acid catalyst. A Heterogeneous base catalyst has the property of absorbing the water content in vegetable oil. The eggshell is hydrophilic in nature and can be easily removed or reused by filtration without any loss of catalyst. The most important aspect is reusability, which can lower the cost of producing biodiesel. The physical and chemical properties are necessary to be evaluated as they are required to figure out the stability for use in diesel engines. The acid level increases in biodiesel, if kept for a longer duration under storage. The cetane number in biodiesel made from saturated fats is greater than that of the unsaturated fats. The viscosity of the biodiesel must be less as it gives higher FAME yield. Viscosity affects the combustion process, smoke and emission. The flash point of biodiesel is higher than that of petroleum diesel, making it less volatile.

The optimum reaction was found out to be at 22.5:1 of methanol to oil ratio, 3.5 wt.% of catalyst loading at a reaction temperature of 65 ºC. The eggshell catalyst can be reused over 10 cycles of transesterification reaction, which can reduce the biodiesel production cost.

3. CONCLUSIONS

In conclusion, eggshell was calcined to produce the CaO catalyst for biodiesel production. At optimum conditions, it was discovered that calcined eggshell worked better as a catalyst for biodiesel processing. The catalyst was reusable.

CaO prepared from waste eggshells ES outperformed the chemical CaO in the production of high-quality biodiesel. The design of experiments was used in this study to minimize the number and expense of experiments while obtaining more information per experiment. Regression models were obtained as a result of the relation between% BDF yield and operational variables M: O molar ratio, catalyst concentration wt.%, reaction time min and mixing rate rpm. The statistical analysis showed that, within the studied experimental range, the three factors having a positive influence were; M: O, catalyst loading and mixing rate. M: O was the most important factor, followed by catalyst loading and mixing rate, in a decreasing order. However, the interactions between these factors had less significant effects. According to the analysis, the best operating conditions for transesterification with CaO prepared from ES are: 6:1 M: O molar ratio, 3% (w: w) catalyst concentration, 350 rpm mixing rate, 30-minute reaction time, and 60 degrees C, with maximum expected and experimental percent biodiesel yields of 98.8 and 97.5 percent, respectively.

The eggshell catalyst provides small porosity, high calcium carbonate content and gives opportunities for development as a heterogeneous catalyst. Briefly, this study illustrates an environmental recycling process to produce an alternative green fuel.

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