Waste to Energy Conversion from Orange Peel: Heterogeneous Catalyst Preparation and Biodiesel Production

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ABSTRACT

Renewable energy sources play a significant role in shaping the nation's economy and regulating the reliance on non-renewable sources. Non-renewable energy sources, especially the petrol-diesels, are on the verge of extinction, and utilizing these hydrocarbon fuels increases air pollution. To mitigate the challenge, biodiesel from renewable energy sources is gaining wide attention. In this study, an attempt is made to produce biodiesel from waste orange peel oil via a transesterification process. Furthermore, a heterogeneous catalyst is prepared from the waste orange peel extract by calcination at 600^{0}C for 120 minutes. The obtained orange peel oil biodiesel (OPOBD) from the transesterification process is characterized by saturated and unsaturated fatty acid compounds (FAC) and significant fuel oil properties. The maximum OPOBD yield of 86.76% is achieved, and the yield decreases with the increase in the catalyst re-usage. The results show that neat biodiesel and diesel-biodiesel blend (B20) fuel oil properties are in close agreement with the ASTM standards, and the FAC results reveal high levels of saturated FAC in OPOBD.

Keywords: Heterogeneous Catalyst, Orange Peel Biodiesel, Waste to Energy Conversion

INTRODUCTION

The energy sector plays a predominant role in shaping the economy of the nations, especially the imports and exports of petroleum-based products. Crude oil is a widely used energy resource for powering different sectors like land, water and air transportation systems, industry, and agriculture. With the increase in the global population, the utilization of non-renewable energy sources like crude oil is increasing at a steeper rate. Due to rapid consumption, the available crude oil reserves are decreasing alarmingly. Different studies (Sorrell *et al*., 2012; Click and Weiner, 2010) have shown that energy reserves, especially crude oils are on the verge of extinction.

Furthermore, the burning of non-renewable hydrocarbon fuels increases air pollution by pumping its toxic gases like unburnt hydrocarbons (UHC), carbon monoxide (CO), particulate matter (PM), sulfur oxide (Sox), nitrogen oxide (NOx), and exhaust smoke (Click R and Weiner R, 2010). These toxic gases pollute the atmospheric air as well as human health when they are continuously exposed. Environmental pollution is another significant problem that affects the entire world. Since then, pollution in the air has emerged as a significant danger to the continued existence of humans on this planet; it is sometimes referred to as an "invisible killer."

The above discussion shows that burning non-renewable hydrocarbon fuels increases air pollution. Therefore, searing for renewable and clean burning fuels is gaining importance worldwide to mitigate the current energy and environmental challenges. Clean-burning fuels obtained from different edible and non-edible oils like biofuels are considered attractive due to the capability of lowering toxic emissions when utilized for the combustion process, and biofuels are eco-friendly and sustainable. These features made biofuels one of the potential candidates to replace the existing petrol-diesel fuels.

The literature studies on biodiesel applications in different internal combustion (IC) engines reveal that; biodiesel in neat form or blended with different additives improves combustion efficiency and lower exhaust emissions. Victor *et al*. (2017) used neat rice bran oil biodiesel with different percentages of isopropanol alcohol to examine CI engine's heterogeneous combustion performance and exhaust emission. The results reveal that a 2% additive (alcohol) in rice bran biodiesel improves the combustion by improving 4.3% brake thermal efficiency and lowers the CO, NOx, and Smoke by 14%, 36%, and 27%, respectively. K Prasada Rao and BV Appa Rao (2014) investigated the low-temperature combustion in an IC engine with neat mahua oil biodiesel, and small percentages (1%, 2%, 3%, 4%, and 5%) of methanol were injected directly into the combustion chamber (CC) with a secondary fuel injection setup. The results reveal that a 3% additive mixed with biodiesel shows promise by lowering exhaust emissions and improving combustion efficiency. Kolakoti and Rao (2020) utilized preheated jatropha oil in a CI engine to test the viscosity saturation during the combustion. For this purpose, raw jatropha oils are preheated to 100 C from 50 C with a gradual increment f 10 C. The results of combustion and emission at 60 C preheating show better, and the authors concluded that viscosity saturation helps achieve better results. Another study by Aditya and Rao (2019) used palm oil biodiesel mixed with different percentages of coconut oil biodiesel. Authors observed that mixing a small quantity (3%) of coconut oil biodiesel brings a radical change in the fatty acid composition (FAC), due to which the saturated and unsaturated FAC in the palm and coconut oil biodiesel blends refines the combustion propensity and achieve low exhaust emissions. The authors also tested the combustion performance and exhaust emission with neat palm oil biodiesel fueled in naturally aspirated and supercharged diesel engines. The authors concluded that supercharging the naturally aspirated brings more benefits regarding power augment and low exhaust emissions. Kalyani *et al.* (2023) used green algae oil biodiesel (40% blend with diesel fuel) to examine the exhaust emission and combustion. The author proposed small percentage of triacetin as an additive in B40 brings better combustion and low exhaust emissions than neat diesel fuel application. Aditya Kolakoti (2022) investigated the energetic combustion performance and exhaust emission studies using two different biodiesel (neem and waste cooking) mixed with aluminum oxide nanoparticles (50 ppm and 100 ppm). The results for 50ppm nanoparticles in the B10 blend proved high energetic efficiency and low exergy destruction.

Another study by the same author (Aditya Kolakoti, 2022) reveals that using an oxygenated additive (Dioxyethylene Ether) with palm kernel biodiesel helps improve engine performance with regulated exhaust emissions. Kolakoti A and Koten H (2022) studied the engine cylinder vibrations in a CI fueled with neat palm biodiesel. The vibration results reveal that smooth combustion with low-frequency vibrations is observed for neat biodiesel operation than diesel fuel at 75% and 100% loads. Siva *et al*. (2019) conducted experiments on research diesel engines with 94% waste orange peel biodiesel +4% H_2O+ 2% surfactant (Span 80). They observed that NOx and exhaust smoke are reduced for the orange peel oil biodiesel emulsion compared to neat orange peel oil biodiesel. Another study by Kumar AM *et al*. (2020) observed that BTE is improved by up to 1.4% to 3% with the implementation of 50 ppm and 100ppm TiO_2 nanoparticles in orange peel oil biodiesel, and the exhaust emissions like NOx, CO, and UHC are also regulated with the 100ppm nanoparticle. Nataraj and Senthilkumar (2020) investigated the performance and emissions from a diesel engine with orange peel oil biodiesel blended with an antioxidant additive (L-ascorbic acid). The results reveal that adding antioxidants in orange peel biodiesel lowers the BTE and increases the UHC, CO, and Smoke emissions.

From the discussion, it is evident that in the applications of biodiesel in neat form or blend with diesel or blend with additives, the combustion and emission results are more promising than diesel fuel operation; hence biodiesel combustion is called clean combustion fuel. Despite the advantages of biodiesel combustion, the production of biodiesel from different feedstocks is challenging due to different reasons, which include the food versus fuel conflict, availability of feedstocks for production, cultivation of oil crops, availability of land and water, the chemical used in biodiesel production, homogeneous and heterogeneous catalyst usage and their effect on biodiesel yield are some of the challenging aspects for biodiesel production.

Compared to edible oils, non-edible oils are highly recommended for biodiesel production, and waste cooking oil is a promising feedstock for biodiesel production. Similarly, algae oil is also gaining wide attention and is considered a 3rd generation fuel. Using the Soxhlet apparatus, Kalyani *et al*. (2022) investigated algae cultivation and oil extraction from raw algae powder. The extracted raw algae oil is converted to algae oil biodiesel and blended with diesel fuel. The results show that B40 blend fuel oil properties closely match diesel fuel, and the solvent oil extraction process is beneficial in algae oil extraction from dry algae powder. Biodiesel yield and chemical usage are major parameters influencing the production process and cost. Different optimization and artificial intelligence (AI) tools are widely used to improve biodiesel yield and help predict biodiesel yield more accurately, and assess significant and influencing parameters like the catalyst, time, temperature and temperature, and molar ratio on biodiesel yield. Nowadays, heterogeneous catalysts are gaining popularity due to their reusability capability, and different studies provide that using waste chicken egg shells (Aditya & Satish, 2023), moringa leaves (Kolakoti *et al.,* 2022), and calcinated banana leaves in biodiesel production can cut down the production cost of expensive catalysts.

This study aimed to create biodiesel using oil extracted from discarded orange peels. For this purpose, an attempt is made to extract the oil from waste orange peels and utilize the oil-extracted peels as a calcinated heterogeneous catalyst for producing orange peel oil biodiesel (OPOBD). Furthermore, the reusability of the heterogeneous catalyst is verified and obtained OPOBD is blended with neat diesel fuel to examine the important fuel oil properties as per the international biodiesel standards ASTM.

MATERIALS AND METHODS

Orange peel oil extraction

The scientific name for orange is Citrus Sinensis, and it is rich in vitamin C and is a widely consumed fruit and by-product used in cosmetic applications (Anna Geraci et al., 2017). The orange fruits are widely available in the country (India) and are available all year. Nagpur district in Maharashtra state is well known for cultivating and exporting oranges globally. Most orange peels are treated as waste after fruit consumption (Negro V *et al*., 2017). Therefore, orange peels are collected from different sources, cleaned and dried under the natural sun for 36 hours, and then crushed in an oil expeller to extract the oils from the waste orange peels. A total of 10 kgs of dried orange peels are used to extract oil, and 30% orange peel oil is achieved.

Catalyst Preparation

Catalysts play a significant role in biodiesel production, and two types of catalysts are widely used in the application. Homogeneous and heterogeneous catalysts like sodium hydroxide (NaOH) and potassium hydroxide (KoH) give a better biodiesel yield during production(Agarwal *et al*., 2012). However, the homogeneous catalyst cannot be reused(Kiss *et al*., 2010). Heterogeneous catalysts emerged to overcome this challenge (Chouhan AS and Sarma A, 2011). Heterogeneous can be obtained from different potassium and calcium-rich materials, such as waste chicken egg shells, banana fruit peel, sea shells, moringa leaves, Etc.

This study uses the waste by-product of orange peel oil extract as a heterogeneous catalyst. The oil extract from discarded orange peels must be dried in a hot air oven for 24 hours to remove any moisture; after that calcination process is initiated to prepare the catalyst. Calcination is a popular technique that helps improve chemical compounds by heating to high temperatures without melting and under a restricted supply of atmospheric oxygen. In this study, orange peel extract is calcinated up to 600^{o}C for 120 minutes, and the different stages of the calcination process are highlighted in Figure 1.

Transesterification Process

The transesterification process is one of the viscosity-reduction techniques popularly used by different research groups due to the high conversion rate of raw oil to biodiesel. Apart from the transesterification process, microemulsion, dilution, catalytic cracking, pyrolysis, and preheating are some of the viscosity reduction techniques used (S Satya *et al.,* 2019). During the transesterification process, triglycerides in the raw oil react with the alcohol (methanol) to form methyl esters and glycerin. The stages of conversion of raw oil to biodiesel are presented in figure2. The biodiesel setup consists of a hot plate magnetic stirrer, a separating funnel, and glass beakers. Initially, raw orange peel oil is filtered and preheated to remove the impurities and moister content. A measured quantity of filtered raw oil is placed in a conical flask on the hotplate magnetic stirrer. A heterogeneous catalyst mixed with alcohol is added to the raw oil. A filter paper is used when the catalyst with alcohol is mixed with raw oil, and the recovered catalyst is dried and reused to recover the catalyst. During the chemical reaction process, the temperature is maintained between 55^{o}C to 57^{o}C and continuous stirring for 180 minutes, and the molar ratio is maintained at 8:1. After the desired reaction period, the oil mixture is transferred into a separating funnel, as shown in fig.2 and allowed it to settle whole night. The same procedure is repeated three times, and the average value is taken as a biodiesel yield.

Due to the shifting of the organic functional R group of an ester with the organic functional R group of alcohol, the conversion of raw oil to methyl esters will occur. The reaction by-product glycerin will settle in the bottom of the separating flash. The heavy mass glycerin is removed, and orange peel methyl ester (OPME)/ orange peel oil biodiesel (OPOBD) is washed with deionized water to bring a neutral pH. Due to the heterogeneous catalyst, more amount of water is used during the washing process, and available literature(Aditya Kolakoti and G Satish, 2023; Kolakoti A *et al*., 2022; Anna *et al*., 2017) [15-17] on heterogeneous catalyst application in biodiesel production reveals the same. After the washing process, the OPME is heated to 110°C to remove the water content, and then the OPME is cooled and stored in air-tight glass beakers for further analysis of fuel oil properties. The same experimental procedure is repeated with the reused catalyst, and observed the biodiesel yield.

Figure 1. Stages of calcination for orange peel extract.

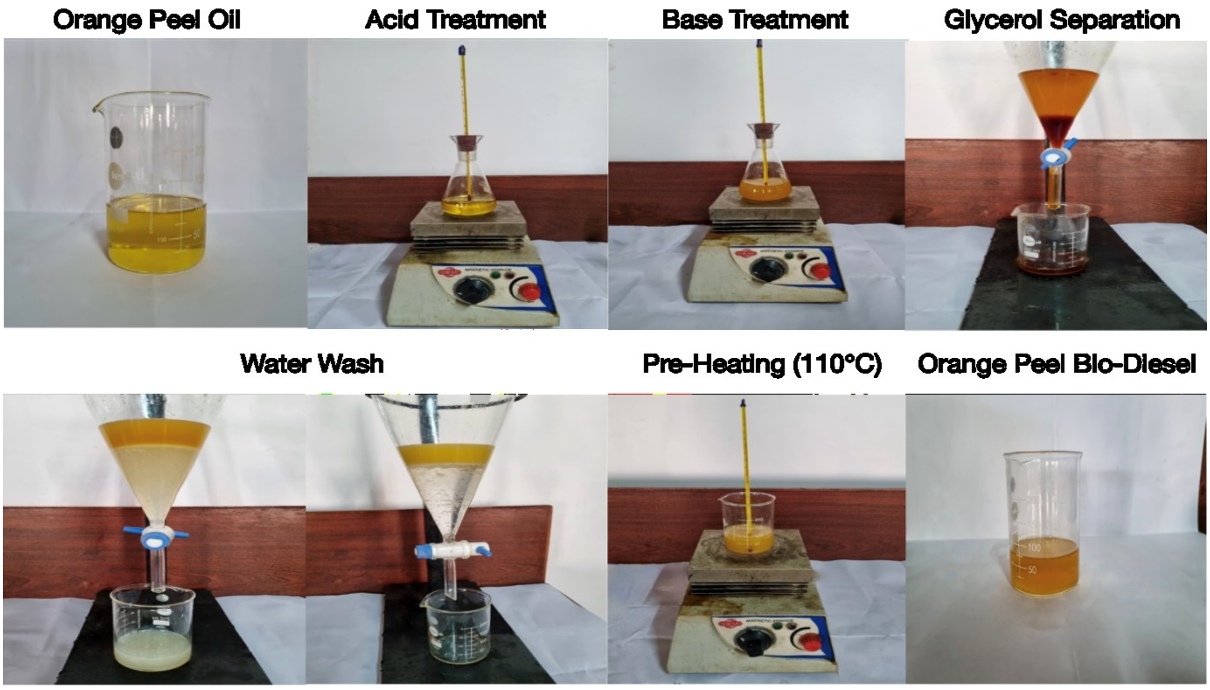

Figure 2. Transesterification process stages.

RESULTS AND DISCUSSION

Fuel Oil Property Characterization

The demand for renewable fuels like biodiesel is gaining significance due to their clean combustion. Fuel combustion inside the combustion chamber mainly depends on the significant fuel oil properties. Therefore, the significant fuel oil properties like viscosity (cSt), density (kg/m^3), heating value (kJ/kg), Cetane number, flash and fire points (°C), and cloud and pour points (°C) are examined experimentally by following the ASTM standards and the obtained results are presented in Table 1.

Table 1. Significant properties of Orange Peel Oil Biodiesel and its blends

Fuel Property	OPOBD100	OPOBD20	Diesel Fuel
Viscosity at 35°C (cSt)	2.7	2.56	2.5
Density at 18°C (kg/m^3)	0.876	0.851	0.846
Heating Value (kJ/kg)	38887	43658	44368
Cloud Point (°C)	-16.39	-13.56	-13
Pour Point (°C)	-9.44	-3.79	-3
Flash Point (°C)	144	71	66
Fire Point (°C)	156	79	68
Cetane number	54	50	49

The kinematic viscosity is measured with a Redwood Viscometer as per the ASTM-D445 specifications, and the test is carried out at 35°C. The result of kinematic viscosity for OPOBD is 2.7 cSt, slightly higher than mineral diesel fuel. The viscosity plays a vital role in fuel combustion; high viscosity will restrict the fuel from atomizing, a crucial parameter for better fuel combustion. Furthermore, high viscosity increases the fuel droplet's size, leading to incomplete combustion of fuel. Therefore, low-viscosity oils are always deserved for combustion, especially for CI engines.

Similarly, the density is measured with a digital density meter by following ASTM-D 4052 specifications, and it is observed that 0.876, which is 3.44% higher than diesel fuel. The high density of biodiesels is due to the presence of FAC. Flash and fire points play a significant role in ease of storing and transportation. In this study, flash and fire points are measured with a Cleveland open cup instrument by following ASTM-D92 specifications, and it is observed that 144°C and 156 C.

The heat of combustion depends on the calorific value, and it is measured with a bomb calorimeter by following ASTM-D242. The results reveal that 38887 kJ/kg is achieved for OPOBD, which is 12.35% lower than diesel fuel. In general, neat biodiesel possess low heating value/calorific value due to the presence of saturated and unsaturated FAC profiles. The cloud and pour points are measured by following the ASTM-D97 specifications, which are observed as -16.39 °C and -9.44 °C. The quality of combustion is measured based on the Cetane index, and in our observation, the Cetane number for OPOBD is 54, which is higher than for diesel fuel. Compared to neat biodiesel applications in CI engines, diesel-biodiesel blends like B20 are popular and are used globally. Therefore, the properties of B20 blends are also examined for significant property analysis and presented in Table 1.

Fatty Acid Composition

The orange peel oil biodiesel is tested for fatty acid composition presences using gas chromatography, and the results are presented in Table 2. These FACs play a significant role in combustion and fuel characterization. The significant fuel oil property like viscosity, density, and heating value depends on the level of saturation and unsaturation FAC. From the table.2, it is evident that high levels of saturated FAC, like palmitic acid (38.56%) and stearic acid (9.82%), are reported. Similarly, unsaturated FACs like oleic and linoleic acid are reported as 32.71% and 12.12%. Due to the bonding difference, oleic acid is mono-unsaturated, and linoleic acid is poly-unsaturated. Figure 3 represents the FAC profile of steric acid.

Table 2. Fatty Acid Compositions of orange peel oil biodiesel

Fatty Acid Composition	Wt.(%)
Palmitic acid	38.56
Stearic acid	9.82
Oleic acid	32.71
Linoleic acid	12.12
Others	6.79

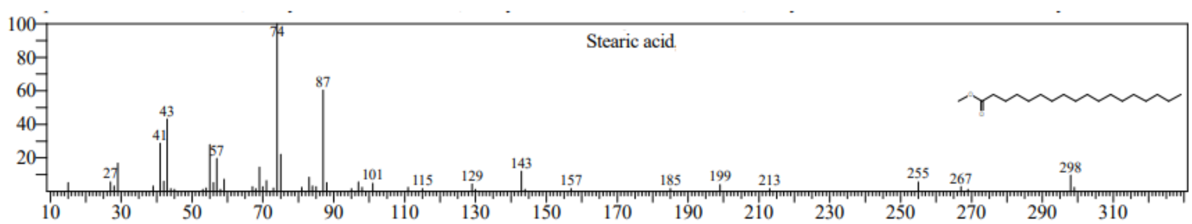

Figure 3. FAC profile of steric acid

Catalyst Characterization

The calcinated catalyst is characterized by the verification of essential elements which play a vital role in the transesterification process. During the calcination up to 600 C, fundamental changes are observed, and different elements, like potassium, sulfur, carbon, calcium, oxygen, etc., are formed. In general, orange peel has a high citric acid and vitamin C level. Figure 4 represents a calcinated orange peel extract. The figure shows that the presence of potassium is visible, which helps convert raw oil to methyl ester during the transesterification process.

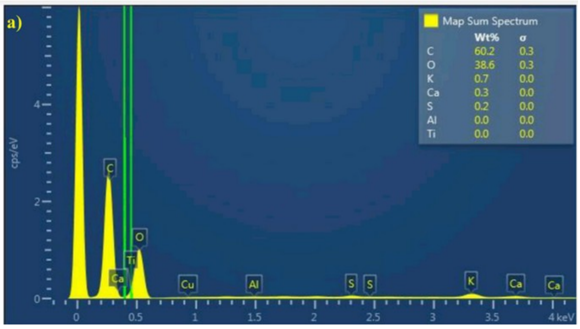

Figure 4. Elemental analysis for calcinated orange peel.[Courtesy(Osman, 2020)]

Reusability of the catalyst

The heterogeneous catalyst is known for its reusability. In this study, the reusability analysis is carried out, and the results are shown in a bar graph in Figure 5. For the first-time application, the biodiesel yield is observed as 89.76%, and the mathematical relation used to calculate biodiesel yield is shown in Equation 1. A gradual reduction of biodiesel yield is witnessed with the repeated usage of the catalyst, which is presented in the bar graph as shown in Figure 5. From the figure, it is evident that up to the fourth time usage of the catalyst, the biodiesel yield is more than 50%, and after the fifth time, the biodiesel yields less than 50%. Furthermore, a comparison (Table 3) was made with the present study's catalyst reusability with the available literature. It is observed that the waste chicken eggshell catalyst and moringa oleifera leaves catalyst dominated the orange peel catalyst.

$$Biodiesel\ Yield\ (\%) = \frac{\text{Amount of biodiesel produced}}{\text{Amount of raw oil used}} * 100 \quad (1)$$

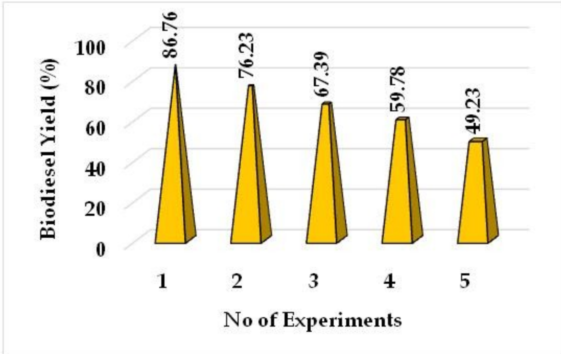

Figure 5. Elemental analysis for calcinated orange peel.

Table.3 Comparison of different heterogeneous catalysts with the present study

No of Experiments	Present study Biodiesel Yield (%)	Waste chicken eggshell catalyst (Aditya Kolakoti and G Satish, 2023)	Moringa oleifera leaves catalyst (Kolakoti A *et al.*, 2022)
1st Experiment	86.76	92	92.82
2nd Experiment	76.23	86	85.62
3rd Experiment	67.39	75	76.38
4th Experiment	59.78	68	63.45
5th Experiment	49.23	48	51.31

CONCLUSIONS

This study shows an effective way to use orange waste peels for biodiesel production and heterogeneous catalyst preparation. The experimental discussions show that the current energy and environmental challenges, especially non-conventional petroleum products, can be mitigated with renewable energy sources. Based on the experimental results, the following conclusions are drawn.

- About 30% of orange peel oil is extracted from dried and wasted orange peels through a mechanical oil expeller.
- The orange peel oil is successfully converted into orange peel oil methyl ester with the transesterification process.
- The heterogeneous catalyst is prepared by calcination technique (heating up to 600°C) and reused multiple times.
- It is observed that more than 50% of orange peel oil biodiesel is achieved for four experimental trials with the heterogeneous catalyst.
- A maximum orange peel oil biodiesel yield of 86.76% is achieved at the optimum molar ratio of 8:1, reaction time of 180 minutes, and reaction temperature of 55°C to 57°C.
- The orange peel oil biodiesel is examined for significant fuel oil properties of viscosity, density, flash point, fire point, heating value, cloud, pour points, and cetane number. The reported results are in line with the international standards of ASTM.
- The FAC by chromatography reveals the presence of high level of saturation than unsaturated compositions.

Therefore, it is concluded that utilizing the waste orange peels will promote sustainable biodiesel production. Furthermore, the economic analysis, engine performance, and exhaust emission testing are suggested as the future scope of the study.

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