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WASTE-TO-ENERGY CONVERSION: INTEGRATED APPROACH FOR BIODIESEL PRODUCTION FROM USED COOKING OIL BY CITRUS WASTE PEEL-BASED BIOCATALYSTS IN SUPPORT OF SDG 3 AND 12.

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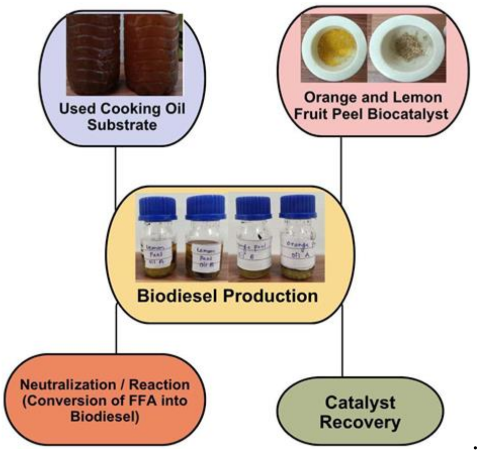
ABSTRACT
The increasing multiple usage of waste cooking oil and citrus waste residues has created significant major health and environmental concerns. This research drives an approach towards sustainable resource recovery over a renewable feedstock. The orange and lemon peel waste performers catalytically were evaluated after the trans-esterification of used cooking vegetable oil (UCO/UVO). This process is optimized by parameters, like 10% catalyst, 12:1 methanol-to-oil ratio, Reaction temperature 80 °C, and reaction time 24 hours, to achieve maximum biodiesel yield up to 14% increment in the taken reactants. The process used two types of oils palm and soyabean, mostly used by hawkers to cook food and synthesized two types with four varied biodiesels. These were characterized using a standard 3/27 physicochemical analyses to determine biodiesels as a fuel quality. The findings indicate to an extent a promising, low-cost, zero-emission and environmentally friendly alternative (citrus peel-derived bio-based) for biodiesel production from UCO/UVO. This integrated multistep green synthesis bioprocess achieves Sustainable Development Goal 3 and 12 of health well-being along with responsible production. This study represents an integrated waste valorization approach towards circular economy, reducing dependence on conventional catalysts and even fossil fuels, and minimizes environmental pollution.

KEYWORDS: Biodiesel production, Citrus waste, Green synthesis, Used cooking oil

Graphical Abstract



INTRODUCTION

The increasing health concerns emerged one of the initiatives towards most common problems of intaking and using used oils in Indian hawkers for cooking purposes. As improper management in food industry and the repeated consumption of used cooking oil (UCO) remain major environmental and public health concerns. Once an edible cooking oil has exceeded its safe frying limit. It started forming unwanted chemical compounds it like trans fatty acids, aldehydes, free radicals, polycyclic aromatic hydrocarbons (PAHs), and acrylamide.[1] The heating leads to deterioration of the cooking oil during repeated heating is characterized by several physicochemical changes, which are visible like darkening of color, viscosity change, continues foaming, rancid odor, and a visible reduced smoke point. Repeated heating shows oxidation, hydrolysis, and polymerization reactions making the edible oil unsuitable for human consumption.[2] Continuous dietary exposure of food from these oils leads to stress, chronic inflammation, cardiovascular diseases, neurodegenerative disorders, and an increased risk of cancer.

UCO should not be discarded into sinks, drains, or open land, as improper disposal contributes to sewer blockages, water and soil contamination. These forms a thin surface film that restricts oxygen transfer, affecting adversely aquatic ecosystems, whilst alters soil properties especially it's fertility inhibiting microbial activity. In addition, the uncontrolled burning of used oil releases particulate matter $PM_{2.5}$ and PM_{10}, volatile organic compounds (VOCs), and other air pollutants that are associated with respiratory illnesses, eye irritation, and long-term health effects.

Despite all environmental and health challenges, UCO represents a valuable and renewable feedstock for biodiesel production rather than a waste requiring disposal.[3] Therefore, they can be converted into promising biofuels with varied usage. scientists extensively work towards green catalyst preparation with the help of biomaterials. At this stage small research based on biodiesels can be a good step at small level.

Biodiesel is produced from edible and even from non-edible vegetable oils,[4] also from animal fats, microalgae, and obviously from UCO. UCO has gained considerable attention as it is inexpensive, readily available, and simultaneously addresses waste management challenges while reducing the overall production cost of biodiesel.[5] As a part of the research, citrus waste is a concern for the agriculture sector. The 2.5 million tonnes of them in India are dumped as waste, resulting in resource waste and environmental degradation.[6] Nearly half of it remains as peel, seeds, and pulp, making the fruit non- edible and 60% of whole fruit discarded as waste. This causes bad odor and pollution to the land, air and water. Citrus processing wastes contain huge amounts of moisture (75–85%) rendering it difficult to dry.[7] Oranges (*Citrus sinensis*) and Lemon (*Citrus limon*) are one of the most widely consumed fruits, globally and high demand created vast quantities of waste. Their peels consist of roughly 70% to 80% water and naturally contain a variety of active enzymes like lipases, pectinases, cellulases, and proteases especially D-limonene (90% of oil). Similarly lemon peels (LP) contain pectin, cellulose, hemicellulose, organic acids, phenolic compounds, and inorganic minerals which allow them to be a stable heterogeneous catalyst. Although they are utilized for many other purposes like LP also gets converted into Solid Biofuel Pellets, still a very lengthy process to upscale and requires lots of energy.[8] Therefore, citrus wastes form a renewable, sustainable and an economic feedstock for obtaining eco-friendly fuel which results in more of OP and LP waste to study.[9]

Biodiesel formation occurs because of its renewable nature, biodegradability, lower sulfur content, and reduced greenhouse gas emissions compared with conventional petroleum diesel. The concept of biodiesel dates back to the late nineteenth century and 1970s global crises renewed its interest now leading to a significant advancement in green transesterification technology and biocatalyst development.

Recent studies demonstrated about OP can be used as an activated carbon, biochar, ash-based, and sulfonated carbon catalysts suitable for biodiesel production. In addition, they even act as catalyst precursors to lower catalyst production cost and minimizes the use of hazardous synthetic chemicals.[10] Therefore, OP serves as an excellent example of converting agricultural waste into functional materials that support sustainable biofuel production. Herein, study reported a “one-pot” green synthesis of solid acid catalyst from OP and LP and used cooking oil (UCO) as a model substrate to produce biodiesel carrying advantages, like easy recovery, reusability, and lower wastewater generation.[11] The integrated utilization of UCO and citrus peel waste provides an environmentally sustainable approach for converting two major waste streams into value-added products. Such a strategy not only minimizes pollution and promotes resource recovery but also supports circular bioeconomy principles and the objectives of Sustainable Development Goal 3 and 12.

2. MATERIALS AND METHODS

2.1. MATERIALS

Orange and lemon citrus peels were collected from juice corner in Malviya Nagar, Jaipur. Used cooking oil (UCO) collected from Local food vendor in Sanganer Industrial Area, Jaipur; Oil A palm oil and Oil B soybean oil used multiple times, Conc. H_2SO_4 (Sigma Aldrich) 20 mL, Ethanol 3.2 mL, Methanol 27 mL, Distilled water (DW). All compounds were utilized as received. They were used without any further purification.

2.2. Preparation of Biocatalyst Orange and Lemon peel waste

Methods used in this research have defined varied temperature range suitable for this work. Following steps are –The steps of this experimental work are as followed (G. Yadav et. al., 2022).[12]

1. Fresh orange and lemon peels was picked and thoroughly washed 3-4 times with DW. Dried in an oven about 80 °C – 100 °C until they became brittle, shown in **Fig. 1**
2. Dried orange peel (OP) and lemon peel (LP) were powdered using a mortar and pestle into a fine dust/ crush, as shown in **Fig. 2**. It was then sieved.
3. OP and LP powders were weighed 1 gm each and were dissolved in concentrated H_2SO_4 in a ratio of 1:20 (20 mL acid per 1 gm powder) as shown in **Table 1**.
4. These mixtures were placed in glass containers (Pyrex) and heated (incubated) in an oven at 100 °C for 24 hours, shown in **Fig. 3**. Ensuring complete carbonization of the fruit waste biomass (peel) into biochar.
5. The OP and LP mixtures were then cooled for 12 hours and then were individually rinsed with DW.
6. Filtered black solid, was again washed repeatedly with hot DW washed and until no residual sulfate particles were left in the filtration. Filtrate was collected to measure its pH. Both processes are as shown in **Fig. 4a and 4b**.
7. Washing was stopped only when the rinsed water attains a pH of 7. This ensures a "solid" acid, and not just leftover liquid acid. Finally, the black biocatalyst powders were incubated in oven at 80 °C for 12 hours, overnight to dry as shown in **Fig. 5a and 5b**.
8. A major precaution to be taken during performing the steps is that Conc. H_2SO_4 should be very slowly added to the powder. Else the acid night releases heat and pungent fumes

Fig. 1: OP and LP dried at 80 °C for 36 hours to get brittle.

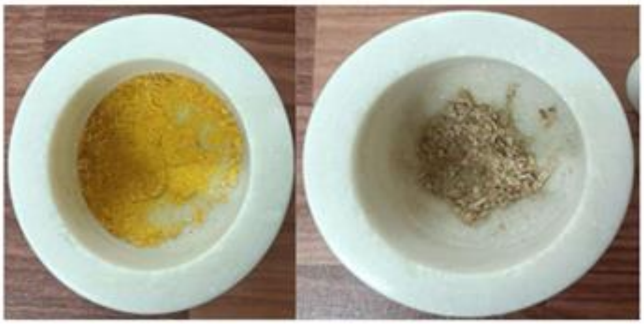


Fig. 2: Crushed and fine powdered of OP and LP.

Fig. 3: Incubated OP and LP at 100 °C for 24 hours.

Fig. 4a: Rinsing OP 1st wash and last 5th wash.

Fig. 4b: Rinsing LP 1st wash and last 6th wash.

Fig. 5a: OP pH at first and last wash.

Fig. 5b: LP pH at first and last wash.

Table 1: Description of the procedure to obtain biodiesel.

Sr. No.		Peel taken	Biocatalyst gm	Oil A and Oil B taken		Oil A (%) obtained	Oil B (%) obtained
1.	Orange Fruit	1 gm	0.9	5 ml	5 ml	4	14
2.	Lemon Fruit	1 gm	0.9	9 ml	9 ml	116.67	91.11

In both cases, we began the experiment with 1 gm of peel powder and approximately 0.9 gm of catalyst was weighed after drying.

2.3 Variation in bio catalyst process

1. Orange peel washed and dried at 100 °C for 15 hours.
2. Added 20 mL sulphuric acid and incubated at 100 °C for 24 hours.
3. Cooling the mixture at room temperature for 24 hours.
4. 7 washes of 30 mL each and filtration done unit the wash pH becomes 7
5. Drying of black solid catalyst for 15 hours at 80 °C.

2.4 Reusability of the biocatalyst in biodiesels

The catalyst reusability indeed plays an important role on a large scale. The catalyst recycling was done by filtration, followed by washing with methanol, and kept in a hot air oven at 80 °C for 4 h. Next cycle needs to be tested.

3. CONVERSION OF UCO TO A BIODIESEL

3.1 Biodiesel formation (Trans-Esterification)

Steps to follow as suggested by MK Kumawat (2022).[13]

1. 10% catalyst by weight relative to the oil (w/v of the UCO). For OP biocatalyst, oil 'A' UCO is 5 mL and oil 'B' UCO 5 mL taken. For LP 9 mL each UCO, oil 'A' and oil 'B' taken. **Fig. 6** shows the samples to be taken for further process.
2. So, 0.5 gms of OP and 0.9 gms of LP used for UCO 'A 'and 'B'.
3. A 12:1 molar ratio of ethanol to all 4 varied UCO kept at 80 °C for 4 hours for further incubation in glass bottles. (It was taken into consideration that this step does not dissolve bio charred catalyst with ethanol).
4. Let it settle down for phase seperation and collect the upper layer, which may contain biodiesel. Both the peels biocatalyst with UCO 'A' and 'B' shown in **Fig. 7 and 8** respectivel.

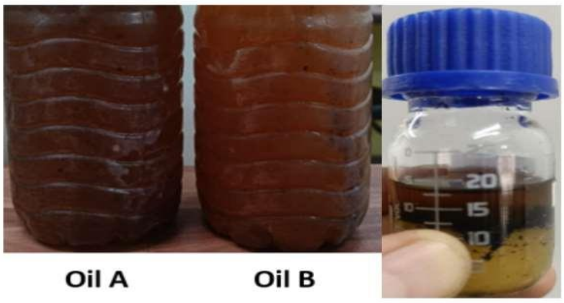


Fig. 6: Samples of oil A and oil B used for two biocatalyst OP and LP. Glass bottle containing final biodiesel product (Clear Visualisation of two layers).

Fig. 7: A very thin upper layer was observed, along with a thick lower layer of petroleum jelly. (OP Front View and Back View).

Fig. 8: A very thick upper layer was observed, along with a very thick lower layer of petroleum jelly (L P Front View and Back View).

3.2 Variation in biodiesel process

In this now, only 5% of both the catalyst were used for 12 mL oil (Oil A and Oil B) in separate. Each tube was added separately with 5.5 mL of methanol for trans-esterification and incubated at 80 °C for 4 hours. Both variation process shown in **Fig. 9 and 10.**

Fig. 9: Varied process for OP for oil A and B.

Fig. 10: Varied process for LP for oil A and B.

3.3 Test for biodiesel conversion (conversion efficiency)

The 3/27 Conversion Test is an indicator of acceptable reaction efficiency. This test recognizes the miscibility of FAME and methanol to provide a qualitative result of the transesterification process. The 3/27 Conversion Test can be used to determine whether are not if a processing variable affected the composition of the reaction product. It's a replacement of alternative to Gas chromatography (ASTMD 6584). The 3/27 Conversion Test is widely recognized by the home biodiesel manufacturing community and endorsed by prominent biodiesel advocate Graydon Blair (2013).[14]

3.3.1 Principle

The 3/27 conversion test is a rapid and qualitative method used to evaluate the completion of the transesterification reaction during biodiesel production. The test is based on the solubility of fatty acid methyl esters (FAME) in methanol. Since fully converted biodiesel is readily soluble in methanol, a clear and homogeneous solution indicates successful conversion. In contrast, the presence of unreacted triglycerides, diglycerides, or monoglycerides results in turbidity or the formation of an oily residue, suggesting incomplete transesterification. Although the 3/27 test provides a quick assessment of biodiesel quality, it is intended only as a preliminary screening method and does not provide quantitative information regarding the exact conversion efficiency.

3.3.2 Procedure

1. Pour exactly 27 mL of pure methanol into the clean, dry graduated cylinder.
2. Add exactly 3 mL of your biodiesel sample to the cylinder, bringing the total volume to 30 mL.
3. Seal the cylinder with a stopper and shake it vigorously for 15 to 30 seconds.
4. Let the cylinder site completely undisturbed on a flat surface for 5 to 10 minutes.
5. Ensure the test is conducted at a room temperature of around 20˚ C to 25˚ C. Cold temperatures can cause false failures.

3.3.3 OBSERVATION

A clear, transparent, single-phase solution without any visible sediment or oily layer was considered a positive (pass) result, indicating that the transesterification reaction was essentially complete and that the biodiesel consisted predominantly of FAME. Conversely, the appearance of cloudiness, precipitate, or an oily layer at the bottom of the container was considered a negative (fail) result, indicating the presence of unreacted glycerides and incomplete biodiesel conversion. It should be noted that the 3/27 conversion test is a qualitative screening technique only and cannot be used to determine the exact percentage of biodiesel conversion or FAME purity. Quantitative confirmation requires analytical techniques such as Gas Chromatography (ASTM D6584 or EN 14105).

Table 2: Criteria to judge 3/27 test.

Sr. No.	Observation	Result	Pass/Fail
1.	Perfectly clear solution	The biodiesel hascompletely dissolved. The conversion rate ishigh (generally above 97.5%).	Pass
2.	Cloudy Hazy solution	Fine droplets of unreacted oil or trace monoglycerides are suspended in the mixture.	Fail (Marginal)
3.	Oil droplets atthe bottom	Distinct, heavy droplets or a clear layer of raw oil has settled out of the methanol.	Fail (Severe)

3.3.4 RESULT

As shown in **Fig. 11a and 11b**, and referring **Table 2**, 100% result obtained. It shows the efficiency of the green process used to produce a biodiesel**.**

3/27 test result: - Biodiesel is pure as there is neither a turbidity nor any bubbles at the bottom of the measuring cylinder. Biodiesel passes the test as the solution is absolutely without any cloudiness and no oil droplets at the bottom.

Fig. 11 a: In oil sample ‘A’ and ‘B’ prepared using orange peel complete transparent clear solution obtained.

Fig. 11 b: In oil sample ‘A’ and ‘B’ prepared using lemon peel with no turbidity at the bottom of measuring cylinder obtained.

4. RESULTS

1. This 5.2 mL of Oil ’A’ and 5.7 mL of Oil’ B’ biodiesel with OP and Oil A 10.5 mL and Oil B 8.2 mL from LP obtained.

Earlier we used Oil “A” 5 mL and Oil ‘B” 5 mL, so the green synthesis converted it into biodiesel increment in volume by 104 % for Oil ‘A’ and 114 % for Oil ‘B’ when OP used. Also, we used Oil “A” 9 mL and Oil ‘B” 9 mL, so the green synthesis converted it into biodiesel increment in volume by 116.67 % for Oil ‘A’ and 91.11 % for Oil ‘B’ when OP used. The Yield is slightly better in Lemon peel extract.

Effective peel for preparing biocatalyst is Lemon fruit.

2. Three times higher yield than the previous experiments.

5. DISCUSSION

This study explored the production of high-quality biodiesel–fuel from UCO using OP and LP waste as the catalyst. By using pre-defined green synthesis demonstrated to be an effective catalyst for generating environmentally friendly biodiesel–fuel. Using a lower catalyst quantity (10 wt.% of OP and LP) and lower methanol to biocatalyst molar ratio (12:1) in a comparatively shorter reaction time (60 min) at 80° C, a conversion yield of 100% catalysts, which are often toxic, unsustainable, costly, and non-biodegradable, the present catalyst has a significant influence on environmental preservation.

The variation in reaction parameters demonstrated that catalyst loading, ethanol availability, reaction temperature, and reaction time significantly influenced the conversion of UCO into fatty acid ethyl esters (FAEE). The optimized conditions provided an effective balance between catalyst concentration, reaction efficiency, and energy consumption. Furthermore, the 3/27 conversion test was employed as a rapid qualitative screening method to assess the completeness of the transesterification reaction. The formation of a clear and homogeneous biodiesel–methanol mixture without visible oily fallout indicated successful conversion and supported the high conversion obtained under the optimized reaction conditions.

Overall, the findings demonstrate that the integrated valorization of UCO and citrus peel waste provides a low-cost, renewable, and environmentally sustainable route for biodiesel production. The proposed approach simultaneously addresses UCO disposal and citrus agro-waste accumulation while reducing dependence on conventional chemical catalysts. Therefore, the use of orange- and lemon-peel-derived bio-based catalysts represents a promising strategy for sustainable biodiesel production and supports the principles of circular economy and Sustainable Development Goal 12 (SDG 12).

6. CONCLUSION

The present study demonstrates a promising potential of citrus waste as a catalyst for certain chemical reactions. The use of this citrus peel is done in a way that neither it lases its originality nor it is converted into a new substance (which usually requires an enormous amount of energy).

Thus, it provides a perfect alterative to the conventional catalysts prevalent in industries which usually show their efficacy only when used in considerable quantities. This study is also associated with the health and well-being of human, animals and other living being as utilizing the used cooking oil is very hazardous and can raise serious concerns regarding the health and physiological state of living beings.[15]

Another notable concern is focused on the environment as the study emphasizes to balance and positively works towards SDG 12 which promotes resource efficiency, waste reduction, public practices involvement and global awareness.

India and other developing and developed countries with their implementation models are largely working on this from the grass root level and thus making our research and study one step closer and therefore contributing towards SDG goals have presenting a bigger and larger picture in the path of achieving a circular economy by 2030.

AUTHORS' CONTRIBUTIONS

The authors confirm their contribution to the paper as follows: SMB prepared and adjusted the Tables and Figures. Dr. JJ conceptualized the study, designed it, and wrote the manuscript. Collected the inter-related data, visualized the results, and validated the manuscript. Conceptualized the study, revised, reviewed, and edited the manuscript for intellectual content. All authors read and approved the final manuscript. All authors reviewed the results and approved the final version of the manuscript.

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CONFLICT OF INTEREST

We declare that we have no conflict of interest.

ABBREVIATIONS

Abbreviation	Full name
UCO	Used Cooking Oils
OP	Orange Peel
LP	Lemon Peel
VOC's	Volatile Organic Compounds
PAH's	Polycyclic Aromatic Hydrocarbons
DW	Distilled Water
FAME	Fatty Acid Methyl Esters

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