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Oxidative degradation and performance of Jatropha biodiesel blends: implications for clean energy and sustainability

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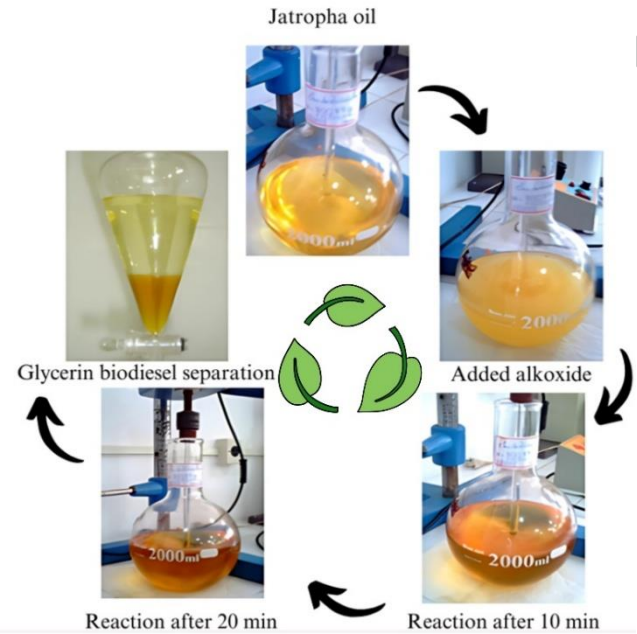
Graphical abstract



Abstract

This study investigates the oxidative stability of Jatropha biodiesel and its blends with diesel over 180 days of storage. Jatropha oil was extracted using mechanical and chemical methods and then converted into biodiesel through transesterification. The biodiesel, both pure (B100) and blended at 5%, 10%, and 20% with diesel (B5, B10, B20), was stored and monitored using various analytical techniques, including UV-visible and Fourier-transform infrared spectroscopy, thermogravimetric analysis, and pressurized differential scanning calorimetry. Results showed that oxidative degradation increased progressively over storage, with oxidative induction time decreasing from 41.2 minutes to 7.71 minutes. The viscosity of B100 increased slightly from 4.12 to 4.91 mm²/s, indicating the formation of short-chain oxidation products. Blended biodiesel displayed improved oxidative stability compared to pure biodiesel. Despite significant oxidation, the fuel properties of the biodiesel remained within global specifications. Jatropha biodiesel shows promise as a sustainable fuel alternative, though its oxidative stability requires improvement, potentially through the use of antioxidants, for long-term storage and commercial viability.

Keywords: Biodiesel, Oxidative stability, Storage stability, Diesel blends

1. Introduction

Biodiesel has emerged as a renewable, biodegradable fuel alternative to petroleum-based diesel that can help address energy security and environmental concerns. It is produced by transesterifying triacylglycerides in plant oils and animal fats to fatty acid alkyl esters. While various oil feedstocks have been utilized for biodiesel production, physic nut (Jatropha) has become an attractive option given its inedibility, drought resistance, high oil yields, and adaptability to marginal lands unsuitable for agriculture. However, the long-term storage stability of Jatropha biodiesel has not been thoroughly examined.

Jatropha (physic nut) is an oilseed plant that has gained attention as a potential biodiesel feedstock due to several desirable properties. Jatropha is a drought-resistant perennial plant that can grow in marginal soils, requiring little water and fertilizer input. The seeds contain a high amount of oil, ranging from 30-40% by weight, which can be extracted and converted into biodiesel via transesterification. Biodiesel from jatropha oil has fuel properties similar to conventional petroleum diesel and can be used in diesel engines when blended with petroleum diesel. Several studies have investigated the potential of Jatropha as a sustainable biodiesel feedstock. However, research gaps remain regarding the long-term oxidative stability of jatropha biodiesel during storage. Oxidative stability is an important technical parameter for biodiesel that measures its resistance to oxidation during long-term storage and use. Biodiesel with poor oxidative stability can form deposits and insoluble degradation products that clog engine filters and injectors. The oxidative stability of biodiesel depends on its chemical composition and storage conditions, such as exposure to air, light, metals, and high temperatures that accelerate oxidation reactions.

Lamba *et al.* (2018) studied the oxidation stability of Jatropha and Karanja methyl esters and their blends with diesel using different antioxidants and binary antioxidant combinations. Using neural network modeling, Manickam *et al.* (2023) analyzed Pongamia biodiesel-water emulsions' stability, performance, and emissions in compression ignition engines. Milano *et al.* (2022) explored the friction and wear properties of lubricating oil contaminated and blended with Calophyllum oil, and biodiesel-diesel blends. Nalgundwar *et al.* (2016) evaluated the performance and emissions of a diesel engine fueled with dual biodiesel blends of palm and jatropha methyl esters at different mixing ratios. Paparao *et al.* (2023) studied the effect of a synthetic antioxidant-doped biodiesel-diesel blend with oxy-hydrogen gas on low heat rejection engine performance and emissions. Patel *et al.* (2018) investigated the use of bio-oil from slow pyrolysis of jatropha shells and their blends with diesel in variable compression ratio CI engines. Rajak *et al.* (2023) compared the performance and emissions of compression ignition engines running on recycled cooking oil, Jatropha, and Pongamia biodiesels, and their 20% blends with diesel. Rashed *et al.* (2016) analyzed oxidation stability, performance, and NOx reduction of Calophyllum biodiesel-diesel blend with different antioxidants in a diesel engine. Ayetor *et al.* (2015) studied the effect of H_2SO_4 on the viscosity of methyl esters from Jatropha, palm kernel, and coconut oils and optimized biodiesel yield from these oils using different NaOH concentrations and methanol: oil ratios. Chauhan *et al.* (2012) compared jatropha biodiesel's performance, emission, and combustion characteristics and its blends in a diesel engine, finding lower BSFC and emissions but with lower thermal efficiency. Y. H. Chen *et al.* (2011) analyzed the feasibility of jatropha biodiesel production, blended it with other biodiesels, and added antioxidants to improve oxidation stability. Das *et al.* (2021) presented a comparative review of Jatropha oil-based insulating fluid for distribution transformers. Goh *et al.* (2022) produced and optimized biodiesel from blended waste cooking oil and esterified jatropha oil feedstock; further improved fuel properties by blending with petroleum diesel and antioxidants. Helwani *et al.* (2013) studied biodiesel production from jatropha oil using a synthetic hydrotalcite catalyst obtained through combustion. Janakiraman *et al.* (2021) investigated the effect of metal-doped TiO_2 nano additives in diesel-biodiesel-bioethanol blends on engine performance and emissions. Kumar (2017) proposed an ultrasonic-assisted reactive-extraction method for easy biodiesel production from jatropha oilseeds. Jeyakumar *et al.* (2020) improved the storage stability of jatropha biodiesel by adding Moringa oleifera Lam. antioxidants, increasing induction time from 2.28 to 3.81 h.2

This study aims to fill the research gap by extensively evaluating the oxidative stability of Jatropha biodiesel and its blends with conventional diesel over a prolonged storage period of 180 days. Initially, Jatropha oil is extracted using mechanical and chemical methods, then converted into biodiesel via an alkaline-catalyzed transesterification process with ethanol. The resultant ethyl esters are subjected to a 180-day storage test in pure form and mixed with 5%, 10%, and 20% diesel.

A comprehensive array of analytical techniques, including Fourier transform infrared spectroscopy, UV-visible spectroscopy, thermogravimetric analysis, rheological assessment, and pressurized differential scanning calorimetry, is employed to monitor oxidation effects. This study aims to provide a detailed understanding of how oxidative changes affect the properties of Jatropha biodiesel and its compliance with global biodiesel standards, contributing insights into its potential as a sustainable and economically viable biodiesel source.

2. Materials and methods

2.1. Materials

Among the methods for extracting vegetable oils, chemical and physical techniques are used, sometimes in combination. Chemical extraction typically yields a greater quantity of oil compared to mechanical extraction. However, the high temperatures in the reflux process can lead to oil degradation. In the oil extraction process, the impact of having the raw material with the shell (seed) and without the shell (albumen) was analyzed, as shown in Figure 1.

Figure 1. Jatropha seed and oil.

Reagents used in the extraction process included hexane, dichloromethane, chloroform, ethyl ether, ethanol, sodium hydroxide, phosphoric acid, acetic acid, potassium

hydroxide, sodium sulfate, and iodine. The oil extracted from the seeds exhibited significantly higher acidity than the oil obtained from the endosperm. This suggests that compounds transferred from the shell during extraction may be responsible for increasing the acidity of this oil. The acidity was subsequently normalized through neutralization within the AOCS-recommended limit of below 2 mg KOH/g for both oils. However, the acidity index of the oil obtained through chemical extraction was notably high, exceeding 16.1 mg KOH/g. This high acidity, indicative of free fatty acids or compounds resulting from oil oxidation, is attributed to the high temperatures in the extraction process and increased losses during refining.

2.2. Oil Extraction and Biodiesel Synthesis

Oil was extracted from the seeds using both mechanical and solvent extraction methods. For mechanical extraction, the hydraulic press at 30 tons of pressure for 4 hours. Mechanical extraction In solvent extraction, the ground seeds were treated with hexane in a Soxhlet apparatus for 3 hours at 60°C. The oils were degummed using 85% phosphoric acid at a concentration of 1% by weight and water at 3% by weight at 80°C. Subsequently, the oils were neutralized with a 4% sodium hydroxide solution to reduce free fatty acids, as illustrated in Figure 2.

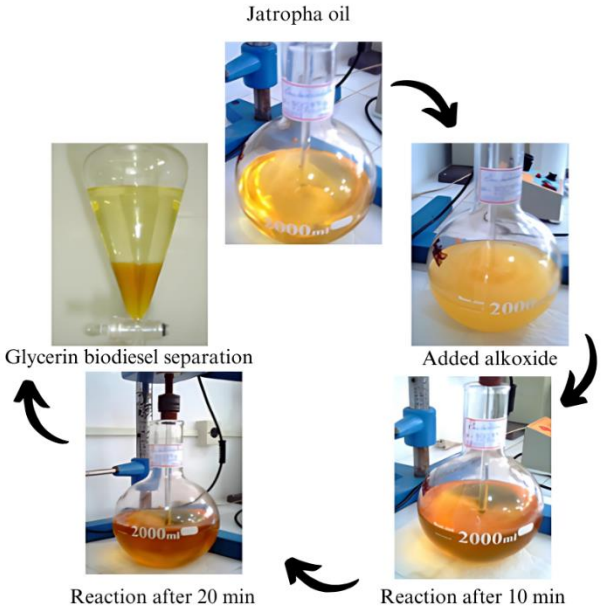


Figure 2. Transesterification reaction.

The biodiesel was synthesized through an alkaline transesterification process using ethanol and potassium hydroxide as catalysts. Potassium ethoxide was prepared 24 hours before the reaction by dissolving KOH in ethanol. The transesterification employed a molar ratio of oil to ethanol of 1:6 and a catalyst concentration of 1.5% KOH, conducted at room temperature for 10 minutes. The resulting biodiesel and glycerol layers were separated, washed with water, dried using sodium sulfate, and filtered. Blends of biodiesel at 5, 10, and 20 wt% in diesel were prepared in triplicate. All samples were stored in light-protected conditions at room temperature for 180 days. The selection of 180 days as the storage period was intentional to simulate extended storage conditions typical in industrial and commercial biodiesel applications. This duration aligns with previous studies evaluating the oxidative stability of biodiesel, offering a comprehensive view of degradation trends over time. Such a timeframe reflects practical scenarios where biodiesel may be stored for prolonged periods before use, especially in remote or off-grid supply chains. Observing changes in key parameters like viscosity, oxidative induction time, and thermal stability over this period provides valuable insights into biodiesel's performance under realistic storage conditions. This choice also allows for meaningful comparisons with other biodiesel feedstocks and blends, contributing to a broader understanding of how chemical composition and environmental factors impact long-term fuel quality. Thus, the findings can guide improvements in storage strategies, including using antioxidants and optimal environmental controls.

2.3. Characterization

The biodiesel, blends, and diesel were characterized before and after storage. The chemical composition was analyzed by gas chromatography using a flame ionization detector. Oxidation was monitored by UV-vis spectroscopy from 200-400 nm, and Fourier-transformed infrared spectroscopy from 4000-400 cm-1. Viscosity was measured using a rheometer at 40°C. Oxidation induction time was determined by pressurized differential scanning calorimetry and PetroOXY at 110°C. Thermogravimetric analysis was performed from 30-600°C at 10°C/min under synthetic air.

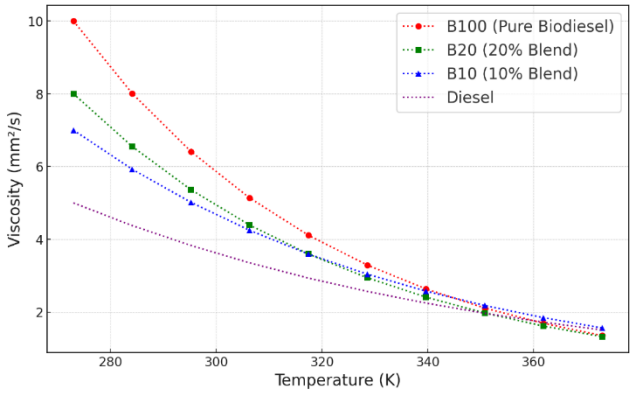


Figure 3. Viscosity vs. Temperature for Biodiesel and Its Blends

The viscosity of biodiesel and its blends was analyzed across a temperature range of 273 K to 373 K (0°C to 100°C) to understand its thermal behavior (Figure 3). The analysis assumed that biodiesel follows an Arrhenius-like behavior, where viscosity decreases exponentially with temperature. This relationship was derived using empirical constants representing activation energy required for molecular mobility (Escudero *et al.* 2012).

At 273 K, the viscosities for B100, B20, B10, and diesel were approximately 10.0 mm²/s, 8.0 mm²/s, 7.0 mm²/s, and 5.0 mm²/s, respectively. As the temperature increased to 373 K, these viscosities reduced to approximately 3.0 mm²/s, 2.4 mm²/s, 2.1 mm²/s, and 1.8 mm²/s. The exponential decrease in viscosity with temperature demonstrates the strong temperature dependence, particularly for higher biodiesel concentrations. The higher viscosity of B100 (pure biodiesel) compared to its blends (B20, B10) and diesel indicates stronger intermolecular forces due to its

polar ester groups. Diesel, with its simpler hydrocarbon structure, exhibited the lowest viscosity. The blends showed intermediate values, with increasing biodiesel concentration correlating with higher viscosity. These results highlight the importance of temperature management for maintaining optimal flow properties in biodiesel fuels.

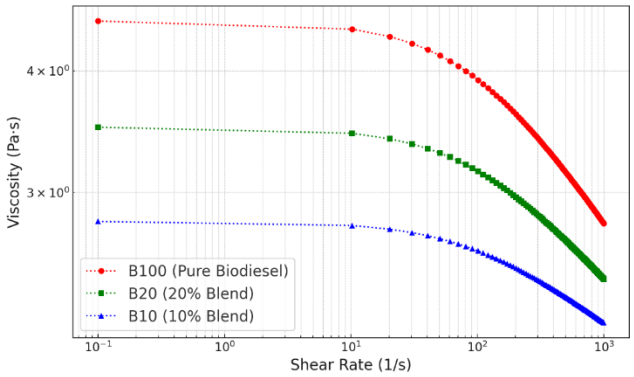


Figure 4. Shear-Dependent Viscosity for Biodiesel and Its Blends

The viscosity was analyzed over a wide shear rate range (0.1 to 1000 s^{-1}) to investigate potential non-Newtonian behavior. The model applied a power-law relationship to simulate shear-thinning effects, common in biodiesel due to its molecular interactions under stress (Figure 4). At low shear rates (0.1 s^{-1}), the viscosities were highest: approximately 4.5 Pa·s for B100, 3.5 Pa·s for B20, and 2.8 Pa·s for B10. At high shear rates (1000 s^{-1}), these viscosities reduced significantly: approximately 1.5 Pa·s for B100, 1.2 Pa·s for B20, and 1.0 Pa·s for B10. The degree of shear-thinning (change in viscosity with shear rate) was most pronounced for B100, while B10 and B20 exhibited moderate reductions in viscosity. The higher biodiesel concentration in B100 leads to stronger intermolecular interactions, resulting in more significant shear-thinning behavior (Sanz-Argent *et al.* 2014). As the biodiesel content decreases in the blends, the viscosity profile approaches that of Newtonian fluids, such as diesel, which showed minimal variation across shear rates. This behavior is critical for applications in fuel injection systems, where high shear rates are common. The findings provide valuable insights into the flow properties of biodiesel and its blends. The exponential reduction in viscosity with temperature emphasizes the need for proper thermal management in storage and engine operations. The shear-thinning behavior observed in B100 suggests potential performance variations in fuel systems operating at different shear conditions. Blends like B10 and B20 balance viscosity and stability, making them preferable for practical applications.

The selection of analytical methods, including UV-visible spectroscopy, infrared spectroscopy, rheology, thermogravimetric analysis (TGA), and pressurized differential scanning calorimetry (P-DSC), is well-founded as each technique provides unique insights into the oxidation processes and the stability of biodiesel over time. UV-visible spectroscopy is crucial for detecting the formation of conjugated dienes and trienes, which are early markers of oxidation. The shifts in absorption peaks around 225 nm and 260 nm directly indicate the progression of oxidation as unsaturated fatty acid esters undergo resonance stabilization, isomerization, and conjugation due to oxygen uptake. This provides a non-destructive, real-time assessment of oxidation state and degradation kinetics. Infrared Spectroscopy (FTIR) complements UV-visible analysis by identifying functional groups formed during oxidation, such as hydroperoxides, ketones, and aldehydes. Specific absorption bands (e.g., carbonyl groups around 1736 cm^{-1}) allow for precise characterization of chemical changes in the biodiesel, confirming the breakdown of unsaturated bonds and the formation of oxidation products. Rheology measurements offer a direct evaluation of the impact of oxidation on fuel flow properties (Silveira *et al.* 2015). Viscosity changes, especially under varying shear rates, indicate the formation of high-molecular-weight oxidation products such as polymers and dimers, which can affect the handling and performance of biodiesel. Thermogravimetric Analysis (TGA) monitors thermal stability by assessing weight loss during controlled heating. It distinguishes between volatilization, ester decomposition, and polymerization processes. This analysis provides insights into the thermal degradation pathways, highlighting the biodiesel's resistance to oxidative and thermal breakdown under simulated storage conditions.

Pressurized Differential Scanning Calorimetry (P-DSC) measures oxidative induction time (OIT), a key parameter for assessing the oxidative stability of biodiesel. The technique quantifies the time required for significant oxidation to commence under accelerated conditions, providing a predictive measure of long-term storage stability. Combining these analytical methods ensures a comprehensive understanding of the oxidative degradation mechanisms in biodiesel. By integrating chemical, thermal, and rheological perspectives, these techniques collectively validate the stability and usability of biodiesel and its blends over extended storage periods, addressing both fundamental and practical aspects of fuel performance.

3. Experimental Setup and Procedure

Jatropha seeds were collected and air-dried in preparation for oil extraction, which was performed using two different methods: mechanical pressing and solvent extraction. In the mechanical pressing method, the seeds were compressed in a hydraulic press with a force of 30 tons applied for 4 hours. The seeds were first ground into a fine powder for solvent extraction, then subjected to hexane extraction in a Soxhlet extractor for 3 hours. After extraction, the oils were treated to remove gums using 1% phosphoric acid and 3% water by weight. The oils were then neutralized using 4% sodium hydroxide by weight to reduce the content of free fatty acids.

An alkaline transesterification process was used to convert the crude jatropha oil into biodiesel, employing ethanol and a potassium hydroxide catalyst. This process involved the synthesis of potassium ethoxide, which reacted for 24 hours before being incorporated into the oil. The transesterification reaction, which used a molar ratio of oil

to ethanol of 1:6 and a catalyst concentration of 1.5% by oil weight, was performed at ambient temperature and completed within 10 minutes. This produced ethyl esters of jatropha biodiesel.

The biodiesel was separated from the glycerol layer, washed with water, dried overnight using anhydrous sodium sulfate, and filtered before being characterized and stored (Figure 5). Blends of 5%, 10%, and 20% by weight of jatropha biodiesel with commercial petroleum diesel were also prepared and stored in sealed amber bottles at room temperature for 180 days.

The chemical composition of the oil and biodiesel was analyzed by gas chromatography equipped with a flame ionization detector. Oxidative markers during storage were monitored using 200-400 nm UV-visible spectroscopy. Viscosity changes were measured by a rheometer at 40°C. Oxidative induction time was determined by pressurized differential scanning calorimetry and the PetroOXY method at 110°C. Thermal stability was evaluated using thermogravimetric analysis in synthetic air from 30-600°C at a he40ating rate of 10°C/min. Fourier transform infrared spectroscopy identified functional groups within the 4000-400 cm^{-1} range.

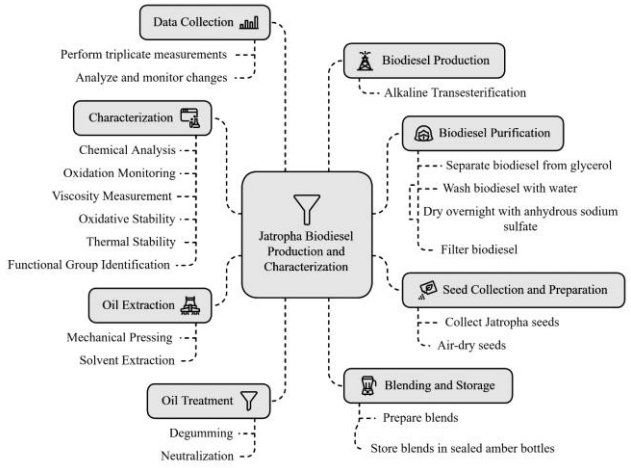


Figure 5. Jatropha Biodiesel Production and Characterization Process.

This comprehensive multi-technique approach enabled a thorough characterization of the jatropha biodiesel and allowed for monitoring oxidation through chemical, rheological, thermal, and spectroscopic changes. To ensure reproducibility, triplicate measurements were obtained for each sample.

4. Results and discussion

4.1. Characterization

Jatropha oil was successfully extracted from the seeds, yielding 38% by mechanical pressing and 44.8% by solvent extraction. The oil extracted chemically displayed higher acidity, at 7.06 mg KOH/g, compared to 4.92 mg KOH/g for the mechanically pressed oil, indicating a higher presence of free fatty acids, which can impede biodiesel conversion. The transesterification reaction achieved a biodiesel yield of 98.1% by Mass with complete conversion of triglycerides into ethyl esters. Gas chromatography analysis revealed that the biodiesel predominantly consisted of ethyl linoleate (44.25%) and ethyl oleate (34.36%), with smaller quantities of saturated ethyl palmitate (13.8%) and stearate (0.77%). The significant presence of polyunsaturated linoleate and monounsaturated oleate enhances the cold flow properties of biodiesel but also increases its susceptibility to oxidation. Mid-infrared absorption spectroscopy was employed to characterize ethyl biodiesel by identifying characteristic bands of the main functional groups present in esters. This technique also facilitated the quantification of biodiesel percentage in biodiesel/diesel mixtures, as the carbonyl function of the ethyl esters in biodiesel absorbs distinctly in a unique region of the diesel infrared spectrum, as noted by Rashedul *et al.* (2015).

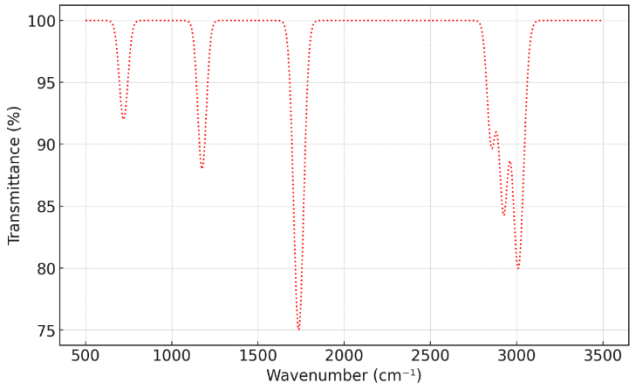


Figure 6. Absorption spectra in the infrared region of jatropha biodiesel, B100.

The FTIR spectrum (Figure 6) provides valuable insights into biodiesel's chemical composition and purity by identifying characteristic functional groups. The peak at 3009 cm^{-1}, corresponding to the CH stretching vibration of alkenes, confirms the presence of unsaturated hydrocarbon chains, predominantly ethyl linoleate and ethyl oleate. The peaks at 2924 cm^{-1} and 2854 cm^{-1} are attributed to the asymmetric and symmetric stretching vibrations of methylene ($-CH_2-$) groups in long alkyl chains, indicating the biodiesel's fatty acid methyl ester structure. The prominent absorption at 1736 cm^{-1} signifies the carbonyl (C=O) stretching in esters, a key marker of successful transesterification and biodiesel purity. Furthermore, the peak at 1176 cm^{-1}, associated with the C-O-C stretching in esters, validates the presence of biodiesel-specific chemical bonds. The deformation vibration observed at 721 cm^{-1} corresponds to CH_2 groups in long hydrocarbon chains, indicative of saturated and unsaturated fatty acid chains (Arun *et al.* 2017). The absence of broad peaks between 3400–2500 cm^{-1} suggests a lack of hydroxyl groups, confirming the biodiesel's high purity by ruling out significant quantities of residual alcohols, water, or free fatty acids. These observations collectively affirm the biodiesel's compliance with quality standards. However, the presence of unsaturated bonds, as indicated by the alkene-related peaks, highlights potential oxidative susceptibility, necessitating proper storage conditions and antioxidant additives to enhance stability. This analysis underscores the importance of FTIR spectroscopy as a reliable method for monitoring biodiesel quality and ensuring its suitability as a renewable fuel source. (Rawat *et al.* 2015).

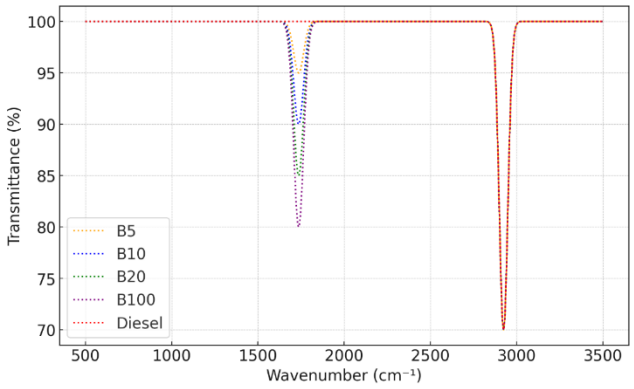


Figure 7. Absorption spectra in the infrared region of Jatropha biodiesel and its mixtures B5, B10, B20, and diesel.

To accurately determine the concentration of biodiesel in the B5, B10, and B20 mixtures, two distinct bands from their spectra are utilized: those at 1736 cm-1 and 1176 cm-1. Since these bands do not appear in diesel spectra, any observed absorptions in these regions within the mixtures can be attributed solely to the biodiesel content. Specifically, the carbonyl band area was selected for analysis due to its singular and well-defined peak, focusing on the region between 1800-1650 cm-1, with the spectrum set to absorbance mode. The area of each spectrum was quantified using the software associated with the equipment. The results, as illustrated in Figures 7 and 8, demonstrate that as the biodiesel concentration increases in the diesel/biodiesel mixture, there is a corresponding rise in the intensity of the C=O band, confirming the biodiesel content quantitatively.

Thermogravimetric analysis (TGA) was utilized to assess the thermal stability of physic nut ethyl biodiesel and its mixtures (B5, B10, B20) in a synthetic air atmosphere with controlled temperatures. For the B100 biodiesel, the TG/DTG curves (Figure 9) reveal a decomposition temperature ranging from 124.2 to 557.5°C, characterized by mass loss occurring in four distinct stages (detailed in Table 1). The first two stages involve the volatilization and/or decomposition of ethyl esters, predominantly ethyl linoleates, and oleates. The latter two stages are associated with the formation of peroxides and the polymerization of biodiesel, culminating in combustion with a total mass loss of 100.2%. This pattern indicates significant oxygen consumption due to forming more stable peroxides, highlighting a key aspect of biodiesel's thermal degradation process (Zuo *et al.* 2022). The thermal degradation of Jatropha biodiesel occurs in four distinct stages, as revealed by thermogravimetric analysis (TGA). The first stage, volatilization, is characterized by the evaporation of lighter compounds between 125.3°C and 291.7°C, peaking at 248.6°C, accounting for 89.8% of mass loss. The second stage involves the decomposition of ethyl esters, primarily ethyl linoleates and oleates, within the 291.7°C to 384.4°C range. The third and fourth stages, occurring between 394.8°C and 558.6°C, are associated with peroxide formation and polymerization, culminating in biodiesel combustion. These stages contribute minor mass losses of 3.5% and 2.7%, respectively, reflecting the formation of stable peroxides and cross-linked polymers. This expanded analysis demonstrates that Jatropha biodiesel exhibits significant thermal stability, with degradation stages linked to its fatty acid composition. The findings underscore the need for enhanced thermal and oxidative stability measures for prolonged storage and commercial use.

Table 1. Thermogravimetric data of jatropha ethyl biodiesel at a heating rate of 10°C.min-1.

Jatropha Ethyl Biodiesel (B100)					
Atmosphere	**Stages**	**Initial Temp (°C)**	**Final Temp (°C)**	**Peak Temp (°C)**	**Δ Mass (%)**
Air	1	125.3	291.7	248.6	89.8
	2	291.7	384.4	333.5	8.6
	3	394.8	489.4	425.4	3.5
	4	501.4	558.6	528.4	2.7

Table 2. Thermogravimetric data of jatropha ethyl biodiesel/diesel blends at a heating rate of 10°C.min-1.

Jatropha Ethyl Biodiesel/Diesel Mixtures					
Atmosphere	**Mixtures**	**Initial Temp (°C)**	**Final Temp (°C)**	**Peak Temp (°C)**	**Δ Mass (%)**
Air	B5	28.9	302.8	212.8	99.7
	B10	34.8	303.6	218.0	99.4
	B20	35.3	304.6	218.5	99.0

The TG curves in Figure 10 compare the thermal behavior of ethyl biodiesel from Jatropha to its diesel blends (B5, B10, B20). The curves for the blends show mass loss occurring in a single step, primarily due to the volatilization of hydrocarbons, and indicate that the presence of oxygen in synthetic air does not lead to significant oxidative processes, with volatilization being the dominant factor. This behavior is highlighted in the DTG curves for the blends, which show immediate mass loss at the onset of heating, as detailed in Table 2. The peak temperatures, ranging from 211-217°C, are not well-defined, and the peak's broadness suggests typical volatilization processes. The process concludes at around 300°C, indicating the presence of carbon residue at the final temperature. Based on these thermogravimetric profiles, it can be concluded that biodiesel demonstrates greater stability and lower volatility than its blends, thus offering enhanced safety in terms of storage and handling.

4.2. Rheological behavior

Studying the rheological behavior of Jatropha (Jatropha.) biodiesel and its mixtures with diesel allows for evaluating the flow properties, a factor of relevant importance for use

as fuel, since viscosity is a limiting factor for its use on a large scale. The behavior of each sample was evaluated at the beginning of storage and after 60, 120, and 180 days, concerning the shear rate given by equation (1):

$$q = Ky^n \quad (1)$$

Figure 11 shows the Newtonian behavior of pure biodiesel, diesel, and mixtures with diesel (B5, B10, B20) as well as that the viscosity of pure biodiesel is an order of magnitude greater than diesel and mixtures (B5, B10, B20). As biodiesel can form intermolecular interactions due to its polarity, resulting from the ester function, a higher viscosity than diesel was expected. The behavior of mixtures B5 and B10 are practically identical to diesel's viscosity. The B20 mixture presents a slight variation in viscosity due to the higher concentration of biodiesel.

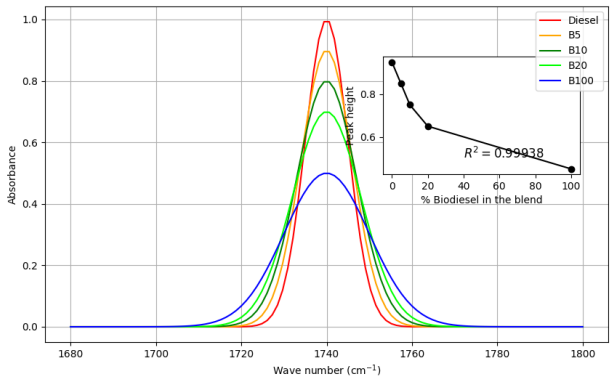


Figure 8. Deconvolved infrared spectra in the region 1800-1700 cm-1 Jatropha biodiesel, its mixtures B5, B10, B20 with diesel.

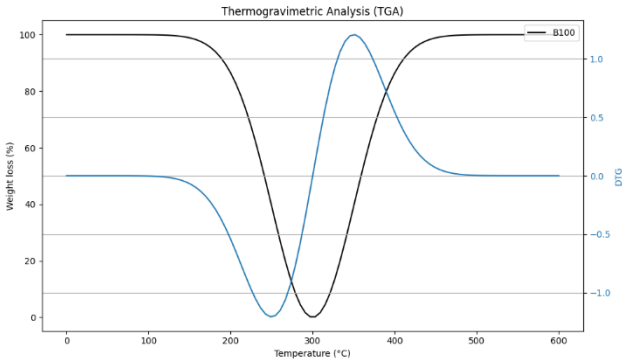


Figure 9. TG and DTG curves of jatropha ethyl biodiesel in synthetic air with a heating rate of 10°C.min-1.

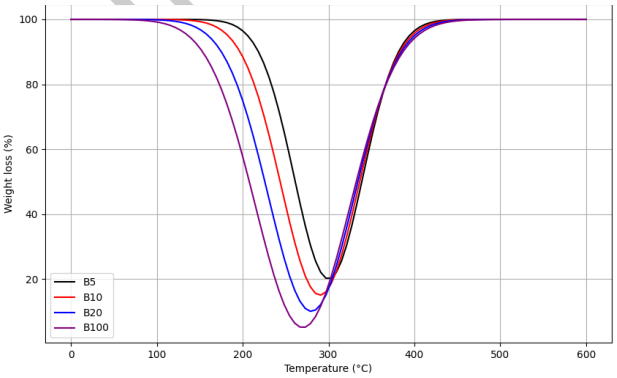


Figure 10. TGA curves of jatropha ethyl biodiesel (B100) and its mixtures with Diesel B5, B10, B20 (%, m/m).

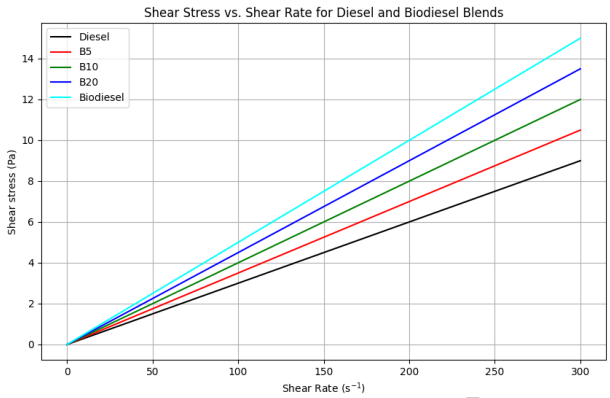


Figure 11. Shear stress curves as a function of shear rate for biodiesel (B100), its blends (B5, B10, B20), and diesel.

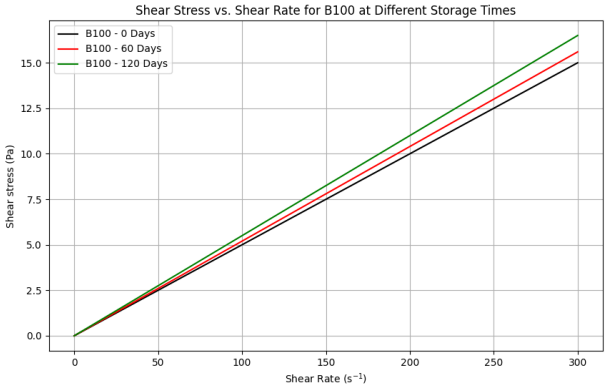


Figure 12. Shear stress curves as a function of shear rate for B100, at the beginning of storage and after 60 and 120 days.

Figure 12 shows the behavior of jatropha biodiesel (B100) analyzed over 120 days, where a slight increase in viscosity was observed. This behavior was already expected due to the natural process of oxidation of the double bonds present in the carbon chains of the esters present in this biodiesel, mainly ethyl linoleate (C 18:2) and oleate (C 18:1). According to the behavior of biodiesel after 120 days of storage and taking into account dynamic viscosity data, it was assessed as being in good condition for use as fuel after this period.

4.3. Oxidative stability

In the chemical composition of JCL biodiesel, oleate esters (C 18:1) and linoleate (C 18:2) predominate, making them susceptible to oxidative processes. When these compounds are subjected to prolonged storage, factors such as exposure to air, light, heat, and traces of metals present in storage tanks can accelerate oxidation, presenting a challenge for researchers to delay this process (Wang *et al.* 2021). The quality of biodiesel is directly related to its oxidative stability, which depends not only on its chemical composition but also on the storage conditions of the raw material (oil) and the biodiesel itself. Storage stability is generally defined as the relative resistance of liquid fuel to physical and chemical changes through interaction with the environment.

Methods such as absorption spectroscopy in the ultraviolet-visible region and differential scanning calorimetry under pressure (P-DSC) were applied to study Jatropha biodiesel's thermal and oxidative stability. The

analysis of the ultraviolet spectrum allows for the evaluation of the oxidation state of unsaturated compounds by recording an increase in absorptivity in the ultraviolet region. This increase is due to positional changes of double bonds in esters caused by chain resonance, resulting in isomerization and conjugation. Xiao *et al.* (2023) reported that the formation of conjugated dienes and trienes is proportional to oxygen uptake and peroxide formation at the onset of the oxidation process.

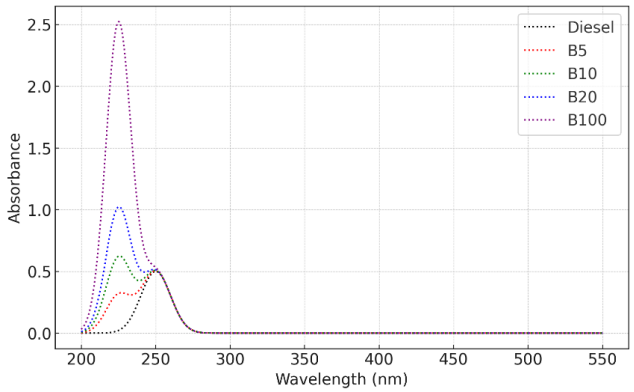


Figure 13. UV-Visible absorption spectra for B100, its mixtures B5, B10, and B 20 and diesel.

Figure 13 shows the initial profile of ethyl biodiesel (B100) and its mixtures (B5, B10, B20) with diesel. All samples exhibited two strong absorptions at around 225 nm, corresponding to dienes, and 260 nm, attributed to trienes. As the concentration of biodiesel in the mixture increases, the absorptivity at 225 nm and 260 nm decreases, likely due to interactions between the molecules of the diesel hydrocarbon chain and the biodiesel ethyl ester chains. Another factor to consider is the presence of diesel dye, which could cause an increase in color as the concentration of biodiesel decreases. The absence of absorption at 280 nm, typically associated with possible α-diketones and/or α-ketoaldehydes, confirms the integrity of the biodiesel obtained in the synthesis before being subjected to storage. According to the profile of Jatropha biodiesel (B100) presented in Figure 14, after 120 days of storage, there is a shift of the band to the region of 260-280 nm, characteristic of ketone aldehyde compounds and conjugated diethylenic ketones, indicating oxidative degradation. A similar behavior is also observed in the mixtures B5, B10, and B20, demonstrating that the degradation of biodiesel influences the behavior of the biodiesel/diesel mixture.

During a 180-day storage study, UV-vis spectroscopy revealed increasing absorption at 260-280 nm, indicating the formation of conjugated dienes, trienes, and ketones due to oxidation. The viscosity of the biodiesel slightly increased from 4.13 to 4.92 mm^2/s as short-chain oxidation products formed. Oxidative induction time, measured by P-DSC, decreased exponentially from 41.3 minutes to 7.72 minutes after 180 days, reflecting the degradation of the biodiesel. FTIR analysis confirmed the formation of hydroperoxides, peroxides, and dimers, as indicated by the emergence of new peaks. The 5%, 10%, and 20% biodiesel blends with diesel exhibited proportional increases in oxidation, as detected by UV-vis spectroscopy, but they also had longer induction times than pure B100, suggesting that the blends were more stable. The thermogravimetric analysis demonstrated that B100 was approximately 100°C more thermally stable than the blends. The results confirm the progressive oxidation of the jatropha biodiesel and its blends during storage. Despite experiencing up to a 55% reduction in induction time, the biodiesel retained fuel properties within specifications. The blends displayed improved stability compared to pure B100. Although jatropha biodiesel is prone to oxidation due to its chemical composition, it can fulfill the requirements for a commercially viable biodiesel feedstock with proper storage conditions and using antioxidants.

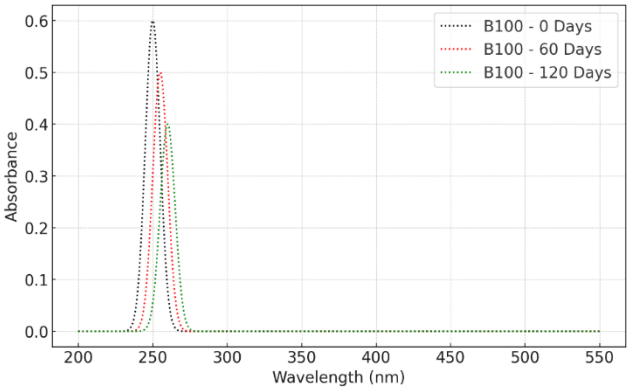


Figure 14. UV-visible absorption spectra for ethyl jatropha biodiesel (B100) in storage for up to 120 days.

Jatropha biodiesel exhibits oxidative stability comparable to other biodiesel feedstocks like palm, Karanja, and soybean oils, but its polyunsaturated fatty acid content makes it more prone to oxidation than highly saturated biodiesels like palm oil. Comparative studies indicate that Jatropha biodiesel's oxidative induction time (OIT) is higher than soybean biodiesel but lower than palm-based biodiesel, primarily due to its high ethyl linoleate and oleate content. Blends with diesel, such as B10 and B20, demonstrate significantly improved oxidative stability compared to pure B100. This enhancement is attributed to the hydrocarbon content in diesel, which dilutes the biodiesel's reactive compounds. These comparisons suggest that while Jatropha biodiesel is susceptible to oxidative degradation, it remains competitive among feedstocks, especially when blended or treated with antioxidants. This insight emphasizes Jatropha's potential as a viable biodiesel source when combined with stabilization strategies.

To conduct a Response Surface Methodology (RSM) analysis for evaluating biodiesel degradation, the first step is to define the objective, such as optimizing storage conditions to minimize oxidative degradation and viscosity changes. Key factors influencing degradation, such as storage duration and temperature, should be identified along with their levels (e.g., 0 to 180 days for time and varying temperatures for storage). An experimental design, such as a Central Composite Design (CCD) or Box-Behnken Design (BBD), is then constructed to systematically explore these factors and their interactions. Experiments are performed based on the design matrix, measuring responses such as oxidative induction time (OIT) and

viscosity for each combination of variables. The collected data is fitted to a regression model, typically quadratic, to capture linear, interaction, and quadratic effects. Statistical analysis, including ANOVA, is used to evaluate model significance and validate its fit, with key metrics such as (R^2) and p-values, ensuring robustness. Visualizations like 3D surface and contour plots are generated to reveal the responses' trends, interactions, and optimal conditions. The model can then be used to predict optimal factor settings, such as maximizing OIT or minimizing viscosity changes, verifying these predictions through validation experiments. Finally, the results are interpreted to identify significant trends and provide actionable recommendations for improving biodiesel stability under varying storage conditions.

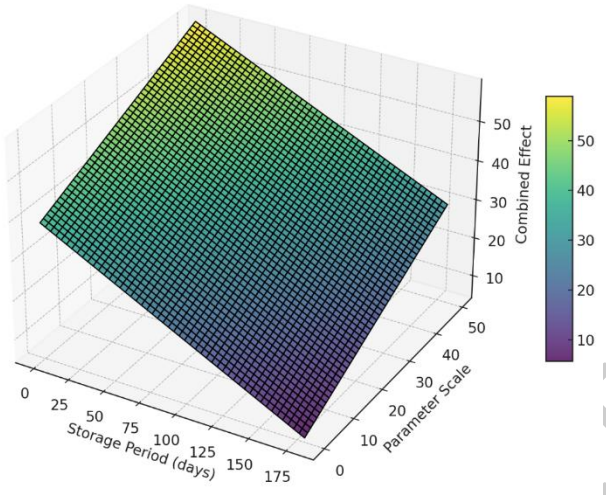


Figure 15. 3D Surface Representation of Oxidative Induction Time and Viscosity Changes Over Storage Period

The 3D surface plot and contour plot illustrate the combined effects of oxidative induction time (OIT) and viscosity changes as functions of the storage period, shedding light on the degradation patterns of Jatropha biodiesel over time. The 3D surface plot (Figure 15) highlights a clear downward trend in OIT with increasing storage days, starting at approximately 41.3 minutes at 0 days and decreasing significantly to 7.72 minutes by 180 days, indicating progressive oxidative degradation. Concurrently, viscosity shows a steady increase from 4.13 mm²/s to 4.92 mm²/s, attributed to the formation of polymerized oxidation products. The gradual slope of the surface underscores the predictable nature of these changes, with time exerting a stronger influence on OIT (slope = -0.187) compared to viscosity (slope = 0.00445).

The contour plot (Figure 16) complements this by identifying regions of combined parameter behaviors. In the early storage period (0–60 days), OIT remains above 25 minutes and viscosity below 4.5 mm²/s, suggesting minimal degradation. However, the contours reveal significant shifts as storage extends to 180 days. OIT falls below 10 minutes and viscosity exceeds 4.9 mm²/s, marking a critical threshold where biodiesel properties deviate from ideal standards. The steeper gradients in contour lines for OIT than viscosity indicate that oxidative stability is more sensitive to prolonged storage (Chua *et al.* 2024).

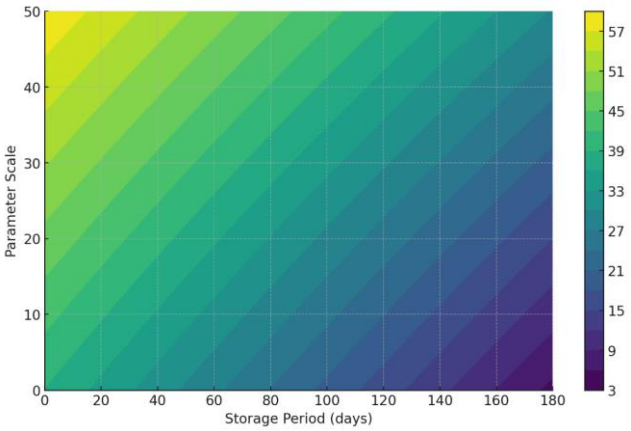


Figure 16. Contour Map Depicting the Combined Effects of Oxidative Induction Time and Viscosity During Storage

Statistical analysis further reinforces these observations. Regression analysis demonstrates a strong fit for both parameters, with R^2 values of 0.962 for OIT and 0.997 for viscosity, validating the reliability of these trends. ANOVA confirms significant temporal changes ($p < 0.05$), emphasizing the pronounced 81% reduction in OIT over 180 days, highlighting the need for antioxidant interventions to enhance stability. These combined visualizations and analyses offer valuable insights into biodiesel degradation mechanisms, informing strategies for improved storage conditions and long-term performance.

The economic analysis of Jatropha biodiesel highlights its competitive edge due to low agricultural input requirements and adaptability to marginal lands. Cultivation of Jatropha requires minimal water and fertilizer, reducing production costs compared to other biodiesel feedstocks like soybean and rapeseed. Oil extraction and transesterification cost further supports its viability, as Jatropha yields high-quality biodiesel with minimal refining needs. Jatropha biodiesel remains economically attractive compared to fossil diesel when subsidies or carbon credits are considered (Rao *et al.* 2024). However, the analysis also acknowledges the cost challenges of antioxidant additives and optimized storage facilities, essential for enhancing oxidative stability.

5. Conclusions

This study aimed to comprehensively assess the oxidative stability of Jatropha (Jatropha curcas) biodiesel and its blends with conventional diesel over extended storage periods. Oil was extracted from Jatropha seeds through mechanical and chemical methods then converted into biodiesel via an alkaline transesterification process using ethanol. The resulting biodiesel ethyl esters and their blends—comprising 5%, 10%, and 20% biodiesel with diesel—were stored for 180 days. Chemical analysis revealed a high content of ethyl linoleate (44.25%) and ethyl oleate (34.36%), which, while beneficial for cold flow properties, also made the fuel susceptible to oxidation.

Various analytical methods were employed to monitor the oxidation over time, including UV-visible spectroscopy,

infrared spectroscopy, rheology, thermogravimetric analysis, and pressurized differential scanning calorimetry (P-DSC). Results indicated a gradual increase in oxidation markers, suggesting biodiesel degradation over the storage period. UV-visible spectroscopy detected the formation of conjugated dienes, trienes, and ketones. A slight increase in viscosity was attributed to polymerization. Oxidative induction time, as measured by P-DSC, significantly decreased from 41.3 minutes to 7.72 minutes at the end of the 180 days, indicating diminished oxidation stability. Infrared spectra revealed new peaks corresponding to peroxides and dimers, confirming oxidation.

The study also observed that diesel-biodiesel blends exhibited a relative increase in oxidation, proportional to the concentration of Jatropha biodiesel in the blend. However, these blends displayed longer induction times and greater thermal stability than pure B100 biodiesel. By the study's conclusion, Jatropha biodiesel showed a reduction in oxidation stability by up to 55%. Yet, its fuel properties remained within the acceptable range for diesel blend usage, underlining its potential viability as a diesel blend component even after long-term storage.

Jatropha oil was effectively transformed into biodiesel with a high yield, meeting commercialization standards. Despite undergoing degradation from auto-oxidation during storage, the blends with diesel exhibited improved stability. While not as oxidation-resistant as highly saturated biodiesels, with appropriate storage conditions and the use of antioxidants, Jatropha biodiesel could likely fulfill stability requirements for commercial use. Future research should continue to evaluate the effectiveness of natural and synthetic antioxidants in stabilizing Jatropha biodiesel, explore oils from different regions for composition and oxidation susceptibility variations, and conduct engine testing to assess long-term operational stability under real-world conditions.This work demonstrated the promising potential of Jatropha as a viable and sustainable biodiesel feedstock.

Conflict of Interest Statement

The author(s) declared no potential conflicts of interest.

Funding declaration

No financial support was provided.

Data availability

The data supporting this study's findings are available from the corresponding author upon reasonable request.

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