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CHARACTERIZATION OF CALCINED PAWPAW DRIED LEAVES ASH AT DIFFERENT TEMPERATURES AS HETEROGENEOUS CATALYST FOR BIODIESEL PRODUCTION

[1*]Oyedele, O.A, [2]Jekayinfa, S. O, and [3]Alade, A.O

[1]Department of Agricultural and Bio-Environmental Engineering, The Federal Polytechnic Ado Ekiti, Ekiti State, Nigeria
[2]Department of Agricultural Engineering, Ladoke Akintola University of Technology Ogbomoso, Oyo State, Nigeria
[3]Department of Chemical Engineering, Ladoke Akintola University of Technology Ogbomoso, Oyo State, Nigeria

ABSTRACT

The increasing global demand for energy has fueled a search for sustainable and renewable alternatives to traditional fossil fuels. Biodiesel, produced through transesterification of triglycerides, stands out as a promising candidate. This study explores the potential use of calcined pawpaw dried leaves ash (CDPLA) as a heterogeneous catalyst for biodiesel production. Pawpaw residues, often overlooked, are rich in bioactive compounds with various applications, and this study aims to harness their potential in the biodiesel production process. Calcination of dried pawpaw leaves at different temperatures (500-900 °C) resulted in CDPLA, which was characterized using X-ray fluorescence (XRF), X-ray diffraction (XRD) and Fourier Transform Infrared (FTIR) spectroscopy. The chemical analysis revealed a significant composition of CaO and SiO_2 in the CDPLA, -indicative of its potential catalytic activity. The XRD patterns suggested phase transformations, with $Ca(OH)_2$ and SiO_2 identified as major components. FTIR analysis further supported the presence of functional groups associated with CaO, highlighting the potential use of CDPLA as a biodiesel catalyst. The study emphasizes the environmentally friendly and cost-effective nature of bio-derived catalysts, with pawpaw residues offering an abundant and sustainable source. The research demonstrates the feasibility of utilizing CDPLA as a heterogeneous catalyst in transesterification reactions for biodiesel production. The findings contribute to the growing interest in bio-based catalysts, showcasing the potential of agricultural residues in addressing environmental concerns and promoting sustainable energy practices.

Keywords: *Heterogeneous Catalyst, Pawpaw Residues, Transesterification, Calcination, Bio-derived Catalyst*

INTRODUCTION

The global need for energy is escalating remarkably due to population growth, urbanization, and the expansion of industrialization and transportation. Beyond electricity, fuel plays a crucial role as a primary energy source for sustaining contemporary society, with fossil fuels predominantly meeting the majority of the energy requirements (Mares *et al*., 2021, Nath *et al*., 2023).

In recent decades, there has been a significant focus on energy and associated concerns globally. The depletion of fossil fuel reserves, coupled with escalating fuel prices, has prompted widespread interest in exploring alternatives that are both renewable and sustainable, while also being efficient and cost-effective (Singh *et al*., 2019, Gohain *et al*., 2020). Many countries have established targets to incorporate a portion of their energy needs from renewable sources, with biodiesel emerging as a leading candidate for substitution in the automotive fuel sector. The production of biodiesel involves the transesterification of oils or fats' triglycerides with

alcohol (either methyl or ethyl) in the presence of an appropriate catalyst (Gohain *et al*., 2020).

Biodiesel comprises alkyl esters of fatty acids sourced from renewable materials like vegetable oils or animal fats. It is produced through either the esterification of fatty acids or the transesterification of acylglycerols with primary alcohols. The length of the alcohol chain significantly affects the conversion rate, and an excess of alcohol is usually necessary due to the reversible nature of the reaction. This process can be facilitated by an acidic, basic, or enzymatic catalyst (Rizwanul Fattah *et al*., 2020, Mares *et al*., 2021)

According to related literature (Oloyede et al., 2022a, 2022b, 2023a, 2023b), biodiesel has had a notable surge in industrial production and usage globally during the past twenty or thirty years. With 14 million tons produced in 2018, the European Union is the world leader in biodiesel production, followed by the USA with 7 million tons (Lv *et al*., 2021). The total amount produced in 2018 was close to 41.2 million tons. Through the transesterification process, oils and fats produced from plants, animals, and microbes are primarily converted into biodiesel (Nguyen *et al*., 2021).

The quality of the feedstock (amount of free fatty acids), the kind of acyl acceptor (alcohols or methyl acetate), and the type of reactions (non-catalytic, chemically catalyzed, and enzymatic) all have a major impact on the process' efficiency (Faruque *et al*., 2020; Rizwanul Fattah *et al*., 2020). One of the most concentrated research areas for creating effective catalysts for the transesterification reaction is improving reaction efficiency. As a result, researchers have looked into a variety of catalysts, including enzymes and alkali and acid catalysts (Talha and Sulaiman, 2016; Thangaraj *et al*., 2019).

The chemical catalysts commonly used for biodiesel production include KOH, NaOH, H_2SO_4, and HCl) (Buasri *et al*., 2013; Saifuddin *et al*., 2015). These chemical catalysts effectively catalyze the reaction, but they have several drawbacks, including complicated purification procedures in the downstream process, unfavorable environmental consequences, and limited reusability (Nguyen *et al*., 2021). Therefore, studies have shifted from this to the use of bio-derived catalysts for biodiesel production.

Bio-based catalysts, also known as biocatalysts, have gained more and more interest in the biodiesel manufacturing industry because they are readily available and environmentally friendly (Nguyen *et al*., 2021). The origins of these catalysts are natural. Three categories exist for them: biocatalysts (enzymes), acid catalysts, and alkali catalysts. Every kind of catalyst has benefits and drawbacks when it comes to producing biodiesel. There have been reports on the use of different biomass materials and synthesis techniques to create these catalysts (Changmai *et al*., 2020). Different sources of catalysts have various catalytic activity.

Biocatalysts may be customized to meet individual needs, their use in the manufacture of biodiesel has hence garnered a lot of attention. It is easily recovered and reused for multiple catalytic reaction cycles, which may reduce the labour required during manufacture. The utilization of ash from leftover eggshells, animal bones, snail shells, etc. as heterogeneous catalysts for the generation of biodiesel has been the subject of the current investigations (Thangaraj *et al*., 2019; Etim *et al*., 2020; Oladipo and Betiku, 2020, Falowo *et al*., 2021).

Alkaline earth metal oxide heterogeneous base catalysts have been studied for the synthesis of biodiesel (Basumatary *et al*., 2018; Thangaraj *et al*., 2019; Changmai *et al*., 2020). It has been demonstrated that Calcium oxide (CaO) found in waste-derived ash can function as a heterogeneous

catalyst. This innovative approach to biodiesel manufacturing is cost-effective and environmentally beneficial (Mohammed *et al*., 2018). Calcium oxide is commonly used as a solid base catalyst in transesterification reactions. It is known for its high catalytic activity and is often supported on various materials to improve its stability and effectiveness (Mazaheri *et al*., 2021). Apart from CaO, others compound reported by literature that are active in the bio-catalysts activity for biodiesel production include: Magnesium Oxide (MgO), similar to calcium oxide, magnesium oxide is a solid base catalyst used in transesterification reactions. It exhibits good catalytic activity and stability (Julkapli and Bagheri, 2016). Zinc oxide (ZnO) is known for its catalytic activity. It and is often combined with other materials to enhance its performance.

The use of agricultural residues which are usually discarded freely to the environment has shown to be a means of controlling environmental pollution through their use as heterogeneous catalysts. Pawpaw, also known as papaya (*Carica papaya*), is a delectable, tropical fruit known for its sweet taste and health benefits. However, after consuming the flesh of the fruit, there are residues left behind such as the peel, seeds, and other plant parts. These residues are commonly disregarded, but they can have various uses and benefits. One of the primary residues of pawpaw is the peel. Pawpaw peels are rich in bioactive compounds like phenols, flavonoids, and carotenoids, which possess antioxidant properties (Devaki *et al*., 2015; Enenya *et al*., 2022). These compounds have been found to have potential applications in the pharmaceutical and food industries due to their health benefits. For example, a study by Enenya *et al*. (2022) demonstrated that pawpaw peel extract had anti-inflammatory and antioxidant activities, making it a potential ingredient for functional food development.

The seeds are another important residue of pawpaw. These seeds contain high amounts of fatty acids, proteins, phenolics, carotenoids, and enzymes (Lim *et al*., 2021). Researchers have explored the potential use of pawpaw seeds in food processing, cosmetic applications, and even as biofertilizers. For instance, a study by (Lim *et al*., 2021) investigated the potential use of pawpaw seeds as a natural preservative in cooked meat products due to their antimicrobial properties.

Pawpaw residues can also be utilized for animal feed production. The peel and other parts, including leaves and stems, of papaw have been considered alternative feed sources for livestock due to their nutritional composition. A study by Onu *et al*. (2012) and Odunsi (2018) examined the value of pawpaw latex as a dietary supplement in broiler chicken feed and found positive effects on growth performance. Pawpaw leaves are often regarded as residues after harvesting the fruit, but they also have various potential uses and benefits. These leaves are rich in bioactive compounds, including alkaloids, flavonoids, phenolics, and enzymes, which contribute to their medicinal properties. Research indicates several potential applications for pawpaw leaves, including traditional medicine, herbal supplements, and even as an agricultural resource.

Traditional medicine has long recognized the therapeutic properties of pawpaw leaves. They have been used for centuries in different cultures to treat various ailments such as malaria, dengue fever, digestive disorders, and inflammation. The presence of alkaloids, such as *carpaine* and *papain*, has been linked to antimalarial and antitumor activities (Onuekwusi *et al*., 2014; Sharma *et al*., 2020). Pawpaw leaves have attracted attention in the field of herbal supplements and functional foods. Studies have explored the potential use of leaf extracts in the formulation of dietary supplements due to their remarkable antioxidant and immunomodulatory

activities. Oloruntola (2019) reported that pawpaw leaf extract enhanced antioxidant enzyme activity and reduced lipid peroxidation in diabetic rats, suggesting its potential as a natural supplement.

In the field of agriculture, pawpaw leaves can also be utilized as a resource for organic fertilizers, biopesticides, and plant growth regulators. Researchers have investigated the use of pawpaw leaf extracts to control pests and diseases in crops. A study demonstrated the antifungal potential of pawpaw leaf extracts against common plant pathogens, such as *Fusarium solani* and *Rhizoctonia solani*. These findings suggest that pawpaw leaves could be a sustainable and natural option for crop protection (Oloruntola, 2019; Sharma *et al*., 2020).

Pawpaw leaves, often considered as residues, are not just waste but possess significant medicinal and agricultural potential. Their bioactive compounds have been linked to antimalarial, antioxidant, and antidiabetic properties (Sharma *et al*., 2022). Furthermore, pawpaw leaves can be utilized in the formulation of herbal supplements, functional foods, and organic resources for crop protection. Despite the numerous uses of pawpaw residues through these applications, the leaves as the residue of pawpaw have not been explored by past researchers for biodiesel production. By exploring these uses, the residues of pawpaw can be utilized effectively, minimizing waste, and maximizing its benefits. This study therefore aimed at examine the active compounds that aid bio-catalyst for through characterization of the Dried Pawpaw Leave (DPL) to assess its energetic potential in biodiesel production.

MATERIALS AND METHODS

Materials collection and pre-treatment

The dried fallen leaves of pawpaw were picked from their parent trees at the Research Farm of the Department of Agricultural and Bio-Environmental Engineering, Federal Polytechnic Ado Ekiti. Sorting of the dirt and foreign matter from fresh leaves was carried out manually. The leaves were sundried for three days. The sun-dried sample was ground to granular using a pestle and mortar. The sun-dried leaves were further dried in the oven at 105 °C to a constant weight to remove moisture content. The ground sample was then sieved into a uniform particle size of 150 µm using a standard sieve through a mechanical shaker. The sieved sample was kept in airtight containers for further analysis. Sample preparation follows the method reported by Betiku *et al.* (2016); Oladipo *et al. (*2018) and Oladipo *et al*. (2020).

Catalyst Preparation

A portion of each DPL powder was kept as the raw sample for further usage. The other portions were calcined at varying temperatures of 500 to 900 ℃ at 100 ℃ intervals, for four hours per sample in a muffle furnace (Galenkamp) to remove carbonaceous and volatile matter from them and to obtain ash products. The calcined ash samples were put in a desiccator and then stored in airtight bags to ensure that no moisture interacted with the sample before further usage. The calcined ashes were designated as Calcined Dried Pawpaw Leaves Ash (CDPLA).The catalyst preparation followed procedures detailed by Betiku *et al*. (2016) and Oladipo *et al*. (2018).

Catalyst Characterization

The samples were characterized by X-ray fluorescence Spectrometer (XRF) method following the procedures of by Betiku *et al*. (2017) and Etim *et al*. (2020) using Rigaku ZSX Primus XRF machine equipped with an end window 4.kW R^H-anode X-ray tube. The phase composition of the uncalcined and calcined samples were determined by the XRD analysis of the sample with Bruker X-ray Diffractometer model operating with a CuKα radiation source (Kα=1.5406 A_O). Fourier Transform

Infrared (FTIR) was used to determine the active surface functional group.

RESULTS AND DISCUSSION

Chemical Analysis

The chemical compositions of the uncalcined and calcined PDL are given in Table 1. It can be seen that the uncalcined contains a large amount of are CaO (59.9 %), SiO_2 (16.1%). Chemical composition of DPL confirmed that DPL is a good biocatalyst for biodiesel production as several works have reported on using these metal oxides as heterogeneous catalysts in biodiesel production (Buasri *et al*., 2013; Sai Bharadwaj *et al*., 2019; Changmai *et al*., 2020; Etim *et al*., 2020).

The chemical composition of the calcined residues indicated similar trend as CaO and SiO_2 still retain the high composition and both increase with increasing temperature from 500 to 700 ^oC and then relatively reduce to constant value between 800 and 900 ^oC of calcination temperatures. Feaviour *et al*. (2020) reported that CaO, a metal-based catalyst has been the most studied catalyst material for biodiesel production, as it advantages of, long catalyst life, relatively high basic strength, high activity, and low solubility in methanol and requires only moderate reaction conditions.

Table 1: Major Chemical composition of uncalcined and calcined samples at different temperatures

			Composition (%)				
			Calcined temperatures for DPLA (^oC)				
S/N	**Oxide**	**Uncalcined sample**	**500**	**600**	**700**	**800**	**900**
1	SiO_2	16.10	20.94	21.19	24.08	23.32	23.13
2	Al_2O_3	3.19	6.70	5.31	4.66	4.60	4.71
3	CaO	59.92	25.24	26.62	27.21	27.08	27.00
4	Fe_2O_3	1.64	6.02	4.79	4.62	4.75	4.68
5	MgO	3.04	4.25	4.03	5.48	5.68	5.36
6	K_2O	5.92	22.73	21.28	21.78	21.12	21.27
7	P_2O_5	0.59	2.00	2.13	2.35	2.61	2.50
8	SO_3	4.15	1.32	1.30	1.63	1.49	1.32
9	MnO	0.35	0.56	0.24	0.33	0.31	0.34
10	TiO_2	0.28	2.10	2.10	2.02	2.00	2.05
11	Cl	4.28	1.30	1.27	1.31	1.44	1.53
12		LOI	5.30	8.34	4.52	5.54	6.02

Note: LOI means Loss of Ignition

Phase analysis

The XRD patterns (Figure 1) show a slightly difference between calcined DPL ash at different temperature, which suggests that phase transformation took place. The XRD patterns DPL are presented in Figure 1. The major peaks are observed at 2θ =23.0^o, 27.0^o, 29.0^o, 39.0^o, 43.2^o, 47.0^o, and 58.0^o at 500,700,800 and 900 ^oC calcination while the peaks for 600 ^oC calcination temperature have a little difference at 2θ = 24^o, 29.5^o, 31.0^o, 39.0^o 44.0^o and 48.0^o. These are characteristics of $Ca(OH)_2$ (Gaurav *et al*., 2019; Oladipo and Betiku,

2019). Also, peaks at 2θ = 31.2° were attributed to SiO_2. The two peaks indicate the crystal phase. Thus, the catalyst tends to form crystals that are made up of CaO, $Ca(OH)_2$, and SiO_2. The XRD result is in good agreement with the FTIR result which also confirmed both CaO and SiO_2.

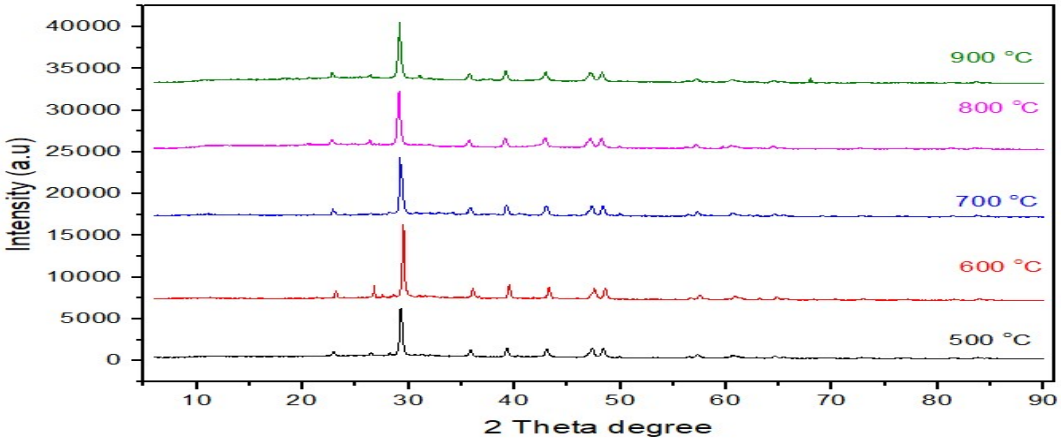


Figure 1: XRD spectrum for DPL

FTIR of calcined Dried Pawpaw Leaves (DPL)

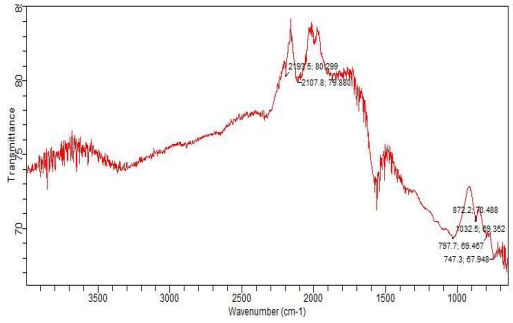
The result in Figures 2a-e shows the major peaks obtained from the Fourier Transform Infrared (FTIR) analysis of DPL. The DPL sample calcined at different temperatures exhibited the following band: 3695.7 cm^{-1} for O-H stretching at 700 °C and 3391.9 cm^{-1} for N-H stretching nitriles groups at 600 °C. The bands of 2193.5, 2119.0, and 2115.3 cm^{-1} appeared at temperatures of 500, 800 and 900 °C respectively as seen in Figures 4.5a, d and e. The observation band at 1654.9 and 2115.3 cm^{-1} was attributed to the C=C stretching vibration of alkene groups (Li *et al*., 2019).

The higher temperatures of calcination of 700 to 900 °C, exhibit an additional band around 1405 cm^{-1} which was related to the S=O bond stretching (Feaviour *et al*., 2020). The highest number of peaks that indicate the presence of CaO was at 900 °C calcination temperature. The CaO appeared at five different peaks as summarized in Table 2. These results suggested that the sample of DPL calcined at 900 °C would be most suitable catalyst. Etim *et al*. (2021) calcined pawpaw peels at this temperature.

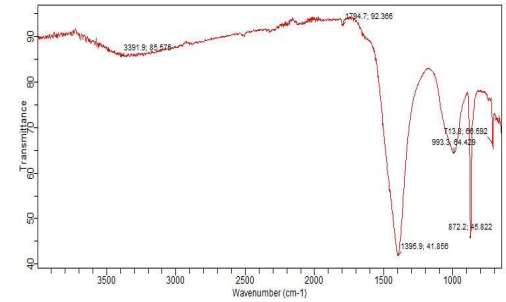
CONCLUSION

The followings conclusions can be drawn from this study:

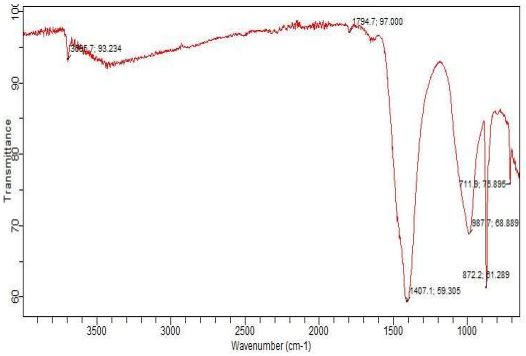
The XRF result showed that the major oxide in DPL is calcium oxide. The XRD results showed that dried pawpaw leaves ash structure have been changed from amorphous nature to crystalline materials. As temperature increases, the PDL ash colour changes from black to grey and white which indicates that the carbon content present in the samples were reasonably reduced. The result of this finding suggested that DPL will be suitable to be used as heterogeneous catalyst in transesterification process.

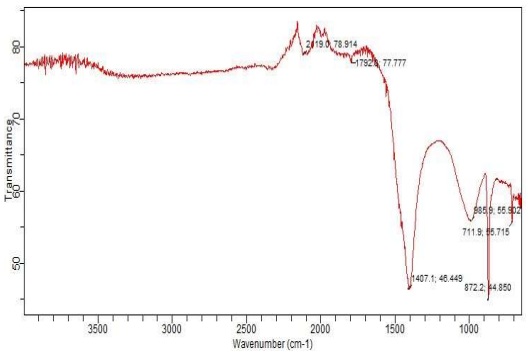


(a)

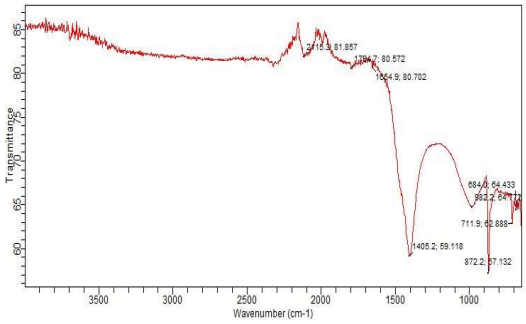


(b)





(c) (d)



(e)

Figure 2: FTIR spectral for DPLA at (a) 500 ºC, (b) 600 ºC, (c) 700 ºC, (d) 800 ºC and (e) 900 ºC

Table 2: Summary of peak from FTIR analysis for calcined DPL

Peak Wavelength (cm^{-1})					Functional Group
500 ºC	600 ºC	700 ºC	800 ºC	900 ºC	
		3695.7			O-H stretching
	3391.9				N-H stretching
2193.5			2119.0	2115.3	CΞC stretching
2107.8					CΞC stretching
	1794.7	1794.7	1792.8	1794.7	C=O stretching
				1654.9	C=C stretching
		1407.1	1407.1	1405.2	S=O stretching
	1395.9				S=O stretching
1032.5					S=O stretching
	993.3	987.7	985.9	982.2	C=C bending

	872.2	872.2	872.2	872.2	872.2	C-H bending
	797.7	713.8	711.9	711.9	711.9	C-H bending
					684.0	C-Br stretching
No. of CaO indicator	4	3	4	4	5	

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