Journal of
Physical Sciences
(JPS)

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CARI
Journals

Journal of Physical Sciences
ISSN: 2791-2485 (Online)
Vol.7, Issue No.1, pp 24 – 48, 2025
www.carijournals.org

Potential of *Jatropha Curcas* Seeds oil for Polyester-amides Production

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Accepted: 28th Mar, 2025, Received in Revised Form: 29th Apr, 2025, Published: 26th May, 2025

Abstract

Purpose: *Jatropha curcas* potential as feedstock for polyester-amides production was studied. Polyesteramides are valuable biomaterials in the activities of petroleum industries where they are utilized as oil field chemicals.

Methodology: *Jatropha curcas* fruits were collected in Ibadan and its environment. Seed oil of this fruit was extracted using n-hexane and concentrated. The phenol *Jatropha curcas* biopolymer (PJCB) and cresol *Jatropha curcas* biopolymer (CJCB) were prepared by polymerizing the oil with phenol and cresol via cationic polymerization method. Polyacrylamide (PAA) was prepared through free radical polymerization technique. Polyester-amides were prepared by polymerizing PJCB, CJCB and polyacrylamide (PAA). They were characterized using FTIR for functional groups modifications, ^{1}H-NMR for changes in the chemical environments and Viscometry techniques for mean molecular weight determination.

Findings: The oil yield is 55.60%. Iodine value ($gI_2/100g$) is 105.33±1.78, 97.32±0.01, 101.33±0.66 for oil, PJCB and CJCB respectively. FTIR band shifts at 1649.48cm^{-1} and 1600.35cm^{-1} confirm C=C stretch of aromatics in PJCB and CJCB. PAA formation was confirmed with C=O stretch at 1680.00cm^{-1}. Peaks at 3234.28cm^{-1} and 3097.14cm^{-1} assigned to N-H stretching vibration of amides confirmed polyester-amide. The ^{1}H-NMR spectra showed peaks at δppm, 7.25 and 7.06 indicating aromatics for the PJCB &CJCB. δppm at 7.18 confirms amide protons in PAA. δppm 6.86 and 8.69 confirm amide formation while peaks at δ2.36ppm and δ3.11ppm were evidences for ester formation. These peaks confirm the new materials as polyester-amides. The mean molecular weight (g/mol) for PJCB, PJCB-PAA, CJCB and CJCB-PAA are 1.041x109, 1.39187x10^{12} 9.929x109 and 1.07919 x 10^{12} respectively.

Unique Contribution to Theory, Practice and Policy: *Jatropha curcas* oil is viable in formulating polyester-amides for industrial usages.

***Keywords**: Polyester-Amide, Biomaterial, Phenol, Cresol, Polymerization, Jatropha Curcas.*

1. Introduction

Jatropha curcas has been widely reported to be a technical oil with the ability to be modified into several useful products. It belongs to the biomass and renewables family that has been seen as one of the long term partial replacement to fossil fuel in the production of feedstock for the petrochemical industries' need [1]. This is because *Jatropha curcas* oil is rich in unsaturated fatty acids such as oleic acid, linoleic acid, myristoleic acid and lots more. The percentage of unsaturated fatty acids has been reported to be above 50% of the oil yield[2]. This offers it as a viable material to the polymer industries for addition polymerization processes such as cationic, anionic and free radical polymerization [9, 17]. It can therefore be relied upon to generate the feedstock similar to alkenes such as ethylene, propylene, polyvinylchloride and so on which are important feedstock in the petrochemical industries[4, 14]. *Jatropha curcas* oil as a triglyceride is also a fatty ester [15]. This shows that it possess important functional groups like ester, alkene and triglycerides that can be modified via various chemical processes and in the presence of suitable reagents to produce trans esterified esters, fatty acids, fatty alcohols, fatty amides, fatty amines and fatty sulphonamides. This shows that the oil has promising potentials in the production of useful materials that are biodegradable and environment loving [3, 6, 10].

Jatropha curcas is cultivated annually, which makes it available yearly[2, 3]. It has high oil yield which makes it commercially viable. It is environmentally friendly since it doesn't generate poisonous byproducts like CO, nitrogenous compounds and sulphur compounds. The plant's oil has also been reported to be useful in developing fatty acid methyl esters (FAME), important in pharmaceutics, medicine biotechnology and drug development. All these viable benefits of *Jatropha curcas* makes it an economic plant[2, 7, 21]. The oil has however been sparsely reported for oil field chemicals development.

It has however been reported that lots of chemicals are used for oil production activities. These are classified into drilling and production activities. These major operations are reported to provide a total yearly market for more than 200,000 tons of chemicals [8, 16]. About half of these two hundred thousand tons are released into the environment annually as pollutants. This is because these chemicals release toxic compounds such as oxides of nitrogen and sulphur into the environment in large amount[11, 18]. These have heavily contributed to the release of green-house gases like CO_2, CH_4, SO_2,

SO_3, H_2S, NO_X and hydrocarbons. Petroleum has been the main source of producing these chemicals. As a result, the oil production businesses are under significant regulatory pressure to switch to using greener chemicals in their different activities in order to protect the environment [20, 21]. The plant '*Jatropha curcas*' with its numerous potentials and its biodegradable tendencies can serve as a component of the viable alternatives in producing these essential chemicals for the oil industries [20].

In the same vein, environmental risks associated with fossil fuels, institutional tendencies toward sustainable development principles, the finite nature of crude oil, political landscapes of petroleum-producing countries, pricing policy uncertainties, and similar factors are reasons why the biomass are becoming the real alternative to fossil fuel in developing oil field chemicals[4, 19].

Based on the United Nations policies on the green chemistry objectives, It has become essential that industries think about ideas that will save the planet for coming generations. One of the suggested ways to achieve this is the consideration of feedstock sources that are expected to be safe, and ecologically acceptable to address the fossil fuel menace [5, 14]. Here, *Jatropha curcas* plays an acceptable vital role since it is ecologically safe.

Generally, chemicals derived from plants and animals exhibits varieties of physicochemical characteristics that are significant to the oil and gas sectors and have uses in key areas of this sector. These chemicals are expected to be manufactured or modified using various polymerization processes. These compounds come in form of polyols, polyesters, polyamides and lots more[8]. The areas of their important applications include: mud synthesis, base oil, lubricants, corrosion inhibitors, emulsifiers, shale inhibitors, wax inhibitors, enhanced oil recovery, surfactant, drilling operations, emulsion breaker, demulsifier and shale stabilizers. They are also useful as absorbents/adsorbent for cleaning up and remediations of polluted oil sites. Because of this, the offshore oil and gas sectors ought to constitute a sizable and ongoing market for chemicals, many of which can be created by employing feedstock from seed oils with similar features to petroleum[12, 16].

A significant amount of researches have been reported on the synthesis of biochemicals with various architectural designs, which have the potential to produce elastomeric, semi-crystalline, and amorphous materials. Additionally, a few of them may result in solvents and grease that are crucial and important to the activities of the oil and gas industries. . These chemicals were reported to be developed from various polymerization techniques and other suitable chemical processes. Some of these chemicals show improved efficiency over traditional petroleum based chemiclas. According to Willey et al. (2007), synthetic greases have a number of benefits over traditional hydrocarbon-based greases, such as high viscosity with good pumpability, local torque, the capacity to operate at lower temperatures, and superior thermal and oxidative durability[14]. Due to these advantages, several commercially available synthetic forms of green polymeric chemicals have been developed for a range of specialized applications in the oil industries [12, 13]. Some of these green polymeric chemicals are eventually characterized as polyols, polyamines, polyesters, polyamides and lots more although they adopt the best name that suits their applications in the oil and gas industries. This research therefore probe into the development of polyester-amides from *Jatropha curcas* oil as possible oil field chemicals.

2. Materials and Methods

2.1 Materials

Seeds of physic nuts *(Jatropha curcas)*, acrylamide, phenol, cresol, distilled water, hydroquinone, Fenton's reagent (Hydrogen peroxide+ Ferric ammonium sulphate in 1:1), ethanol.

2.2 **Methods**

2.2.1Extraction and Grafting phenol and cresol unto the vegetable oils backbone

Jatropha curcas seeds were obtained in Ibadan areas of Oyo state, Nigeria and authenticated at the Herbariun unit of the Botany Department, University of Ibadan, Ibadan, Nigeria with NIH-23014. The seeds were removed from the fruits by mechanical means. The oils were extracted from the fruit through sohxlet extraction using n-hexane as the solvent. The extracted oil was alkylated separately with phenol and cresol via grafting technique as shown in Figure 1. Melted phenol and cresol and the triglyceride were mixed in batch reactors in the presence of nitrogen gas to provide inert environment in ratios 1:4 in separate reactions. The mixture was homogenized for few minutes. The reaction mixture (1% of the combination) was combined with perchloric acid ($HClO_4$) as catalyst. They were re-homogenized, and then allowed to proceed for six hours at 90^0C in an inert atmosphere created by utilizing nitrogen gas. After the reaction materials were polymerized for six hours, they became reddish-brown. The reaction products were observed to have grown very viscous after four hours of polymerization. The final product were reddish-brown viscous liquids. The processes were halted after six hours. They were allowed for twenty minutes to cool. To make sure that the unreacted phenol, unreacted triglycerides, and acid catalysts were neutralized and eliminated, powdered $Ca(OH)_2$ were added to the reaction mixture. To get rid of any remaining impurities, ethanol was used to wash the finished product. The resultant alkylated products were dried for 48 hours over sodium sulfate. The products obtained were stored in tightly fitting metal containers until they were needed for further application and analysis. ^{1}H-NMR, FTIR and iodine value were used in their analysis. The products were used as starting materials for the preparation of the polyester-amides.

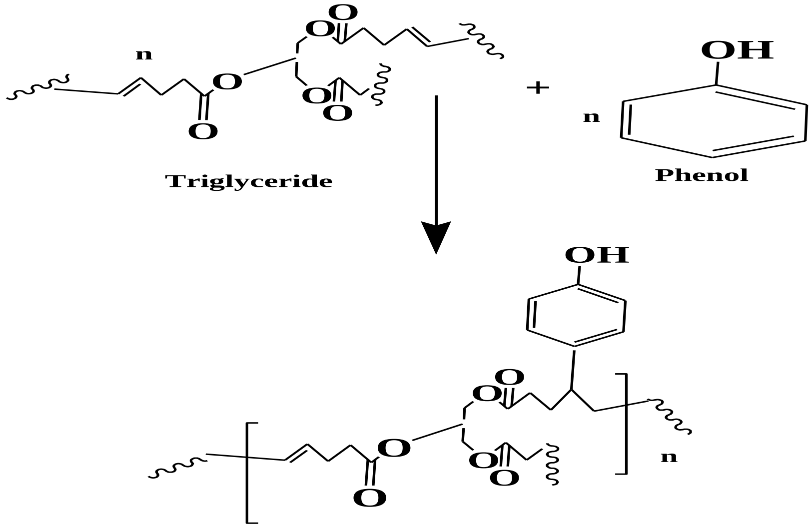


Figure 1: Cationic polymerisation scheme for grafting phenol to JCO triglyceride

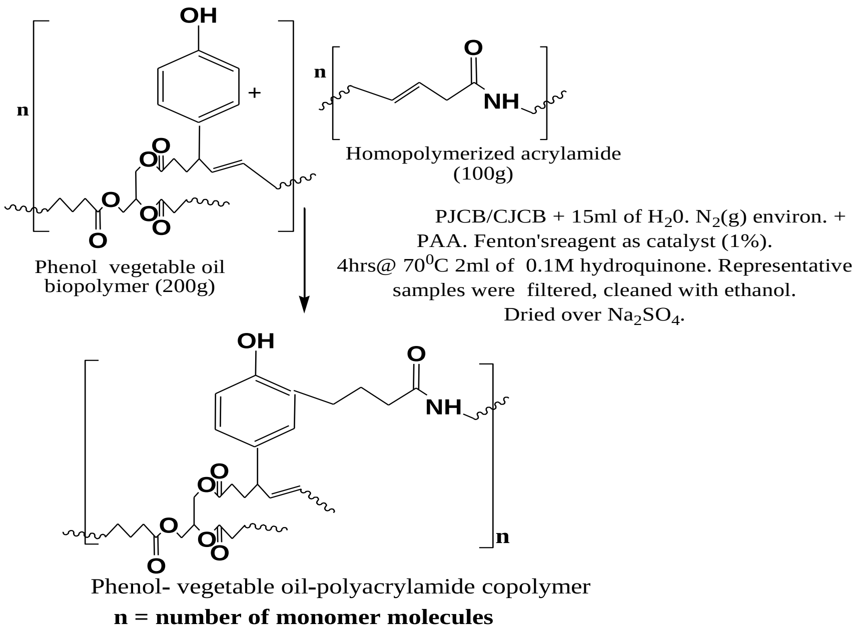
2.2.2Preparation of polyester-amides

2.2.2.1 Preparation of polyacrylamide (PAA)

1.4 litres of 30% wt./vol. acrylamide in water was transferred into a three necked batch reactor, 30ml of 0.05M $NaNO_3$ solution was added. The mixture was thoroughly stirred for 20 minutes under nitrogen gas. Fe^{2+}-H_2O_2 (Fenton's reagent) was added as initiator and this was stirred for 5 minutes. Nitrogen gas was bubbled through for few minutes and the reaction continued for the next four hours. After the desired reaction time, the reaction was terminated by adding 0.2 g hydroquinone powder and the contents were stirred for another 10 minutes before being halted. The homopolymer was cleaned with ethanol to remove unreacted acrylamide, remnants of $NaNO_3$ solution and the catalyst. The product was allowed to dry for half a day over sodium sulphate to ensure the total removal of the moisture content. It was kept in a cool dry place until the period of further application and analysis. The product was analysed with FTIR and ^{1}H-NMR.

2.2.2.2 Copolymerization reactions.

The scheme of the polyester-amides grafting process follows the sequence of reaction in Figure 2. PJCB and PJCB each were loaded into different batch reactors and mixed with 15 ml of distilled water. Each of the mixture was thoroughly stirred for a few minutes under nitrogen gas. This was followed by adding homopolymerised polyacrylamide in ratio 2:1. The mixture was stirred for 5 minutes and was heated under nitrogen gas at 70ºC for 25 minutes. The temperature was increased to 80ºC for 6 hours for the polymerisation process. The polymerisation reaction process was terminated by adding calculated amount of hydroquinone and the contents were stirred for another few minutes before opening the reactor to collect representative samples of products. The product obtained was separated from the unreacted homopolymer by cleaning a representative samples of the product with 5ml of ethanol several times until the unreacted PAA, PJCB, CJCB and catalyst could not be detected. The final products were brown in colour. The products were dried over sodium sulphate for 48 hours. They were transferred into separately labeled glass containers for analysis.



PJCB/CJCB + 15ml of H_20. N_2(g) environ. + PAA. Fenton'sreagent as catalyst (1%). 4hrs@ 70^0C 2ml of 0.1M hydroquinone. Representative samples were filtered, cleaned with ethanol. Dried over Na_2SO_4.

n = number of monomer molecules

Figure 2: Schematic process for the preparation of polyester-amide.

2.3 Polyesteramide analysis.

The synthesized materials and their precursors were analysed using FTIR for changes in functional groups, ^{1}H-NMR for changes in chemical environments and viscometry technique for molecular weight estimation using Mark-Hauwink-Sakurada relations.

2.4 FTIR Analysis

The oil sample, intermediates and polyester-amides were qualitatively examined by the FT-IR method. These were obtained on a Perkin-Elmer Fourier transform infrared spectrometer. The spectra were recorded between 4000-350 cm^{-1} using KBr disc.

2.5 NMR Analysis.

The oil sample, intermediates and the polyester-amides were quantitatively and qualitatively examined via ^{1}H-NMR technique. The results were gotten on a Bruker av400 MHz NMR.

2.6 Viscometry Technique

This was used to obtain the intrinsic viscosities for the polyester-amides and their precursors. The Oswald capillary viscometer was used to determine the solvent flow rate and the solvent-polymer ratios. Toluene was used as the solvent. Various concentrations were calculated for the various samples. The flow rates were obtained from the viscometer, regulated water bath and a stop watch.

The relative viscosities (η_r) were obtained from the relation:

Efflux time of solution (t)

Efflux time of solvent (t_0) $\eta_r = t/t_0$

The specific viscosities (η_{sp}) were obtained from the relation:

Efflux time of solution (t) - Efflux time of solvent (t_0)

Efflux time of solvent(t_0) $\eta_{sp} = t - t_0/t_0$

The reduced viscosities (η_{red}) were obtained from the relation

Specific viscosity

Concentration $\eta_{red} = \eta_{sp}/C$

The inherent viscosities(η_{inh}) were obtained from the relation

In (Relative viscosity)

Concentration $\eta_{inh} = In\eta r/C.$

Where η_{sp} = specific viscosity, C. = Concentration (g/ml)

Graphs were constructed by plotting reduced viscosities versus concentration and inherent viscosities against concentration. The intrinsic viscosities were determined by extrapolating the two straight lines graphs to zero concentration as shown in Figure 3 below. The equations of the straight-line graphs were obtained and were simultaneously analysed. The intercept of the two graphs is called the intrinsic viscosity. The differences between the slopes of the relative and specific viscosities were not less than 0.5 and this was used to check the quality of the flow rates. From the values of intrinsic viscosity, the molecular weights of each polyester-amide and its precursors were estimated using Mark-Houwink-Sakurada equation [8].

$$\eta = KM^a$$

Where K and a are constants based on solvent-polymer concentrations. M equals molecular weights. The constants employed for this work are based on polymer -solvent ratios of polyamides and polyesters where toluene has been employed as solvents. The values were taken from American Polymers standard.

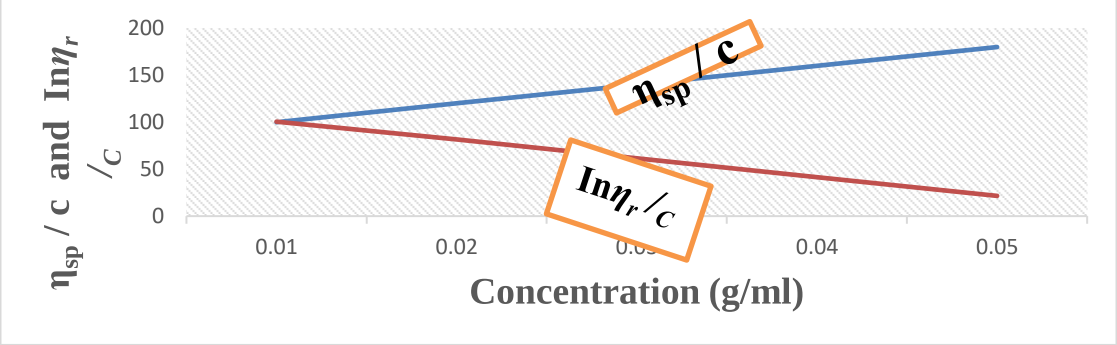


Figure 3: Sample of how the intrinsic viscosities were determined.

3.0 Result and discussion

Table 1: **Physicochemical analysis of oil**

Samples	Oil yield (%)	Iodine value of oils (gI_2/100g)	Iodine value of Biopolymers from oil(gI_2/100g)		Peroxide value (meq/kg)	Saponification value (mgKOH/g)	Free fatty acids (%)
Jatropha curcas	55.6%,	105.33±1.78	PJCB	CJCB	0.32±0.05	196.35±0.77	9.33±0.02
			97.32±0.01	101.33±0.66			

Mean value ±standard deviation (n=3).

Jatropha curcas seeds (JCO) yield 55.6% as oil. This is presented in Table 1. The value is in tandem with values for oils commonly deployed for polymerization reactions and commercials. The average iodine value (gI_2/100g) of the oil is 105.33±1.78. This suggests that the oil is semi drying. They are in tune with those reported in literatures such as sesame (104g/100g), rapeseed (104g/100g), Cottonseed oil (98g/100g). [1, 13]. As such, the iodine values suggest that the oil contains a high level of unsaturation that can make it an important feedstock for chain growth polymerization processes. The alkylated products (PJCB and CJCB) however showed lesser iodine values to suggest that the grafting reactions took place at the vinyl moiety in the triglyceride backbone. The average peroxides value (meq/kg) is 0.32±0.05. The value shows that the oil is not rancid since the value is within the range expected for good oils. The mean saponification value (mgKOH/g) of the oil is 196.35±0.77. It shows that the oil is not adulterated. The high saponification values of the samples indicates the presence of high percentage of fatty acids [12]. The saponification values agree with those found in the literature for same and similar oils. The high saponification values show that the oils possess the potential to be employed as feedstock in industrial processes [14]. The free fatty acids of the oils(%) is 9.33±0.02. The value is in tandem to those reported for *Jatropha* oil by other authors. From the physicochemical parameters, the oil shows viable potential in developing good feedstock for industrial processes.

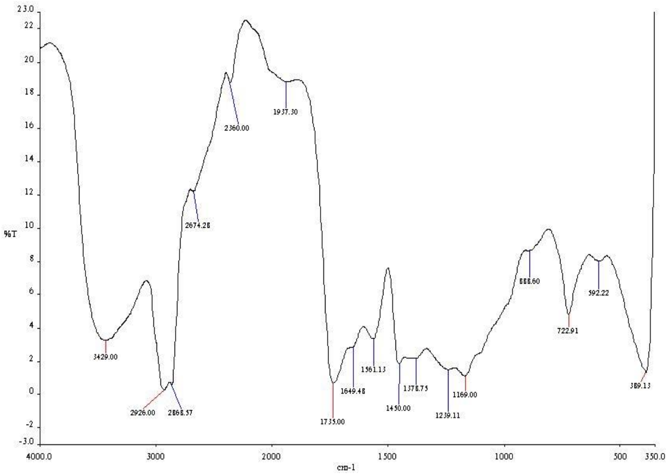
3.2 FTIR Analysis of synthesized polyester-amide and its precursors

The modifications that occur from the starting *Jatropha curcas* oil to the final polyester-amide were observed using FTIR studies. This was done by observing the changes that occur to the various functional groups in all the stages. The details is presented in Figure 4 and are labeled A, B,C,D E & F. A showed carbonyl group stretch at 1727.65cm^{-1}, C-H stretch at 2926.75cm^{-1} and 2860.35cm^{-1} appearing as a doublet. C-O stretch was found at 1165.29cm^{-1} 1241.95cm^{-1} and 1108.15cm^{-1} within the finger print region to confirm the ester/triglyceride functional group in the oil moiety. C≡C stretch of alkynes was also found at 2346.70cm^{-1} to confirm unsaturation in the oil. The peak at 3436.80cm^{-1} is an evidence of O-H peak of phenol. C=C stretch of alkene with a medium intensity was also found in the Jatropha spectrum at 1597.53cm^{-1} to establish the oil as being rich in unsaturation and phenolic. Several other prominent peaks within the finger print region include 1456.90cm^{-1} which confirms C=C bend of substituted alkene. C≡C stretch of alkynes was also found at 2346.70cm^{-1} in the sample. C-H bend of alkene with strong intensity was found at 747.15cm^{-1} to further establish unsaturation in the seed oil. The IR analysis shows that *Jatropha curcas* seed oil is an unsaturated fatty ester. The result complements the physicochemical analysis results earlier discussed for the sample. It also establishes that the oil can be used as feedstock for producing precursors for chemical industries.

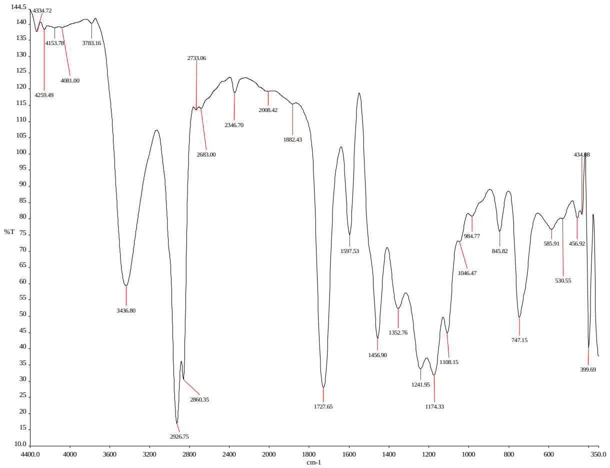
Spectrum B is the FTIR of the phenol *Jatropha curcas* biopolymer (PJCB) made from the oil (A). The spectrum showed the triglyceride/ester carbonyl peak at 1735.00cm^{-1}. C-H stretch occurring as doublet was found at 2926.00cm^{-1} and 2868.57cm^{-1}. The C-O peaks occurred in the finger print region with strong intensities at 1169.00cm^{-1} and 1239.31cm^{-1}. They showed that the initial ester/triglyceride group is still present in the new material. O-H stretch of phenol was found at 3429.00cm^{-1}.. C=C stretch of aromatics occurred at 1649.48cm^{-1} and 1561.13cm^{-1}. These peaks were not found in the vegetable oil spectrum and hence are evidences for the grafting of phenol to the triglyceride backbone. Other notable peaks in the finger print region include 722.91cm^{-1} and 888.60cm^{-1} which are due to di and mono substituted C=C bend of aromatics in the phenolated intermediate. They also confirm that the grafting took place. Another intermediate made from the oil is cresolated *Jatropha curcas* and the spectrum is presented in Figure 4C. Triglyceride /ester carbonyl peak stretch occurred at 1721.00cm^{-1}. C-H stretch peaks appearing as a doublet occurred at 2931.00cm-1 and 2808.37cm^{-1}. C-O stretching vibration within the finger print region occurred at 1227.00cm-1 and 1016.83cm^{-1}. These set of bonds confirm the ester/triglyceride functional group in the starting material to be present in the cresolated intermediate(C). The spectrum also show phenolic O-H peak at 3398.00cm^{-1}. The evidence of this peak was further substantiated with the presence C=C stretch of aromatics vibration at 1600.35cm^{-1} and

1469.00cm^{-1}. Also, Substituted C=C bend of aromatics were found at 818.67cm^{-1}, 747.01cm^{-1}, 700.51cm^{-1} and 880.05cm^{-1} within the finger print region. These set of bands serve as evidences that the cresol was grafted to the triglyceride backbone. Similar observation was found in spectrum B as the phenolated intermediate. The two intermediates and the polyacrylamide were used for copolymerization process that led to the polyesteramide. The Spectrum D in Figure 4 is that of the polyacrylamide that was synthesized. C=O vibrational stretch occurred at 1680.00cm^{-1} . C-N stretch band peak occurred at 2285.71cm^{-1}. C-H bend of alkenes was found around 615.02cm^{-1} to show that the PAA homopolymer is unsaturated. Polymerizing PJCB and PAA gives the copolymer in spectrum E. The spectrum shows comparable features with the starting materials. It shows the triglyceride group and the amide moiety. In E, C=O stretching vibration occurred at 1727.22 cm^{-1}. C-H stretch appearing as a doublet was found at 2930.00cm^{-1} and 2862.85cm^{-1}. C-O stretch occurred at 1108.03cm^{-1} and 1167.87cm^{-1} within the finger print region. These peaks were found in the oil (Spectrum A) in the PJCB intermediate (Spectrum B) and the phenolated grafted polymer (Spectrum E). All these bands confirm that the ester functional group is still present in the new material. C=C stretch of aromatics occurred at 1452.83cm^{-1} and 1535.89cm^{-1} as evidence of the existence of phenolic groups in the new material. Conjugated C=C stretch of alkenes peak occurred at 1608.79cm^{-1} to show unsaturation in the spectrum of E. C-N peak was found at 1336.29cm^{-1}. N-H stretch of medium intensity which appears as a doublet occurred at 3234.28cm^{-1} and 3097.14cm^{-1}. The peaks suggest that the compound contains amide group. The doublet suggests it's a primary amide. The presence of N-H, C-N and C-O bond in the new material shows it's a polyester-amide rather than polyester or polyamide. ^{1}H-NMR was further utilized to confirm this observation. C-H bend of aromatics were found at 968.05cm^{-1}, 845.85cm^{-1} and 743.90cm^{-1} hidden in the finger print region of spectrum E as additional evidences for the presence of phenolic in the new polyester-amide material. CJCB and PAA was also copolymerized and the FTIR spectrum is presented in Figure 4F. The spectrum of C=O stretch occurred at 1724.22cm^{-1}. C-H stretch vibration occurring as a doublet occurred at 2929.00cm^{-1} and 2862.21cm^{-1}. N-H stretching vibration was found at 3420.00cm^{-1}. C-N stretch similarly occurred at 1355.95cm^{-1}. These peaks confirm the amide functional group in the new material. However, C-O vibrational bands occurred at 1180.00cm^{-1} and 1113.73cm^{-1}. The presence of this peak confirm that the new material still possesses the ester/triglyceride functional group in the starting material. Coupling these pieces of information shows that the compound in spectrum F (Figure 4) is more of an ester-amide. Hence the name polyester-amide. ^{1}H-NMR was similarly deployed in further confirming this. The compound shows C=C stretch of alkenes at 1600.70cm^{-1} to show its unsaturated. C=C bends of alkenes and aromatics similarly occurred at 968.05cm^{-1}, 845.85cm^{-1} and 743.90cm^{-1} within the finger print region of the spectrum to confirm alkenes and phenolic. All these peaks shows that the compound in spectrum F is an unsaturated polyester-amide. The FTIR analysis suggests that both phenolic and cresolic polyester-amides were formed.

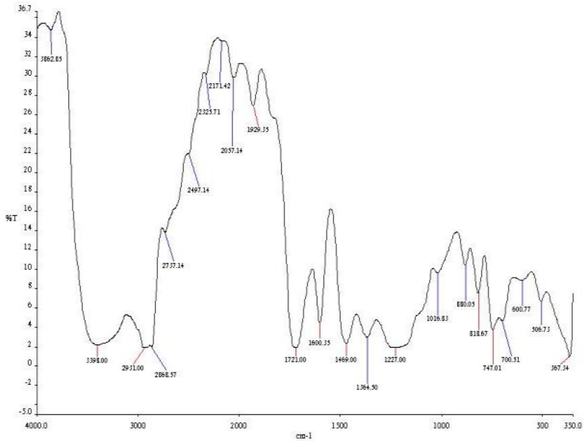
FTIR spectrum of Phenolated *Jatropha curcas* biopolymer (B)

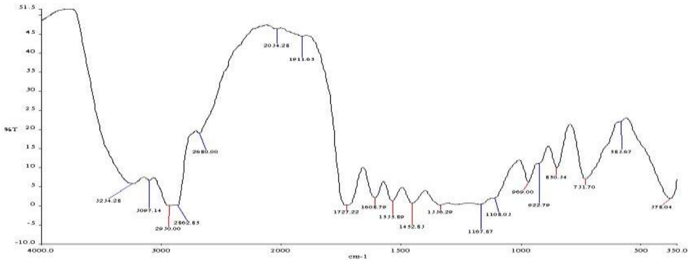


FTIR spectrum of *Jatropha curcas* oil (A)

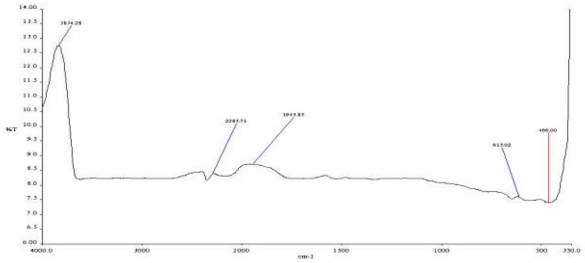


FTIR spectrum of cresolated *Jatropha curcas* polymer (C)

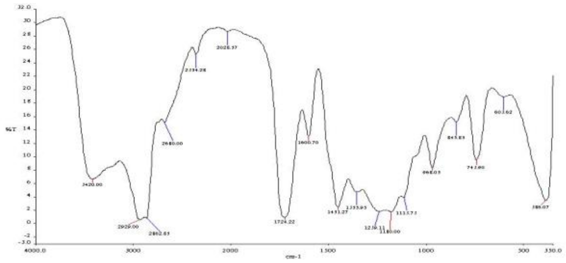




FTIR spectrum of Phenolated *Jatropha curcas* polyester-amide(E)



FTIR spectrum of polyacrylamide (D)



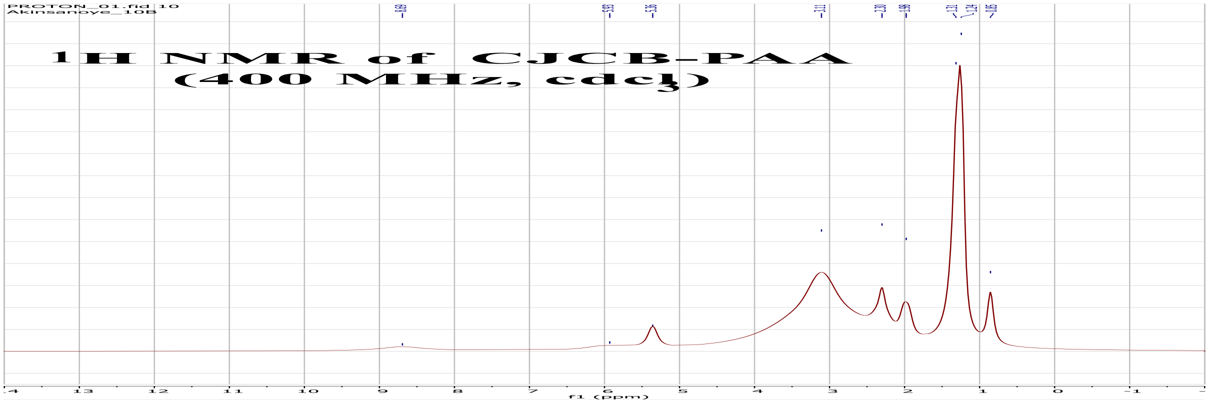
FTIR spectrum of Cresolated *Jatropha curcas* polyester-amide (F)

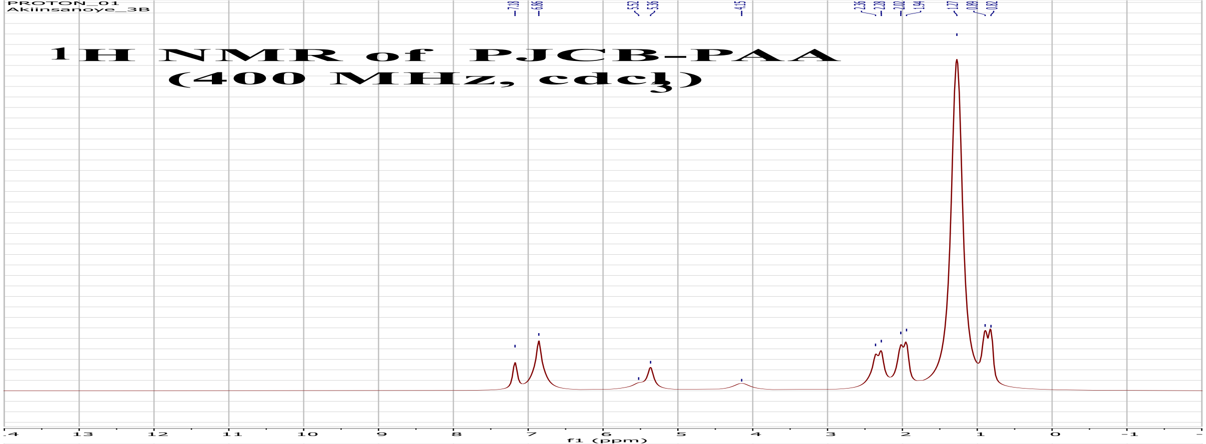
Figure 4: FTIR spectrum of synthesised polyester-amide and its precursors

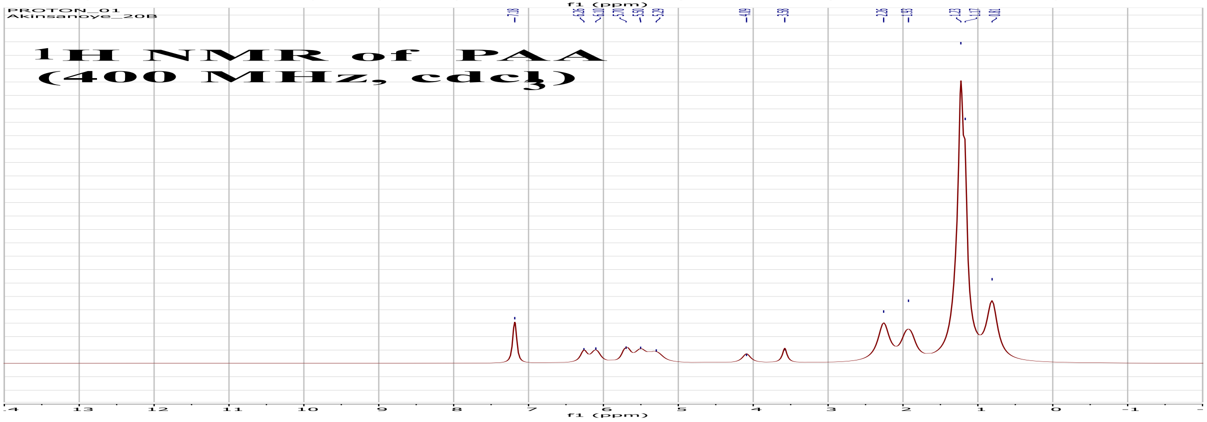
3.3 ^{1}H-NMR Analysis of Polyester-amides and the precursors

Figure 5 is the ^{1}H-NMR spectra showing the various chemical environments in the *Jatropha curcas* oil and its various modifications that led to the production of the polyester-amides. Spectrum 1 showed ^{1}H NMR of *Jatropha curcas* oil(JCO) (400 MHz, cdcl$_3$) with five distinct chemical environments. δppm 5.48 (s, 1H) is an evidence of unsaturated vinyl protons in the triglyceride moiety, δppm 2.46 (s, 1H) and 2.16 (s, 1H) confirms the presence of O=C-O-C-**H** peaks in the triglyceride. δppm 1.42 (s, 5H), 1.02 (s, 1H) are evidences of saturated protons in the oil moiety. These chemical environments confirm the triglyceride group in the oil. They are in tandem with the FTIR result for the sample. Spectrum 2 (^{1}H NMR of PJCB (400 MHz, cdcl$_3$) is for Phenol *Jatropha curcas* biopolymer (PJCB). The spectrum showed 12 different chemical environments. δppm 7.25 (d, *J* = 7.6 Hz, 1H), 7.09 – 6.86 (m, 3H) and 6.53 (s, 1H) are evidences of phenolic protons in the PJCB spectrum. These peaks were absent in the JCO. They serve as evidence that the phenol was grafted to the triglyceride backbone. The FTIR result also confirm this. δppm 5.52 (s, 1H), 5.50 – 5.30 (m, 1H) are evidences of vinyl protons. δ 2.37 (p, *J* = 6.4 Hz, 1H), 2.24 are evidences of O=C-O-C-**H** protons. Peaks at δ 2.08 (m, 1H), 2.11 – 2.03 (m, 1H) and 1.65 (d, *J* = 7.2 Hz, 1H) are evidences of allylic protons. 1.48 (s, 2H), peaks at δ 1.44 – 1.28 (m, 10H), 1.12 – 0.97 (m, 1H) and 0.93 (dd, *J* = 7.0, 2.0 Hz, 1H) are saturated protons. The high number of saturation here is an evidence that some of the unsaturated peaks in the starting triglyceride have been polymerized or hydrogenated during the polymerization process (Figure 5). This is in tandem with the result of the iodine value for the PJCB when compared with the starting material. ^{1}H-NMR of CJCB (400 MHz, cdcl$_3$) showed 10 different chemical environments (Spectrum 3). It also showed distinct features from the starting *Jatropha curcas* oil. δppm 7.06 (s, 2H) and 7.00 (s, 1H) occurs like doublets and serve as evidence of aromatic protons in the intermediate material. These peaks were absent in the starting material. Vinyl protons were found at δppm 5.60 (s, 2H) and 4.52 (s, 1H). δ 4.39 (s, 1H), is an evidence for phenolic protons. Substituted O=C-O-C-**H** presence was established with peaks like 2.56 (s, 4H), 2.47 (s, 4H) and 2.20 (s, 4H) in the spectrum. Similar peaks were found in the seed oil. Peaks at 1.84 (s, 5H) and 1.11 (s, 5H) are evidences of saturation in the CJCB intermediate. The result is also in tandem with the FTIR result for this intermediate material.

^{1}H-NMR of PAA (400 MHz, cdcl$_3$) showed amide protons at δppm 7.18 (s, 2H), 6.26 (s, 1H) and 6.10 (s, These peaks were also present in the FTIR spectrum for this sample. Vinyl protons occurred at δ 5.70 (s, 1H), 5.50 (s, 2H) and 5.29 (s, 1H), Peaks at δ 4.09 (s, 1H) and 3.58 (s, 1H) occurred due to non- substituted O=C-N-**H** groups in the homopolymer. α-carbonyl protons of amide showed up at 2.26 (s, 5H). Allylic protons in the PAA moiety occurred at 1.93 (s, 4H), Peaks at 1.23 (s, 21H), 1.17 (s, 3H) and 0.81 (s, 6H) are due to unsaturation emanating from the homoplymerization process.


PROTON_01.fid.10
Akinsanoye_10B
1H NMR of CJCB-PAA
(400 MHz, cdcl3)
8.69
5.95
5.36
3.11
2.30
1.96
1.31
1.24
0.85
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)


PROTON_01
Akinsanoye_3B
1H NMR of PJCB-PAA
(400 MHz, cdcl3)
7.18
6.86
5.52
5.36
4.15
2.36
2.28
2.02
1.94
1.27
0.89
0.82
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)


PROTON_01
Akinsanoye_20B
1H NMR of PAA
(400 MHz, cdcl3)
7.18
6.26
6.10
5.70
5.50
5.29
4.09
3.58
2.26
1.93
1.23
1.17
0.81
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

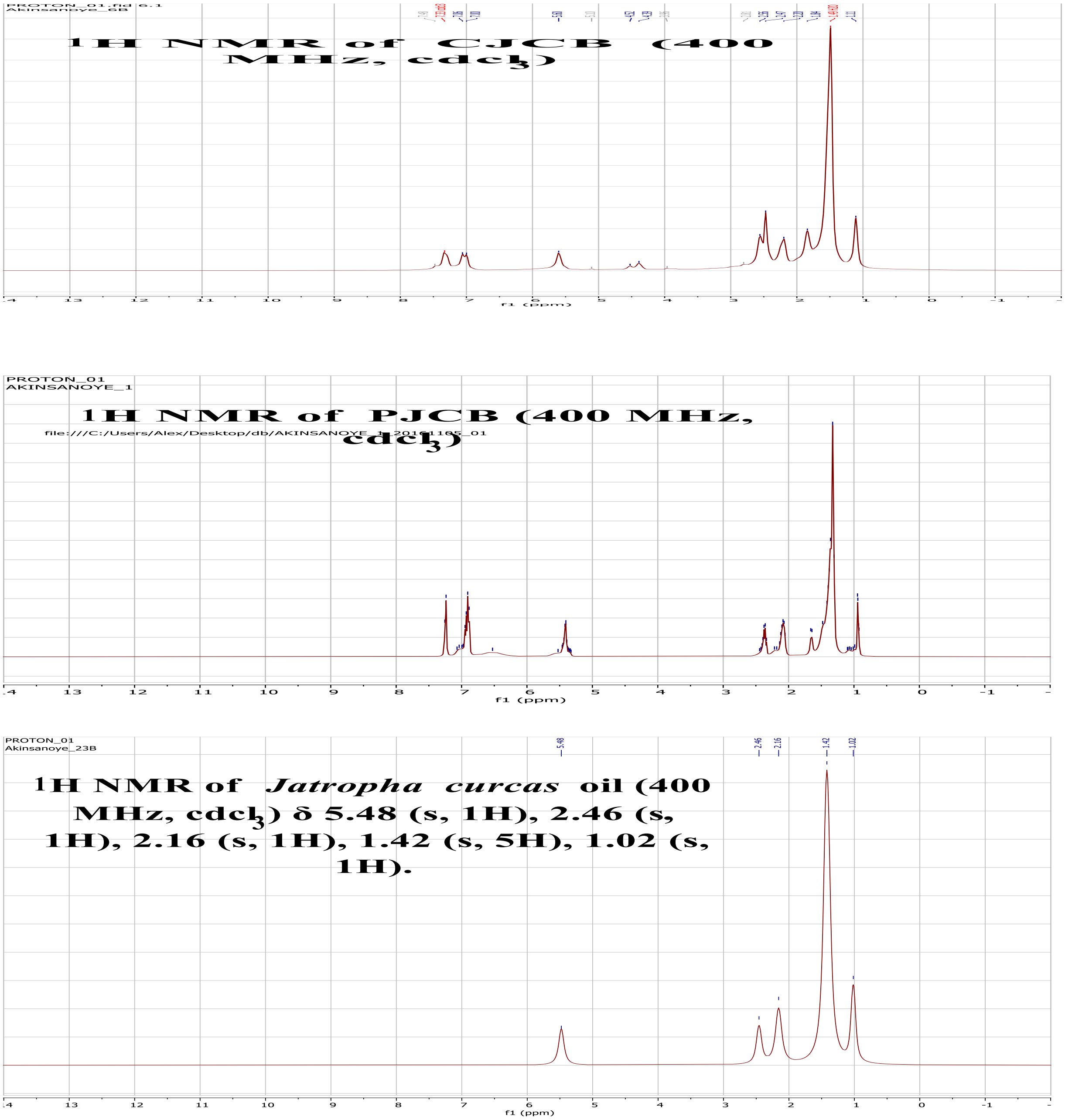


Figure 5: ^{1}H-NMR spectrum of *Jatropha curcas* & the polyester-amides from it

The ^{1}H NMR spectrum of PJCB-PAA (400 MHz, cdcl$_3$) is also presented in Figure 5. The spectrum showed different chemical environments for the new material. Peaks at δ 7.18 (s, 1H) is an evidence for a substituted arene in the new material. Similar peak was also found in the PJCB backbone. Amide or peptide links occurred at 6.86 (s, 3H) and 5.52 (s, 1H) to show that the PAA was grafted to the PJCB in the course of the polymerization process. They are evidences that PJCB-PAA contains substituted and non-substituted amides respectively. This may have occurred on the different triglyceride groups in the oil to have given two distinct chemical environments. They confirmed that the new material is an amide. Substituted vinyl protons were found at 5.36 (s, 1H) to show that the vinyl moiety is still present in the new material. Phenolic proton is confirmed at δ 4.15 (s, 1H) to further establish the presence of phenolic group in the new material. The FTIR result also complement this. O=C-O-C-**H** protons occurred at δ 2.36 (s, 2H) and δ 2.28 (s, 1H) in the triglyceride moiety. These peaks suggests that the condensation process is not total and hence some ester functional groups are still present in the new material. They are therefore classified as polyester-amides (presence of amide and ester groups in some proportion). The fact that they are singlets implies that they have been substituted. Presence of allylic protons were confirmed at 2.02 (s, 2H) and 1.94 (s, 1H). These allylic protons further established that the vinyl groups in the new material are substituted. Peaks at δ 1.27 (s, 23H), δ 0.89 (s, 2H), δ 0.82 (s, 1H) are evidences of substituted saturated protons. This is the first polyesteramide prepared from the *Jatropha curcas* seed oil.

The second material is named CJCB-PAA. It has its spectrum also in Figure 5. The ^{1}H NMR of CJCB-PAA (400 MHz, $cdcl_3$) showed substituted amide peaks δ 8.69 (s, 1H). The peak confirm that the PAA was also grafted to the CJCB backbone in the course of the polymerization process. The peak confirms the material also as an amide. Substituted vinyl protons occurred at 5.36 (s, 1H) as an evidence of substituted vinyl group within the material. It was found that aromatic protons were completely absent here. This may have resulted from the fact that the cresolic group had undergone hydrogenation due to its high reactivity. The increase in elctron density around the cloud of pi bonds in the phenolic groups may have enhance this observation. Also , this may be confirmed with the presence of C-O-H found at δ 3.11 (s, 18H). The high number of singlets here could also be due to some hydrolysis reactions around the amide groups to amine. The presence of C-O-H peak and O=C-NH- peak in the spectrum shows that the compound is also a polyester-amide and not just an amide, hence the name polyester-amide. Substituted α-carbonyl protons occurred at 2.30 (s, 2H) to further establish the ester-amide moiety in the new material. Allylic protons were observed at 1.98 (s, 1H). Substituted Saturated protons were found at 1.31 (s, 7H), 1.24 (s, 3H) and 0.85 (s, 1H) in the new poly-esteramide material.

3.5 Proposed mechanism for cationic polymerisation of phenol/ cresol and *Jatropha curcas* oil

The cationic polymerisation method was followed. The initiator is a super acid which is perchloric acid in water. The reaction follows the rule that govern electrophilic addition reaction. The three stages involved in deducing the mechanism are the initiation., propagation and termination. The H^+ ion from the super acid reacts with the triglyceride backbone to generate the reactive electrophile initiator. The proposed mechanism is presented in Figure 6. The reactive electrophiles react with a second monomer to form a new cation that reacted with a third monomer. As each subsequent monomer adds to the chain, the new positively charged propagating site is at the end of the most recently added unit. The reaction continued for hours. The polymerisation was brought to a halt by introducing a calculated amount of powdered calcium hydroxide to the system after stopping the reaction for a few minutes. The $HClO_4$ will combine with calcium hydroxide to form salt and water. This serves as an easy way of removing the liquid catalyst. The catalyst were then removed as white solid via decantation and filtrations. The mechanistic pathway is as shown in Figure 6.

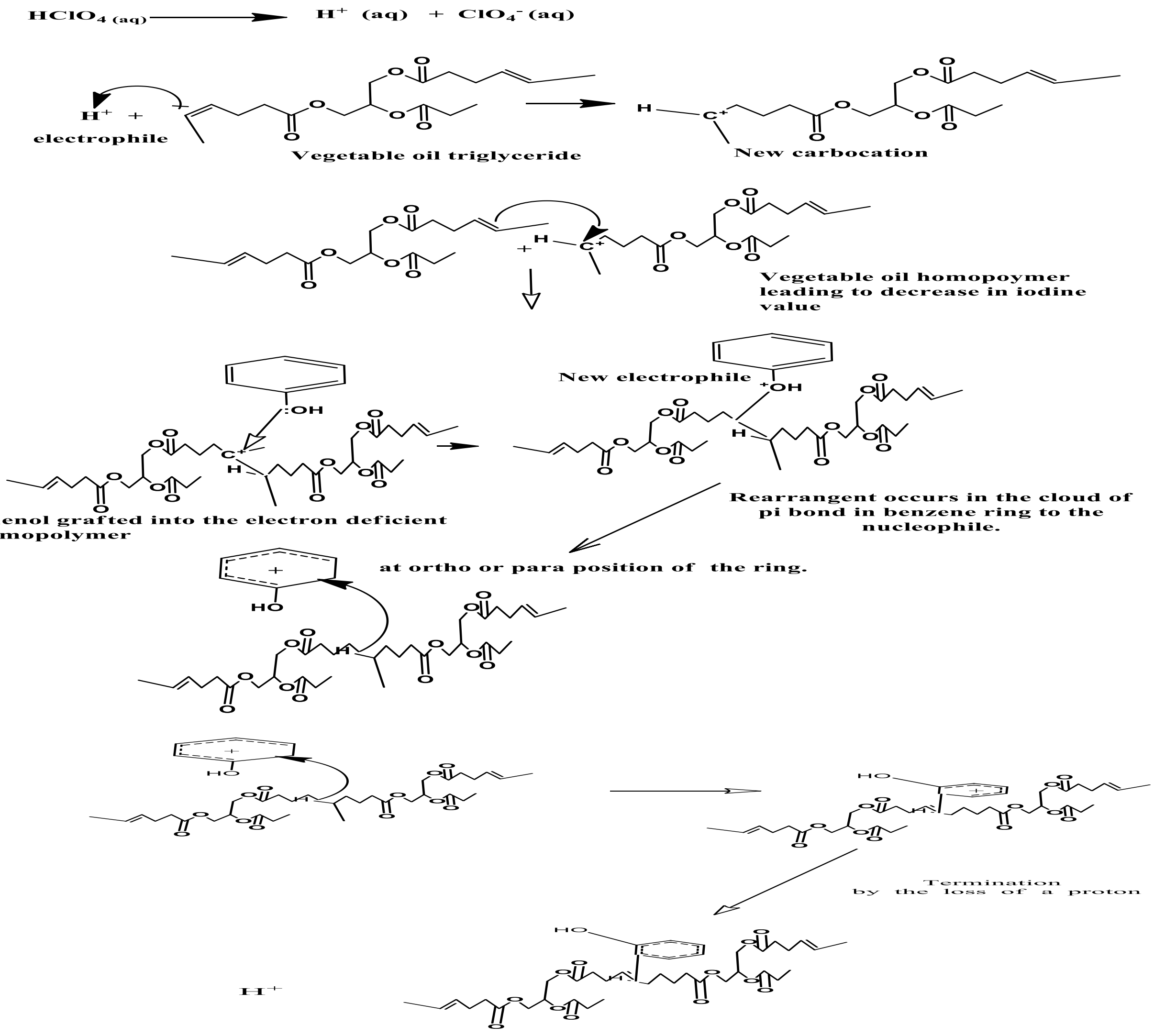


Figure 6: Proposed mechanism for cationic polymerisation of phenol/ cresol and vegetable oil

3.6 Proposed mechanism for polyester-amide formation using Fenton's reagent (Fe (II) – H_2O_2)

Free radical substitution was utilised for the copolymerisation process. The radical polymerisation consists of chain-initiating, chain propagating and chain terminating steps similar to the three steps followed for cationic polymerisation. The catalyst employed here is Fenton's reagent. The reagent created the much needed radicals required for the chain growth polymerisation reaction as described in Figure 7. The hydrogen peroxide react with ferrous ion and thus ferric ion and hydroxyl radicals were created. The hydrogen radical then abstract a hydrogen atom from the monomer to produce a secondary radical and the grafting was initiated from the hydrogen abstracted site on the phenol/cresol vegetable material backbone to create a reactive radical from the Phenol/cresol vegetable backbone. This is known as initiating process. Propagation follows as monomers combine with the secondary radical to generate a growing chain. Several monomers were believed to combine in this way so as to produce long growing chain over the period in which the reaction lasts in slow steps fashion. The reaction is assumed to be terminated by the combination of some of these radicals. Similar observation has been reported[20]. Hydroquinone was added to prevent further polymerisation since it can act as a free radical scavenger. The hydroquinone is also important in prolonging the life of the sensitive copolymers. The mechanistic pathway is presented in Figure 7.

3.7 Proposed kinetics for the free radical polymerisation

Hydrogen peroxide and ceric ammonium nitrate ions in acidic media have been reported to have good oxidizing power for different organic substrates. This they do either by themselves or along with reducing agents. They function as initiators for chain growth polymerisation. The initiator employed for this research is the Fenton's reagent and hydrogen peroxide. The initiator employed for this research is the Fenton's reagent and hydrogen peroxide [7]. Based on the experimental results, the following reaction kinetic scheme was proposed. The first step was the reaction of hydrogen peroxide with a reducing agent. The iron (II) hydroxide-peroxide system (Fenton's reagent) is employed as the redox initiator. The mechanism for the creation of free radical has been proposed [7,18]. The generation of the radical occurs at room temperature. The O-H radical created from Fenton's reagent combine with a monomer to form secondary radical. The reaction is expected to occur very fast. The mechanism involve the initiation, propagation and termination stage.

3.8 Proposed kinetics for the free radical polymerisation

$$\mathrm{Fe^{2+}(q) + H_2O_{2(aq)} \longrightarrow F^{3+}(aq) + {}^{-}OH(aq) + {}^{\cdot}OH + Fe^{3+}(aq).}$$

$$\mathrm{{}^{\cdot}OOH(aq) + H_2O_{2(aq)} \rightarrow {}^{\cdot}OH(aq) + O_2(g) + H_2O\,(l)}$$

$$\mathrm{{}^{\cdot}OH(aq) + M}\ \frac{ko}{fast}\ \ \mathrm{OH\text{-}M^{\cdot}}$$

Initiation

$$\text{OH-M}^{\cdot} + \text{M} \xrightarrow[slow]{K1} \text{OH-M}_{+1}^{\cdot}$$

$$\text{OH-M}_{+1}^{\cdot} + \text{M} \xrightarrow[slow]{k1} \text{OH-M}_{+1} - \text{M}$$

Propagation

$$\text{OH-M}_{+1}^{\cdot} - \text{M} \xrightarrow[slow]{kp} \text{OH-M}_{+2}^{\cdot} \xrightarrow{nM} \text{OH-M}_{n+3}^{\cdot}$$

Termination

$$2\,[\text{OH-M}_{n+3}^{\cdot}] \xrightarrow{Kt} \text{Polymer} + \text{catalyst.}$$

Where M = the Phenol/cresol vegetable oil biopolymers . K_0 is the initiation constant for fast reaction. Ki is the rate constant for the initiation reaction. Kp is the rate constant for propagation phase. K_t is the rate constant for termination phase.

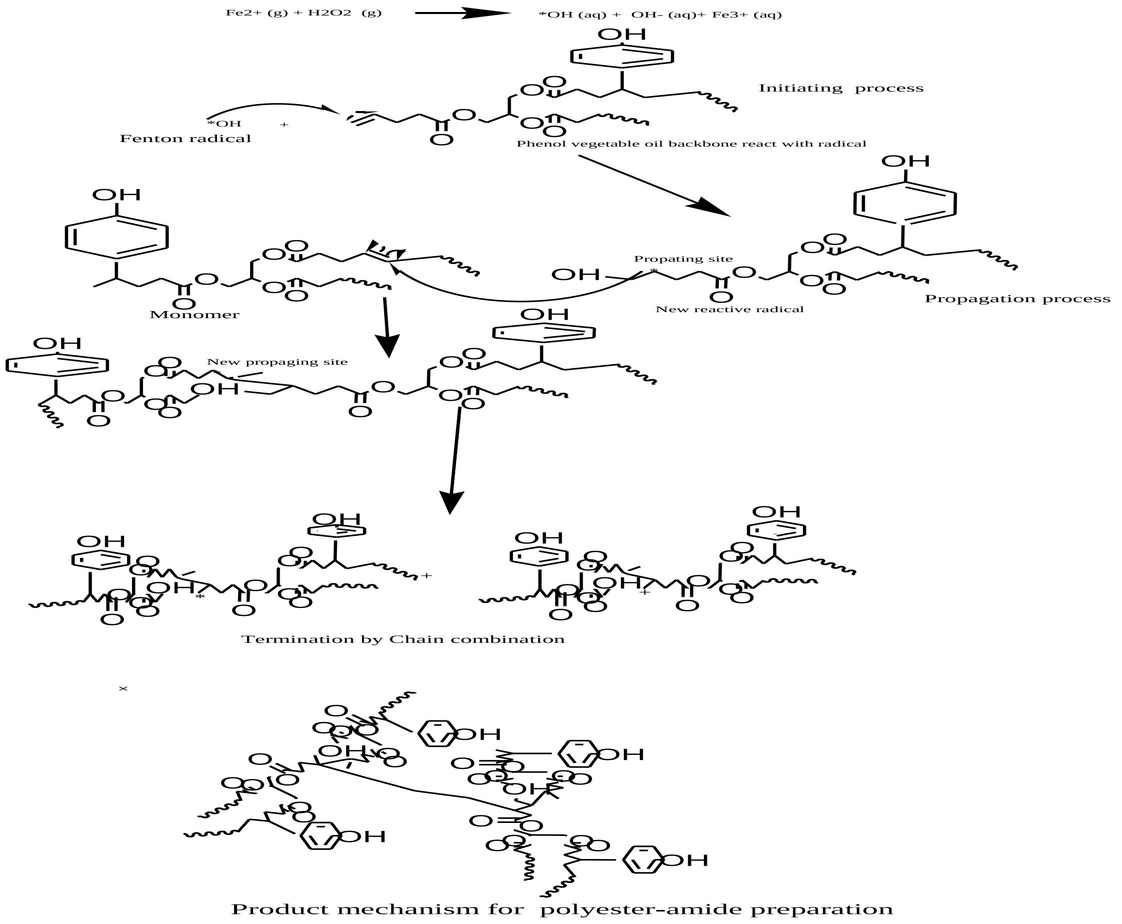


Figure 7: Proposed mechanism for copolymer formation using Fenton's reagent (Fe (II) –H_2O_2).

3.9 Molecular weight determinations of the synthesized polyester-amides

Table 2 is the summary of the calculations for flow rate, concentrations and calculated viscosities of PJCB. The flow rates in seconds increases as the concentration (g/ml) increases. Similar trend was found in the relative viscosity and specific viscosity to suggest linear relationship between these parameters and the flow rates. The inherent relationship however showed an inverse relationship trend with the flow rates. The intrinsic viscosities were obtained graphically, and these were used to calculate the molecular weights from Mark Houwink-Sakurada relation: $\eta = KM^{a.}$ The Table of similar parameters for the polyester-amide is presented in Table 3. It shows a similar trend to Table 2. The mean molecular weight (g/mol) from Tables 2 & 3 for PJCB & PJCB-PAA is presented in Table 6. PJCB has a mean value of 1.041 $\times 10^{9}$ g/mol while PJCB-PAA gave a mean molecular weight of 1.07919 $\times 10^{12}$. The higher value of the polyesteramide is also an evidence that the PAA was grafted to the PJCB backbone.

Table 2 . Flow rate, concentration and calculated viscosities of PJCB

Flow rates (s)	**Concentrations (g/ml)**	**Relative Viscosity (t/t_0)**	**Specific viscosity (η_r -1)**	**Reduced viscosity (η_{sp}/C)**	**Inherent viscosity ($In\eta_r/C$)**
63.0±1.24	-	-	-	-	-
131.2±0.14	2.22×10^{-4}	1.62	0.62	2792.79	2173.09
200.9±0.14	4.44×10^{-4}	2.48	1.48	3333.33	2045.63
306.0±0.07	6.66×10^{-4}	3.78	2.78	4174.17	1996.58
433.0±0.81	8.88×10^{-4}	5.35	4.35	4898.15	1888.62
547.0±0.41	1.11×10^{-3}	6.75	5.75	5180.18	1720.31

n=3

Table 3. Flow rate, concentration and calculated viscosities for **(**PJCB-co-PAA).

Flow rates (s)	**Concentrations (g/ml).**	**Relative Viscosity (t/t_0)**	**Specific viscosity (η_r 1)**	**Reduced viscosity (η_{sp}/C)**	**Inherent viscosity Inη_r/C**
13.0±0.10	-	-	-	-	-
29.0±0.14	4.44 x10^{-4}	2.23	1.23	2770.27	1806. 30
51.0±0.14	8.88x 10^{-4}	3.92	2.92	3284.59	1536.66
101.0±0.13	1.33x 10^{-3}	6.77	5.77	4338.35	1437. 97
133.0±0.11	1.78 x 10^{-3}	9.23	8.23	4623.60	1184.15

n=3

Table 4: Flow rate, concentration and calculated viscosities of CJCB

Flow rates (s)	**Concentrations (g/ml)**	**Relative Viscosity (t/t_0)**	**Specific viscosity (η_r -1)**	**Reduced viscosity (η_{sp}/C)**	**Inherent viscosity Inη_r/C**
63.0±1.24	-	-	-	-	-
139.0±0.81	4.44 x10^{-4}	1.42	0.48	1091.91	882.98
187.1±0.41	8.88 x 10^{-4}	1.99	1.99	1114.86	774.93
255.0±0.24	13.3 x 10^{-4}	2.71	1.71	1285.71	749.59
345.9±0.41	17.8 x 10^{-4}	3.68	3.68	1505.62	731.97
469.1±0.21	11.1 x 10^{-4}	4.99	3.99	1797.30	724.07

n=3

Table 5 . Flow rate, concentration and calculated viscosities for(CJCB-co-PAA)

Flow rates (s)	**Concentrations (g/ml)**	**Relative Viscosity (t/t_0)**	**Specific viscosity (η_r -1)**	**Reduced viscosity (η_{sp}/C)**	**Inherent viscosity In$\eta_{r/}$C**
108.0±0.02	-	-	-	-	-
154.0±0.14	4.44 x10^{-4}	1.43	0. 43	968.46	805.57
215.0±0.14	8.88x 10^{-4}	1.99	0.99	1114.86	774.93
275.0±0.07	13.3x 10^{-4}	2.55	2.55	1165.41	703.83
348.0±0.13	17.8 x 10^{-4}	3.22	2.22	1247.19	656.96

n=3

Table 4: Mean molecular weight of synthesized polyesteramides & Respective backbones

Biopolymers and Copolymer synthesised.	**State of polymer**	**Molecular Weights(g/mol)**
Phenol-*Jatropha curcas* polymer (PJCB)	viscous	1.041 x10^9
Cresol-*Jatropha curcas* polymer (CJCB)	viscous	9.929 x 10^9
CJCB-co-PAA	solid	1.07919 x 10^{12}
PJCB-co-PAA	solid	1.39187 x 10^{12}

Table 4 consists of the values obtained for CJCB. The flow rate of the solvent is the fastest at 63.0 seconds. It also increase with increase in concentration. The other parameters such as relative viscosity, specific viscosity and reduced viscosity also follow similar trend as the concentration. The inherent viscosity similarly showed an inverse trend as observed in Table 2 for PJCB. The Tables of values for CJCB-PAA is also presented in Table 5. The Table shows a similar trend to the CJCB. The mean molecular weight (g/mol.) obtained from the Table 3 is 9.929 x 10^9 g/mol. and the polyester-amide has 1.39187 x 10^{12} g/mol. The difference in value is also an evidence that the grafting occurs. Table 6 also shows that CJCB has slightly higher mean molecular weight than the PJCB. Similar trend was also observed in the in the polyester-amides with CJCB-PAA > PJCB-PAA (Table 6). The slight differences may have been due to the phenolic and cresol groups on aromatic or arene chain.

4.0 Conclusion

Polyester-amides were synthesized from *Jatropha curcas* oil. The work started with the extraction of oil from the *Jatropha curcas* seeds. This was followed by the determination of the physicochemical properties of the oil. The oil was found to be rich in iodine value and saponification value. The oil also has small free fatty acid value to suggest there has been no oxidation. The oils were polymerized with cresol and phenol to obtain resonated and phenolated intermediates. These were termed CJCB and PJCB respectively. Homopolymer of acrylamide was prepared and characterized.

The PJCB/CJCB and polyacrylamide were copolymerized via free radical polymerization process to obtain the corresponding polyester-amides with the reaction mechanism and kinetics defined. The oil, alkylated derivative and polyester-amides were characterized using FTIR to determine the functional groups, ^{1}H-NMR was used to determine the various chemical environments and Viscometry technique was used along with Mark-Hauwink Sakurada relation to obtain the average molecular weights. The new materials showed characteristics features of ester and amide to be termed polyester-amides. The choice of polyester-amides is because of its versatility in applications in the drilling and production activities of oil and gas industries. Producing them locally will therefore mitigate the problems associated with their importation. Sourcing them from materials like Jatropha curcas and likes therefore offers a green alternative that will not only minimize importation/economic issues, it will also combat the problems associated with greenhouse gases since it can both biodegrade and photo-degrade.

Recommendation: Despite the huge success of this work, other methods for amide production may be considered to produce amide from the sample. The shaft from the seed also may be researched as viable amide production material for further stability of the oleo-chemical industries and enhancing zero wastes from the material.

Acknowledgment: The Authors acknowledged the contributions of Mr. Abiodun Usman, Industrial Lab, University of Ibadan for his efforts during this research. We are also grateful to Mr. Silas for his contributions to the ^{1}H-NMR analysis in Central Lab, Ahmadu Bello University, Zaria, Nigeria.

Authors' individual contribution:

Dr. Olakunle Alex Akinsanoye was involved in the experimental design, carrying out the experiment, analysis of results, financing and manuscript design.

Mrs Abimbola Odunola Oginni was involved in the composition, result analysis, financing and proof reading of the manuscript.

Professor Mopelola Abeke Omotoso was involved in the conception of the work, financing and guide throughout the research.

Conflict of interests: The authors declare no known financial or personal conflict of interests that influence the work reported in this manuscript.

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