



Catalytic and Noncatalytic Fast Pyrolysis of Jatropha Seeds: Experimental Measurements and Modeling



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THE main problems facing worldwide especially the developing countries are the shortage and high prices of fossil fuels, therefore, searching for renewable sources of energy is highly recommended. Therefore, the conversion of biomass into alternative fuel has become one of the attracting sources of renewable energy. The bio-energy is now accepted as having the potential to provide the major part of the projected renewable energy in the future. However, there are many conversion processes such as pyrolysis, gasification, combustion, and liquefaction. Among them the thermochemical conversion (pyrolysis) which acquired special attention as it can convert it directly into liquid, solid and gaseous products by thermal decomposition in absence of oxygen. The present work will consider *Jatropha curcas* as the biomass source because *Jatropha* seeds are high energy bearing seeds available in nature. The aim of this work is to examine the fast pyrolysis process of *Jatropha* seeds with and without catalyst. Further, a model has been tested using experimental data measured in this study. The thermal decomposition of the *Jatropha* seed and the kinetics of the fast pyrolysis behavior has been investigated in the two cases using thermogravimetric analysis (TGA) at a heating rate of 20°C/min in air atmosphere which indicated the range of temperature in which rate of decomposition is maximum. The results of TGA and DTG data revealed that iso-conversational free model (KAS) method gave excellent prediction with a good agreement of oil yields. The activation energies of *Jatropha curcas* seeds were obtained in the range of (60 to 65 kJ/mol⁻¹) which was much lower than that of the literature (196 to 222 kJ/mol⁻¹) concluding that the reaction rate was fast as a fast pyrolysis process already used and the correlation coefficient (R²) is 0.9987.

Keywords : Fast pyrolysis, *Jatropha* seeds, Catalyst, Modeling, TGA, Activation energy.

Introduction

The biomass is now being examined as an important alternative fuel all over the world. There are many sources of biomass being considered as a potential standards of fossil fuel and chemical feedstock. It has the potential to supply 10–14% of the world's total energy if utilized duly [1]. Pyrolysis is a valuable economic conversion thermal process for solid wastes into alternative fuels into solid fuel char, liquid bio-oil, and gases [2]. The pyrolysis parameters such as time and heating rate can eliminate the type of the process into slow pyrolysis and fast pyrolysis.

At the Slow Pyrolysis, the heating rate is (less than 10°C/s), the reaction rate has occurred with slow heat transfer rate, and the retention time is relatively long time [3]. Usually, slow pyrolysis is used for charcoal production. Furthermore, the fast pyrolysis has occurred at a high heating rate up to 100°C/s and short retention time less than 2 seconds [2]. The fast pyrolysis process in most cases is produced 45–75wt% of liquid fuel, 15–25 wt.% of solid residues, and 10–20 wt.% of gases, depending on the feedstock and pyrolysis parameters used. In general, pyrolysis products are interested in using as a primary fuel. while, there was a lot of researches [4, 5] interested to study

the effect of pyrolysis's parameters on increasing the yield of pyrolytic liquid fuel which shows similarity as an alternative fossil fuel. since the pyrolytic liquid fuel or bio-oil produced from the pyrolysis process is contained a lot of oxygenated compounds, the Bio-oil fuel properties weren't conformed to the standards of fossil fuels [6]. So, it is recommended to use catalyst to enhance the fast pyrolysis process [7].

The *Jatropha* seed consider as a form of biomass. This plant grows wildly in many areas all over the world and even found on infertile soil. The *Jatropha curcas* is a plant that is usually grown as a source of bio-oil. The seeds are the basic source of oil extraction. Because of its toxicity, they are not utilized by humans. Therefore, the prime goal of the *jatropha* cultivation is extracting *jatropha* oil [8]. Fresh *jatropha* oil extracted from the seeds is low-drying, odourless and colour less oil which turns yellow after aging [9–11]. The fact that *jatropha* oil cannot be used for nutritive purposes without reclamation makes its usage as fuel, a very attractive option as biodiesel. The seeds of the *Jatropha* contain 30–40% oil that can be easily converted to biodiesel by processing (transesterification) and refinement [12,13,14]. Crude oil extracted from seeds contains free fatty acids (FFAs), phospholipids, sterols, water, odorants and other impurities [15] as well as exhibit high viscosity, low volatility and the polyunsaturated character of the vegetable oils [16,17]. Their usage as fuel directly in compression engines is not done because of the high viscosity, contamination by acid and formation of gum due to oxidation and polymerization of FFAs during storage and combustion, deposition of carbon on engines and increase in thickness of lubricating oil [18]. Therefore, vegetable oils are treated so as to acquire characteristics similar to fossil fuels especially viscosity and volatility characteristics [19]. *Jatropha* oil is generally extracted from its seeds by mechanical processes or by using Soxhlet apparatus. According to Barua [20], if we use Soxhlet apparatus with petroleum ether at 40–60°C, the kernels of JC give 53% oil. Operating parameters such as type of solvents, temperature, solvent-to-solid ratio, processing time and particle size were investigated to achieve the maximum oil yield from seeds. The optimum conditions were found to be 8h reaction time, temperature of around 68°C, coarse particle size (0.5–0.75 mm), solvent-

to-solid ratio of 6:1 and hexane as solvent. Hexane was found to be the best solvent for the process as compared to petroleum ether, and the extraction of *jatropha* seeds was observed to be an endothermic process [21]. The maximum oil yield was obtained under Soxhlet extraction method, using hexane as solvent. Use of petroleum ether as solvent results in lesser oil yield as compared with hexane and isopropanol but it gives pure and colourless oil, whereas hexane gives faint yellow colour oil. The acid value of oil has been found to be 2.24 ± 0.01 mg potassium hydroxide (KOH)/g [22]. So, Due to the various usages of pyrolysis products and especially the scope implementation of bio-oil, the recent study is concentrated on the fast pyrolysis of *Jatropha* seeds which is adequate to produce a high proportion of liquid bio-oil product in a short time with maximum oil yield in addition with using CaO Catalyst to decrease FFAs ratio to acquire characteristics similar to fossil fuels in one step (catalytic fast pyrolysis).

Materials and experimental apparatus

The *Jatropha* seed samples used as feedstock are collected. These seeds are crushed into small sizes and kept in the oven for about 5-6 hours at a temperature of 60 -70 °C.

Catalyst has been prepared from eggshells. Eggshells were first washed with water repeatedly to remove impurities and then dried in drying oven at 110 °C for about 24 hours. The dried shells were grounded to fine powder to uniform size of the sifted flour using a sieve of 200 mesh sieve and the result is used as raw material for the synthesis of catalyst. The calcination of Eggshells powder was done in a muffle furnace at 900 °C for about 2 hours.

The thermogravimetric analysis (TGA) of raw material has been done using the DTG 60 instrument. TGA was done at a heating rate of 20°C/min. The sample was heated up to a final temperature of 700 °C.

Proximate analysis (using ASTM), is the determination of moisture, volatile matter, fixed carbon (by difference) and ash by prescribed Methods. The ultimate analysis of the material was carried out in CHNS elemental analyzer (Vario El Cube Germany) to know the elemental composition.

The experimental setup which consists of a cylindrical fixed bed batch reactor with electrically heated furnace, condensation system,

nitrogen gas cylinder, PID controller and biomass feeding and char removing system. The reactor was made of 304 stainless steel with 11 cm internal diameter and 40 cm height. The reactor was externally heated by electrical furnace with 2.5 kW power which insulated to reduce the heat loss. A K-type thermocouple was inserted inside the reactor to measure the pyrolysis temperature. The temperature inside the reactor was maintained constant by using a PID controller. The nitrogen tank was used for the supply of N₂ gas which used to create an inert condition inside the pyrolyzer and sweeping the pyrolysis vapours from the reactor to the condensers. The reactor was connected to three condensers (one stainless steel and two glass condensers) and the condensed liquid was collected in a collecting flask at the end of each condenser and weighted for yield. The flask of the first condenser was immersed into the ice-cooled bath to increase the condensation efficiency. the feeding system consists of feeding hopper, screw feeder, and water-cooling jacket to prevent the decomposition of biomass before the heating zone. After the pyrolysis process, the reactor was cooled and char was removed from the reactor by using the screw removing system and collected and weighed.

Pyrolysis Procedures

Pyrolysis experiment was carried out for Jatropha seed at different temperatures starting from 400°C to 550°C at 50 intervals. The experiment was allowed to continue for at least 30 min after the oil production stopped see Table 1. For each sample run amount of liquid produced and char remained in the reactor were noted. The temperature at which the amount of liquid fuel production became maximum is the optimum temperature.

The liquid products were condensed by using water bath. The temperature is measured by Fig.1 shows the schematic diagram of the pyrolysis experimental set-up. Water is circulated as cooling medium in the condenser. The liquid product collected in the measuring cylinder contains liquid fuel and oily water. This Oily water is basically water with some dissolved nitrogen and oxygen containing hydrocarbons.

TABLE 1. Process Operating Parameters.

Process Parameter Specification	
J. S. Particle Size (mm)	1.5
Temperature range (°C)	400 -550
Catalyst size (mesh)	200

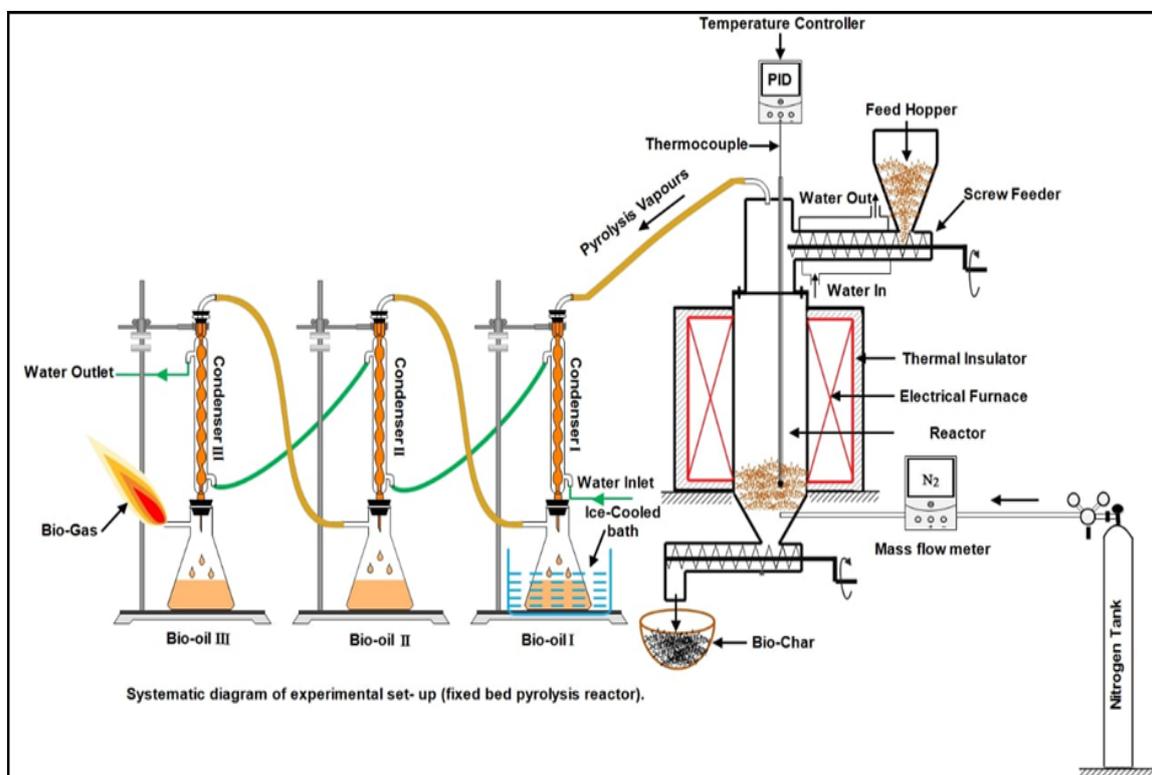
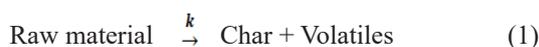


Fig.1. Experimental apparatus of the pyrolysis process.

A Perkin-Elmer infrared spectrometer analyzer was used to determine (FTIR) analysis of pyrolytic oil obtained at a temperature of 500 °C in the range of 400 – 4000 cm⁻¹ with a resolution of 4 cm⁻¹ to know the presence of various functional groups with and without using CaO catalyst. Furthermore, GC-MS-OP 2010 [SHIMADZU] analyzer was used to analyze the pyrolytic oil at 500 °C to find out the chemical compounds present in the pyrolytic oil and to study the effect of using Cao catalyst on the JCSPO.

Kinetic Modeling

The biomass conversion could be denoted in the pyrolysis process as the reaction products of biochar and volatiles. Moreover, the kinetic expression is given by the Arrhenius equation as [23]:



Where k is the rate constant of the reaction following the Arrhenius law, so the rate of pyrolysis decomposition is defined in the following manner.

$$d\alpha/dt = k(T)f(\alpha) \quad (2)$$

$$k(T) = A \exp(-E/RT) \quad (3)$$

Where, f(α) is the reaction model that depends on the reaction mechanism; k(T) is the rate constant; A is the frequency factor; E is the activation energy (KJ/mol); T is the absolute temperature (K); t is the time (min); and R is the universal constant (8.314 KJ mol⁻¹K⁻¹) [24, 25].

$$\text{Heating rate } (\beta) = dT/dt = \text{constant} \quad (4)$$

$$dt = dT/\beta \quad (5)$$

Where β is heating rate (K/min);

Hence, the final kinetic equation in non-isothermal first order TG experiments is:

$$d\alpha/dT = A/\beta \exp(-E/RT) (1 - \alpha) \quad (6)$$

Model fitting methods

The Coats-Redfern is one of the most extensively used approach to obtain the kinetic parameters of carbonaceous materials [26]. The thermal kinetics of a reaction could be estimated by applying thermogravimetric data to determine the mass or heat interchange as a function of temperature and time according to equation (2), then:

$$f(\alpha) = (1 - \alpha)^n \quad (7)$$

where k(T) is the reaction rate constant, α indicates the amount of conversion or the fractional weight loss, and n is the reaction order.

$$\alpha = (m_i - m_o) / (m_i - m_f) \quad (8)$$

Where, m_i, m_t, and m_f are the initial mass, the current mass at time 't' and the final mass of the sample respectively (mg); note that α value is always between 0 and 1.

$$\int_0^\alpha \frac{d\alpha}{f(\alpha)} \int_0^T A \exp(-E/RT) dT/\beta = g(\alpha) \quad (9)$$

Based on approximation analysis, the resulted Coats-Redfern (CR) equation form could be as follow to evaluate the non-isothermal data for computing kinetic parameters as in equation (10):

$$\ln(g(\alpha)/T^2) = \ln(AR/\beta E) - (1 - 2RT/E) - E/RT \quad (10)$$

Where g(α) represents the function of kinetic mechanism in an integral type. The 2RT/E term is negligible because it is less than 1. So, equation (11) is simplified to:

$$\ln g(\alpha)/T^2 = \ln AR/\beta E - E/RT \quad (11)$$

For a certain heating rate, the ln(g(α)/T²) term varies as 1/T with a slope of -E/R linearly, and the intercept of the line corresponds to ln(AR/βE). Thus, pyrolysis kinetic parameters such as activation energy (E) and pre-exponential factor (A) in the prime pyrolysis stage were calculated by the above equations.

Model free methods

Kissinger method

Kissinger [27] was established a free non-isothermal model. This model allows for getting the value of activation energy from a plot against (1000 / β) for a series of experiments at different constant rates (β), where T_m is the temperature peak of the DTG curve. The equation is the following:

$$\ln(\beta/T_m) = \ln(AR/E) - E/RT_m \quad (12)$$

TABLE 2. Kinetic models used in pyrolytic reaction mechanisms.

Model	Mechanism	f(α)	g(α)
1	I. order	(1-α)	[-ln(1-α)]
2	II. order	(1-α) ²	[(1-α)-1-1]
3	III.order	(1-α) ³	[(1-α) ² - 1]/2

The activation energy E can be calculated from the slope of the plot which is equal to $-E/R$.

Model-free Iso-conversional methods

Kissinger-Akahira-Sunose (KAS) method was used the following equation to calculate the activation energy and frequency factor [28].

$$\ln\left(\frac{\beta}{T^2}\right) = \ln\left(\frac{AR}{Eg(\alpha)}\right) - E/RT \quad (13)$$

The activation energy can be given from a plot of versus for a given value of conversion, α , where the slope is equal $-E/R$.

OFW method was developed equation (14) to use it for calculating the Arrhenius parameters [29].

$$\log\left(\frac{\beta}{T}\right) = \log\left[\frac{AE}{Rg(\alpha)} - 2.315 - 0.457 E/RT\right] \quad (14)$$

The plot of $\log \beta$ versus $1/T$, in this case, allows the activation energy to be calculated from the slope ($-0.457 E/RT$) of a straight line.

Results and Discussions

The results of proximate and ultimate analyses of *Jatropha curcas* seed (JCS) sample and its pyrolysis oil. It can be seen from Table 3 that JCS samples are characterized by high volatile matter (85%), which makes it more readily devolatilized than solid fuel. So, the release of less fixed carbon makes them more useful for pyrolysis and gasification. Also, there are small amounts of moisture, ash and higher weight percentage of oxygen followed by carbon and hydrogen with no Sulphur content. The biomasses with high heating values make them very attractive for the source-feeds for clean energy production instead of fossil-based solid fuels see Table 4. High carbon and low oxygen in JCS as compared to coal are favorable for combustion applications, while the higher proportion of carbon (relative to hydrogen and oxygen content) increases the energy content of a fuel because energy contained in carbon-carbon bonds is greater than that of carbon-oxygen and carbon-hydrogen bonds.

TABLE 4. Ultimate analysis of *Jatropha* seed.

Material\composition	% Carbon	% Hydrogen	% Nitrogen	% Sulphur	C/H ratio	C/N ratio
Jatropha seed	56.55	4.23	4.68	Nil	12.33	12.08

Characterization of the catalyst

The morphology of the eggshell waste-derived catalyst was investigated by SEM as shown in Figure 2 (a), (b), (c) and (d). According to the SEM images, the calcined chicken eggshell waste typically comprises irregular shape of particles. In other words, there were various sizes, and shapes of particles. The smaller size of the grains and aggregates could provide higher specific surface areas. The size of the particle should directly respond to the surface area.

XRD spectra of calcined chicken eggshell samples were obtained with Cu radiation ($\lambda=0.154056$ nm) at 30 kV, 30 mA, a scan speed of 2 °/ minute, and a scan range of 5-60°. The decomposition products of chicken eggshell at variation of temperature were identified using XRD powder as shown in Figure 3. The data from Fig. 2 are compared to the XRD pattern of the shell after decomposition at the XRD pattern of CaO from the Joint Committee on Powder Diffraction Standards (JCPDS) file. It can be seen that powder has high crystallinity and sharp spectra There are sharp peaks on the fabric coated CaO, at 2-theta of 32,3401; 37,3924; 53,8966, these peaks obtained from calcined eggshells are similar to calcium oxide data collected by JCPDS [30].

TABLE 3. Proximate analysis of *Jatropha* seed.

Composition of raw material	<i>Jatropha</i> seed
% volatile matter	86.5
% moisture	04.5
% ash content	04
% fixed carbon	05

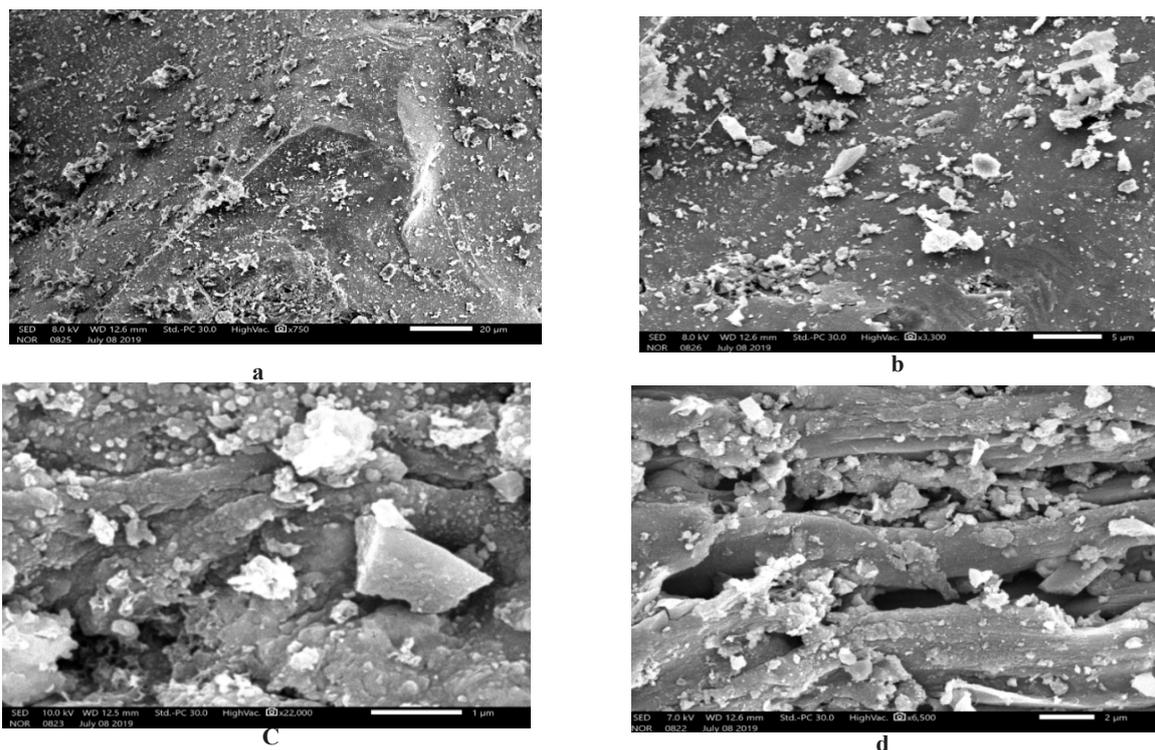


Fig. 2. The SEM image of Calcium Oxide powder surface (a) 750 x Magnification, (b) 3300x Magnification, (c) 6500x Magnification and (d) 22000x Magnification.

Thermo Gravimetric Analysis of Raw Materials (TGA)

Figures 4 and 5 show experimental TG (Thermogravimetric) DTG (Derivative thermogravimetric) curves for thermal decomposition of JCS with and without catalyst under an atmosphere of nitrogen at the heating rate of 293 K min⁻¹. Consistent with, Figs 4-5 the moisture was removed from raw materials up to (306-312) K, and main decompositions of investigated samples started around 307 K and finished around 873 K. the TG-DTG curves (Fig 3-4) show three distinct mass loss stages could be determined ($\Delta T_I = 306-535$ K, $\Delta T_{II} = 535-689$ K, and $\Delta T_{III} > 689$ K, respectively), and it is in agreement with researches related to JCS. The 1st. stage decomposition represents the evaporation of moisture contents, 2nd decomposition indicates the formation of volatiles mainly. In the 3rd stage, the pyrolysis residue slowly decomposes, with the weight loss velocity becoming smaller and smaller and the residue ratio tends to become constant at the end of the hydrocarbon decomposition. Due to the high decomposition rate, the rapid decomposition zone or 2nd stage of decomposition is treated as an active pyrolytic zone. During the 2nd stage, the intermolecular

combination and weaker chemical bonds are broken. The side aliphatic chains are broken and some small gaseous molecules are produced because of the lower temperature. During the 3rd stage, chemical bonds are broken at higher temperature and the parent molecular skeletons are destroyed. As a result, the larger molecule degrades into smaller molecules in the form of a gaseous phase. Finally, char remains [32-37]. In the DTG curves as noticed from Fig. 2, the initial weight loss rates at heating rate 293K⁻¹ were fast, accompanied by a big shoulder peak within 368 to 514.7 °K. The weight loss in this pyrolysis zone made great contributions to the total weight loss (about 74 wt.%) during pyrolysis, Munir et al. [38] suggested that the weight loss occurring near 373 °K represents the initial degradation of lignin and hemicellulose components in biomass. and it was caused by the emission of volatiles from thermal degradation of the three main components in biomass, cellulose, hemicellulose, and lignin. The DTG curves illustrate that the temperature pertinent to the maximum weight loss rate tended to shift to a lower temperature zone. Thus, JCS pyrolysis at a higher heating rate resulted in a more complete decomposition.

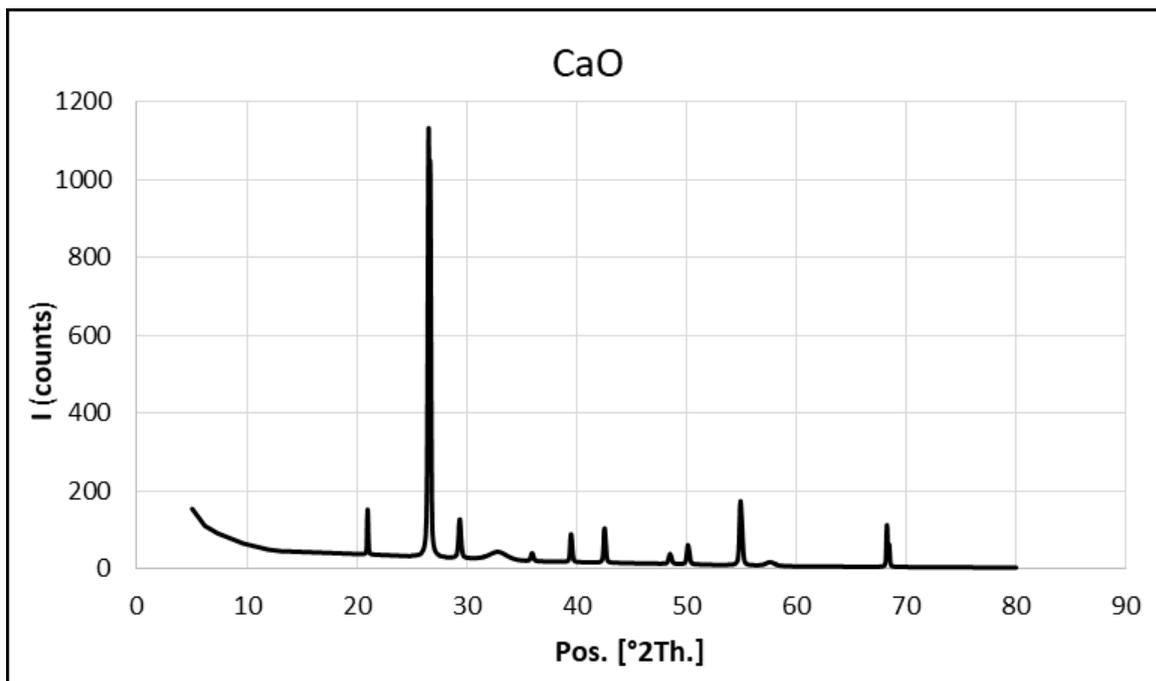


Fig. 3. XRD pattern of Calcium Oxide powder surface.

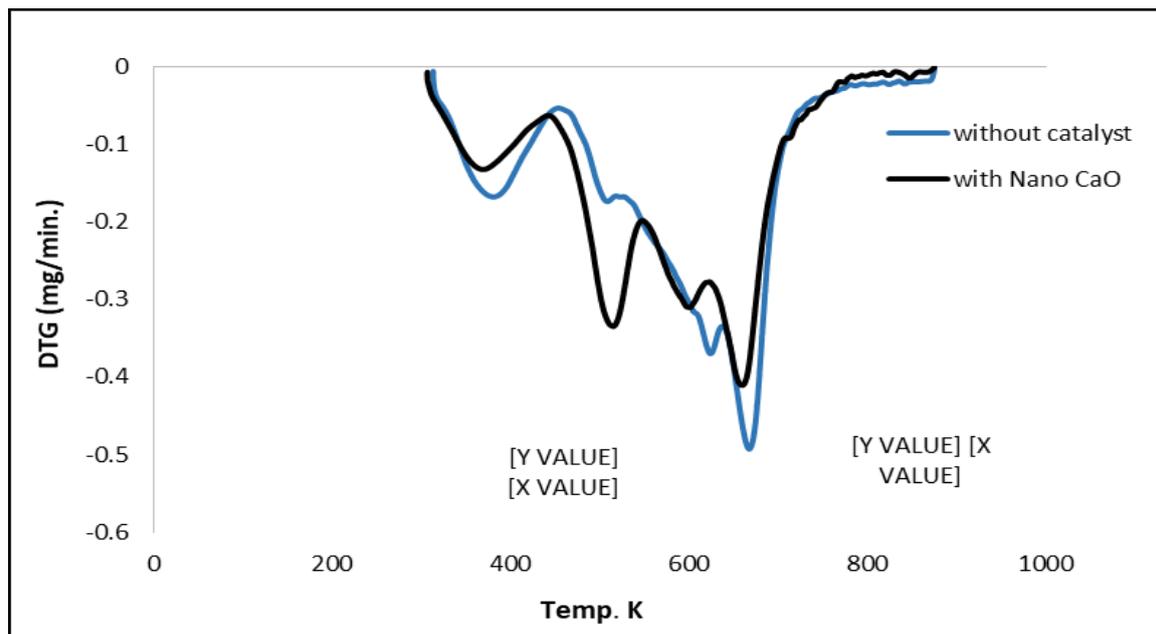


Fig. 4. TGA thermograph of Jatropha seed with and without using catalyst.

TABLE 5. Results of XRD Pattern in experiment, JCPDS (20) and literature.

CaO	2(θ°)				
In this research	32.292	37.604	54.233	64.678	67.861
JCPDS 772376 (1997)	32.2	37.3		64.1	67.3
Literature [30,31]	32.2	37.4	53.9	64.2	67.4

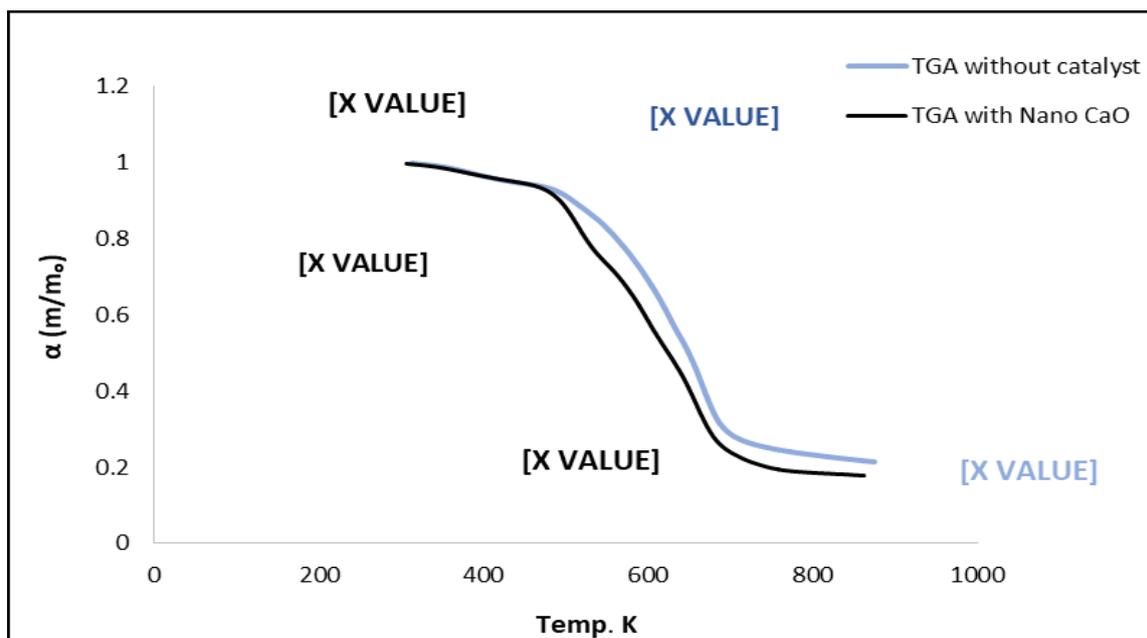


Fig.5. DTG thermograph of Jatropha seed with and without using catalyst.

Characterization of Biofuel

Chns Analysis Of Jatropha Seed Pyrolytic Oil

Ultimate analysis of Jatropha seed pyrolytic oil was done to find the elemental composition of bio-oil and are compared with diesel in Table 6. The CHNO analysis of the pyrolytic oil shows that it contains the maximum amount of C, H and O. Higher the C/H ratio shows the oil having good calorific value, can be observed from the Table 6.

Ftir (Fourier Transform Infrared Spectroscopy)

FTIR is an important analysis technique which detects the presence and the various characteristic of functional groups present in the bio-oil. As demonstrated in Tables 7 and 8. The FTIR analysis performed on samples at optimum temperature 500 °C with and without catalyst. The C-H bending vibrations at 528.28 cm^{-1} detect the presence of alkenes. C-H stretching at 2922.95, 2856.81 cm^{-1} shows the presence of alkanes. C-H bending vibrations at 1452.464, 1377.001 cm^{-1} shows the presence of alkanes. The -NO₂ stretching vibrations at 1513.89 cm^{-1} indicates the presence of nitrogenous compounds. O-H stretching at 3245.317 cm^{-1} shows the presence of H-bonded and water impurities. C=O stretching vibration at 1704.341 indicates the presence of ketones, aldehyde, carboxylic acid as depicted in Figure 6. The results were found consistent with the literature [39] and when compared with GC-MS. Phenol compounds disappear by using CaO catalyst as demonstrated in Table 8.

Gc-Ms (Gas Chromatography Mass Spectrometry)

The major compounds of Jatropha seed pyrolytic oil with and without catalyst characterized by GC-MS at a pyrolytic temperature of 500 °C was given in Tables 9-10. Different types of compounds such as normal alkanes, alkenes, saturated fatty acids and their derivatives such as esters and nitriles were identified. All the compounds were classified with an increase in retention time. Figure 8-9 shows the GC-MS images of Jatropha seed pyrolytic oil with and without using CaO catalyst.

Identified Jatropha non-catalytic oil consists of both saturated and unsaturated fatty acids. Among these, n-Hexadecanoic acid, Octadecanoic acid, 9-Octadecenoic acid and (E)- Pentafluoropropionic acid were found to be in the highest proportions about (24%) of the total compounds while the amount of phenol compounds was (2% wt.). By using Nano CaO catalyst in a portion of 1%wt, the level of phenols was reduced to (1.5%wt.) and the acids eliminated and reduced to (4.5% wt.), while the formation of toluene, cyclopentanones, hydrocarbons, and several light compounds was increased as demonstrated in Table 9. and Table 10. Approximately 60% (on a mass basis) of the diesel fuel compounds have been identified [39]. Among the identified fractions, n-alkanes, branched alkanes, saturated cycloalkanes, alkylbenzenes.

TABLE 6. Ultimate analysis of Jatropha seed pyrolytic oil .

Element	(%.Jatropha seed oil(wt	(%.Diesel(wt
C	60.69	85.72
H	8.67	13.2
N	3.02	0.18
S	Nil	0.3
O	27.62	0.6
H/C	7	1.85
(Calorific value (MJ/Kg	14.63	43.8

TABLE 7. Functional groups present in Jatropha seed pyrolytic oil without CaO catalyst [39].

(Wave number (cm -1	Type of vibration	Nature of functional group
3245.317	(O-H (stretching	H-bonded, water impurities
2922.959	(C-H (stretching	(Alkanes (aliphatic
2856.818	(C-H (stretching	(Alkanes (aliphatic
1704.341	(C=O (stretching	ketones, aldehyde, carboxylic acid
1513.894	(NO ₂ (stretching-	nitrogenous compounds
1452.464	(C-H (bending	Alkanes
1377.001	(C-H (bending	Alkanes
1267.026	C-O (stretching) asymmetric	(carboxylic acid)
1113.728	C-O bending	ester
1023.071	(C-O (stretching	alcohols, ethers, esters, carboxylic acids, phenol
Alkenes		
907.641	(O-H (bending	aromatic compounds
720.019	(C-H group (bending	Methylene groups (cis disubstituted alkenes (and aromatic
528.288	C-H bending	

TABLE 8. Functional groups present in Jatropha seed pyrolytic oil with Nano CaO catalyst [39].

(Wave number (cm -1	Type of vibration	Nature of functional group
3779.185	H ₂ O stretching	water impurities
3255.989	(O-H (stretching	H-bonded, water impurities
2924.768	(C-H (stretching	(Alkanes (aliphatic
2859.034	(C-H (stretching	Alkanes
2347.688	O-H stretching	Carboxylic acids and derivatives
2116.156	(C=O (stretching	alkanes
1642.681	(C=C (stretching	alkenes
1556.137	(NO ₂ (stretching-	nitrogenous compounds
1447.548	C-H bending/ scissoring	Alkanes
1267.265	C-O (stretching)/ asymmetric	Carboxylic acids
1105.793	(C-O (bending	esters
714.412	(C-H group (bending	(Methylene groups (cis disubstituted alkenes and aromatic
623.989	(O-H (bending	aromatic compounds

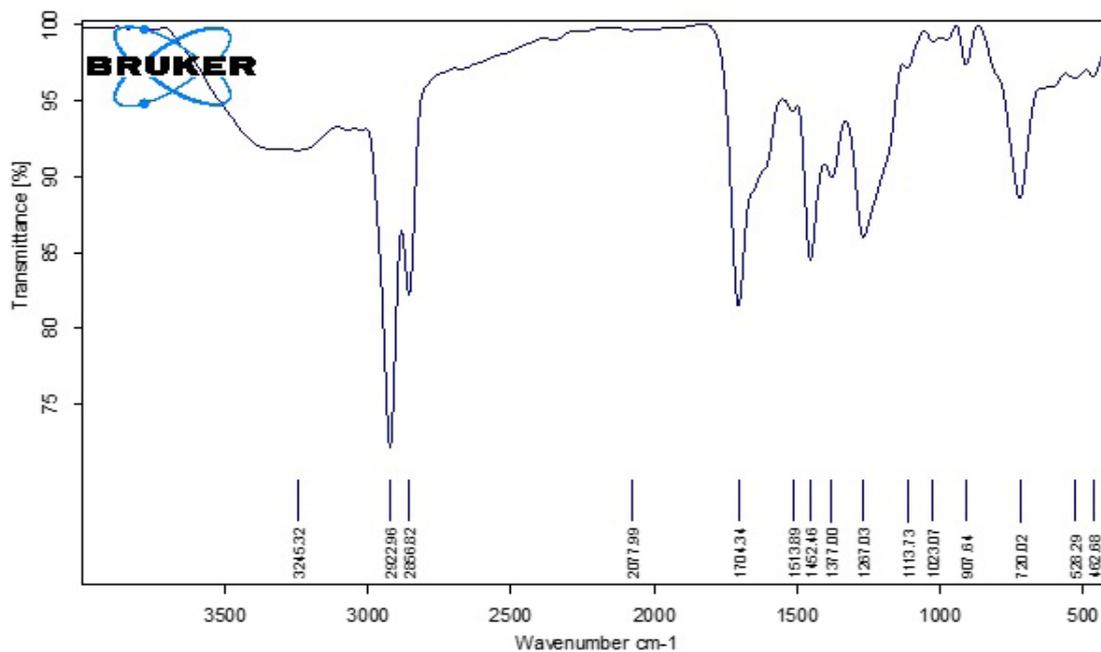


Fig. 6. FTIR spectrum of Jatropha seed bio-oil without using CaO catalyst.

TABLE 9. GC-Mass composition of the major compounds in bio-oils Jatropha seed with catalyst.

R. Time	% Area	Name of compound	Molecular formula
3.8994	9.6732	Toluene	C_7H_8
5.1239	1.371	-Hexane, 2,4-dimethyl	C_8H_{18}
6.3598	3.092	Ethylbenzene	C_8H_{10}
6.6116	1.4479	p-Xylene	C_8H_{10}
7.2868	3.7051	Bicyclo[4.2.0]octa-1,3,5-triene	C_8H_8
7.5557	1.0708	Nonane	C_9H_{20}
10.4739	1.9591	1-Decene	$C_{10}H_{18}$
10.7314	2.0187	Decane	$C_{10}H_{22}$
11.4123	0.5669	Dodecane	$C_{12}H_{26}$
12.305	0.6005	-Naphthalene, decahydro	$C_{12}H_{22}$
12.448	3.8368	-Decane, 2,3,7-trimethyl	$C_{13}H_{28}$
12.6082	0.7555	Sulfurous acid, hexyl octyl ester	$C_{14}H_{30}O_3S$
13.4093	2.1447	1-Decanol	$C_{10}H_{22}O$
13.5695	0.7074	Benzoic acid, methyl ester	$C_8H_8O_2$
13.6439	1.818	Undecane	$C_{11}H_{24}$
13.7068	0.9939	-Decane, 3,7-dimethyl	$C_{12}H_{26}$
14.3477	0.619	-Cyclohexene, 1-pentyl	$C_{11}H_{20}$
15.2003	0.9148	-Benzene, pentyl	$C_{11}H_{16}$
16.07	2.3076	1-Dodecene	$C_{12}H_{24}$
16.276	1.5405	Dodecane	$C_{12}H_{26}$
18.2329	1.9255	Dodecane	$C_{12}H_{26}$
18.5076	0.9563	1-Tridecene	$C_{14}H_{28}$

TABLE 9. Cont.

R. Time	% Area	Name of compound	Molecular formula
18.6907	0.5122	Tridecane	C ₁₃ H ₂₈
19.2972	0.625	-Tridecane, 1-iodo	
20.7677	1.6625	-(2-Tetradecene, (E	C ₁₄ H ₂₈
20.9337	0.8769	Tetradecane	C ₁₄ H ₃₀
22.8849	1.3892	1-Pentadecene	C ₁₅ H ₃₀
22.965	1.265	-Tridecane, 1-iodo	C ₁₃ H ₂₇
23.0394	1.8744	Pentadecane	C ₁₅ H ₃₀
23.8748	0.7085	-Tridecane, 1-iodo	C ₁₃ H ₂₇
24.8818	1.4153	1-Tridecene	C ₁₄ H ₂₈
25.0192	0.9647	Hexadecane	C ₁₆ H ₃₄
26.4954	1.5408	8-Heptadecene	C ₁₇ H ₃₄
26.6042	1.7616	8-Heptadecene	C ₁₇ H ₃₄
26.7701	0.9565	1-Heptadecene	C ₁₇ H ₃₄
26.9017	1.7778	Heptadecane	C ₁₇ H ₃₆
27.1077	1.3435	Heneicosane	C ₂₁ H ₄₄
27.8859	0.621	Heneicosane	C ₂₁ H ₄₄
28.5668	0.8421	1-Octadecene	C ₁₈ H ₃₆
28.687	0.6565	Octadecane	C ₁₈ H ₃₈
30.2777	0.5346	1-Heptadecene	C ₁₇ H ₃₄
30.3864	0.625	Nonadecane	C ₁₉ H ₄₀
30.455	2.5402	Hexadecanenitrile	C ₁₆ H ₃₁ N
30.8098	0.8253	Octacosane	C ₂₈ H ₅₈
30.8441	1.3848	Hexadecanoic acid, methyl ester	C ₁₇ H ₃₄ O ₂
31.485	1.7717	Pentacosane	C ₂₅ H ₅₂
31.9084	0.7195	Pentafluoropropionic acid, tetradecyl ester	C ₁₇ H ₂₉ F ₅ O ₂
32.0057	0.6364	Eicosane	C ₂₀ H ₄₂
33.3389	2.1512	Oleanitrile	C ₁₈ H ₃₃ N
33.4019	2.2416	Oleanitrile	C ₁₈ H ₃₃ N
33.4648	1.0301	1-Nonadecene	C ₁₉ H ₃₈
33.6136	0.9265	trans-13-Octadecenoic acid, methyl ester	C ₁₉ H ₃₆ O ₂
33.7051	3.2845	Heptadecane nitrile	C ₁₇ H ₃₃ N
34.1514	0.506	Pentadecane	C ₁₅ H ₃₂
34.9639	0.4247	1-Docosene	C ₂₂ H ₄₄
35.4847	1.9255	Retene	C ₁₈ H ₁₈
37.7677	0.4239	1-Nonadecene	C ₁₉ H ₃₈
38.2312	1.5484	Phenol, 2,2'-methylenebis[6-(1,1-dimethylethyl)-4-methyl	C ₂₃ H ₃₂ O ₂

TABLE 10. GC-Mass composition of the major compounds in bio-oils *Jatropha* seed without CaO catalyst.

R. Time	% Area	Name of compound	Molecular formula
10.7313	1.4707	Decane	C ₁₀ H ₂₂
12.3049	0.5499	-Naphthalene, decahydro	C ₁₂ H ₂₂
12.4479	3.3092	-Decane, 4-ethyl	C ₁₂ H ₂₅
12.6024	0.786	-Decane, 4-ethyl	C ₁₂ H ₂₅
13.6381	1.4026	Undecane	C ₁₁ H ₂₄
13.7067	1.0855	-Undecane, 4,7-dimethyl	C ₁₃ H ₂₈
15.2002	1.3346	-Benzene, pentyl	C ₁₁ H ₁₆
15.6579	0.9901	-Phenol, 4-ethyl	C ₈ H ₁₀ O
15.8983	0.7953	-1H-Indene, 1-methylene	C ₁₀ H ₈
16.0699	0.5972	1-Dodecene	C ₁₂ H ₂₄
16.2759	1.1232	Dodecane	C ₁₂ H ₂₆
18.2328	1.5482	Tetradecane	C ₁₄ H ₃₀
18.5018	0.6245	1-Tridecene	C ₁₃ H ₂₆
18.6906	0.4657	Tridecane	C ₁₃ H ₂₈
19.0224	0.643	-Naphthalene, 1-methyl	C ₁₁ H ₁₀
19.2971	0.5671	-Heptadecane, 2,6,10,15-tetramethyl	C ₂₁ H ₄₄
19.9208	0.4029	Triacetin	C ₉ H ₁₄ O ₆
20.7619	1.5538	-(2-Tetradecene, (E	C ₁₄ H ₂₈
20.9336	1.0311	Tetradecane	C ₁₄ H ₃₀
22.879	1.276	1-Pentadecene	C ₁₅ H ₃₀
22.9649	0.988	Octadecane	C ₁₈ H ₃₈
23.0336	2.182	Pentadecane	C ₁₅ H ₃₂
24.5041	0.789	Cyclododecene	C ₁₂ H ₂₄
24.5956	0.8735	1,13-Tetradecadiene	C ₁₄ H ₂₆
24.876	1.4327	1-Nonadecene	C ₁₉ H ₃₈
25.0191	1.067	Hexadecane	C ₁₆ H ₃₄
26.4953	2.3098	8-Heptadecene	C ₁₇ H ₃₄
26.6041	2.4723	-(3-Heptadecene, (Z	C ₁₇ H ₃₄
26.77	0.747	1-Heptadecene	C ₁₇ H ₃₄
26.9016	2.1115	Heptadecane	C ₁₇ H ₃₆
27.1076	1.2196	Hentriacontane	C ₃₁ H ₆₄
28.5667	0.6791	-(5-Octadecene, (E	C ₁₈ H ₃₆
28.6811	0.5977	Octadecane	C ₁₈ H ₃₈
30.3805	0.4618	Nonadecane	C ₁₉ H ₄₀
30.455	4.4447	Pentadecanenitrile	C ₁₅ H ₂₉ N
30.8383	2.8924	Hexadecanoic acid, methyl ester	C ₁₇ H ₃₄ O ₂
31.4792	6.2059	n-Hexadecanoic acid	C ₁₆ H ₃₁ O ₂
32.0056	0.5491	Eicosane	C ₂₀ H ₄₂

TABLE 10. Cont.

R. Time	% Area	Name of compound	Molecular formula
33.3388	4.5592	Oleanitrile	$C_{18}H_{33}N$
33.4018	4.9365	Oleanitrile	$C_{18}H_{33}N$
33.6135	2.154	7-Octadecenoic acid, methyl ester	$C_{19}H_{36}O_2$
33.705	5.3974	Heptadecanenitrile	$C_{17}H_{33}N$
33.9969	1.0477	Methyl stearate	$C_{19}H_{38}O_2$
34.1513	1.6609	Dodecane	$C_{12}H_{26}$
34.2315	4.6969	-(9-Octadecenoic acid, (E	$C_{20}H_{38}O_2$
34.3058	4.284	-(9-Octadecenoic acid, (E	$C_{20}H_{38}O_2$
34.5519	2.0914	Octadecanoic acid	$C_{18}H_{36}O_2$
35.4845	0.8868	Retene	$C_{18}H_{18}$
35.8164	0.5276	8-Heptadecene	$C_{17}H_{34}$
38.2311	1.0214	Phenol, 2,2'-methylenebis[6-(1,1-dimethylethyl)-4-methyl	$C_{23}H_{32}O_2$
39.8561	0.4899	Diisooctyl phthalate	$C_{24}H_{38}O_4$

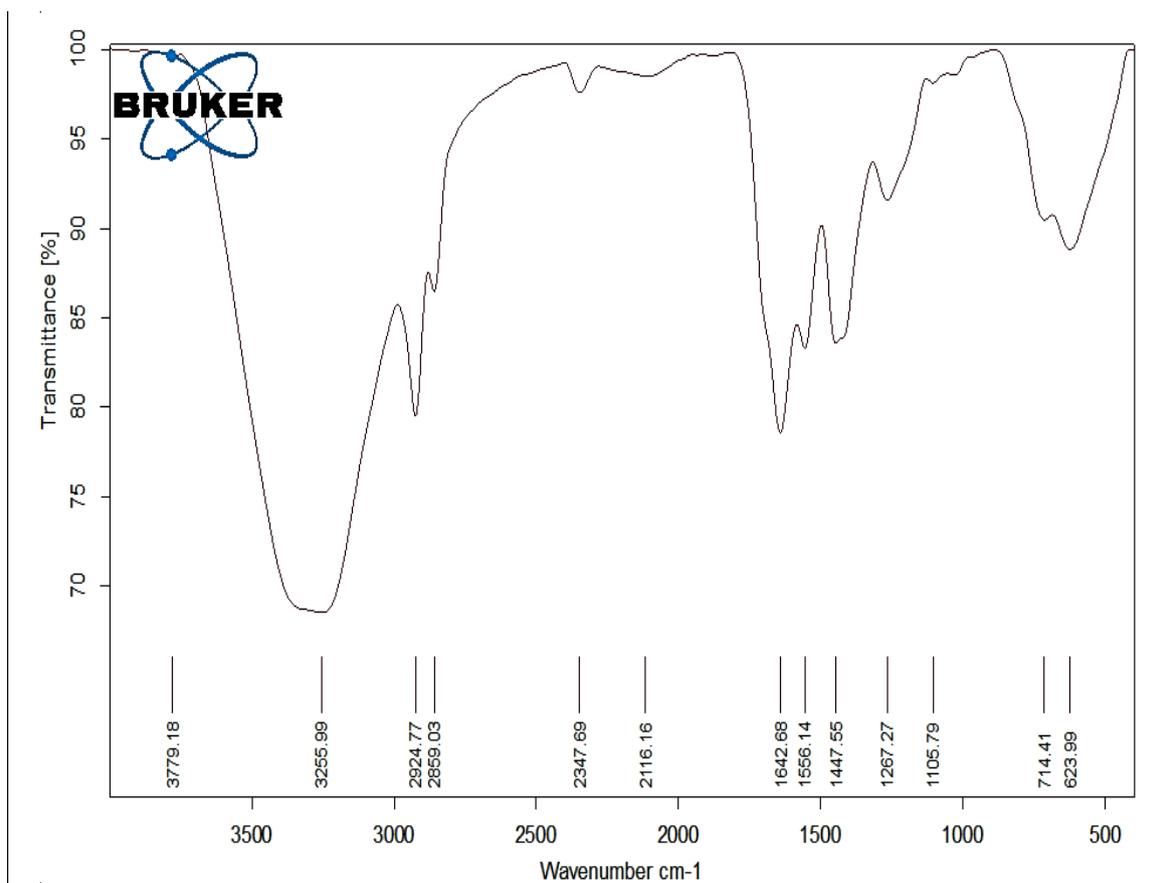


Fig. 7. FTIR spectrum of Jatropha seed bio-oil with using Nano CaO catalyst.

Jatropha charcoal (solid pyrolytic residues)

Ultimate and proximate analysis of *Jatropha* seed pyrolytic coal was done to find the elemental composition of bio-coal and compared it with conventional coal in Table 11.

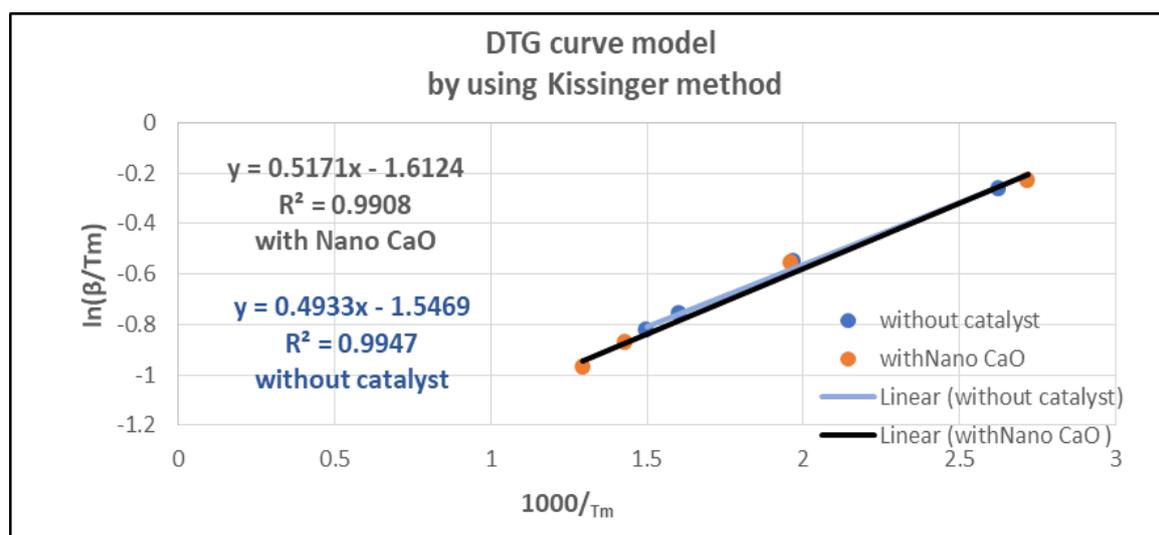
Pyrolytic kinetic models' analysis for JCSCO

The results obtained from thermogravimetric analysis were elaborated according to model-free and model fitting methods to calculate the kinetic parameters. The activation energy (E_a) and pre-exponential factor (A) were obtained using Kissinger, FWO, KAS, and Coats-Redfern methods. In the first method, the E_a and A were calculated from Eq. (12), where T_m is temperature

which corresponds to the maximum weight loss peaks. The peak temperatures were obtained from Fig. 10 Kissinger plot of $\ln \beta$ versus $1000/T \text{ K}^{-1}$ of fast pyrolysis process for *Jatropha curcas* seeds shown in Figure 10. The regression equations and the square of the correlation coefficient (R^2) is also presented. The activation energy (E_a) and pre-exponential factor (A) were derived from the slope and intercept of plotting regression line, respectively. The results obtained from the Kissinger method are 6.7 and 7.2 kJ mol⁻¹ and 1.58E-04 and 2.85E-01 min⁻¹ for activation energy and pre-exponential factor, for catalytic and non-catalytic process respectively.

TABLE 11. Ultimate and proximate analysis of *Jatropha* seed pyrolytic oil.

Element	<i>Jatropha</i> seed char coal (wt.%)	Conventional coal (bituminous) (wt.%) [40]
Ultimate analysis		
C	66.85	65-80
H	4.76	4.5-6
N	3.25	0.5-2.5
S	Nil	0.5-6
O	25.14	-10 4.5
H/C molar ratio	14.04	13.3-14.4
Proximate analysis		
% Moisture content	2.2	2-15
% Volatile matter	80.9	15-45
Fixed carbon	8.1	50-70
% Ash	6	4-15
GCV, MJ/Kg	27.59	26.45


Fig. 10. Kissinger plot of JCSCO.

The kinetic parameters obtained by CR and KAS methods were calculated according to Eq. (11) and (13), respectively, for given values of conversion, α . Figures 11(a) shows the change of the conversion with temperature of the JCISO samples at any moment at a certain heating rate $\beta=20^\circ\text{C}/\text{min}$. To determine the kinetic parameters. The CR plot versus $1/T \text{ K}^{-1}$ for different values of conversion are shown in Figures 11(a). The KAS plots of verses $1000/T$ for a given α of different values of conversion are shown in Fig. 11(b). The apparent activation energies were obtained from the slope and pre-exponential factors from the intercept of regression lines are given in Table 12. The calculated squares of the correlation coefficients, R^2 , correspond to linear fittings in Figures. 11(a) and 11(b), were higher for all cases and were from 0.9622 to 0.9987.

The model-free iso-conversational methods (KAS) allow to estimate activation energy as a function of conversion without previous assumption on the reaction model and allows nearly unmistakably detecting multi-step kinetics as a reliance of activation energy on conversion in comparing with Kissinger method which produces a single value of the E_a for the whole process [41]. With (KAS) method the TG with and without catalyst give the same R^2 . Regarding to FWO equation (14), it can't suit our TG analysis because it plots of $\log \beta$ versus $1/T$ so it depends on varies heating rate (β) in our case heating rate is constant. The heating rate has a significant influence on activation energy in the decomposition process. Where, the smaller activation energy shows that the reaction rates are faster [42,43]. Also, the activation energies increase by increasing the extent of conversion [44].

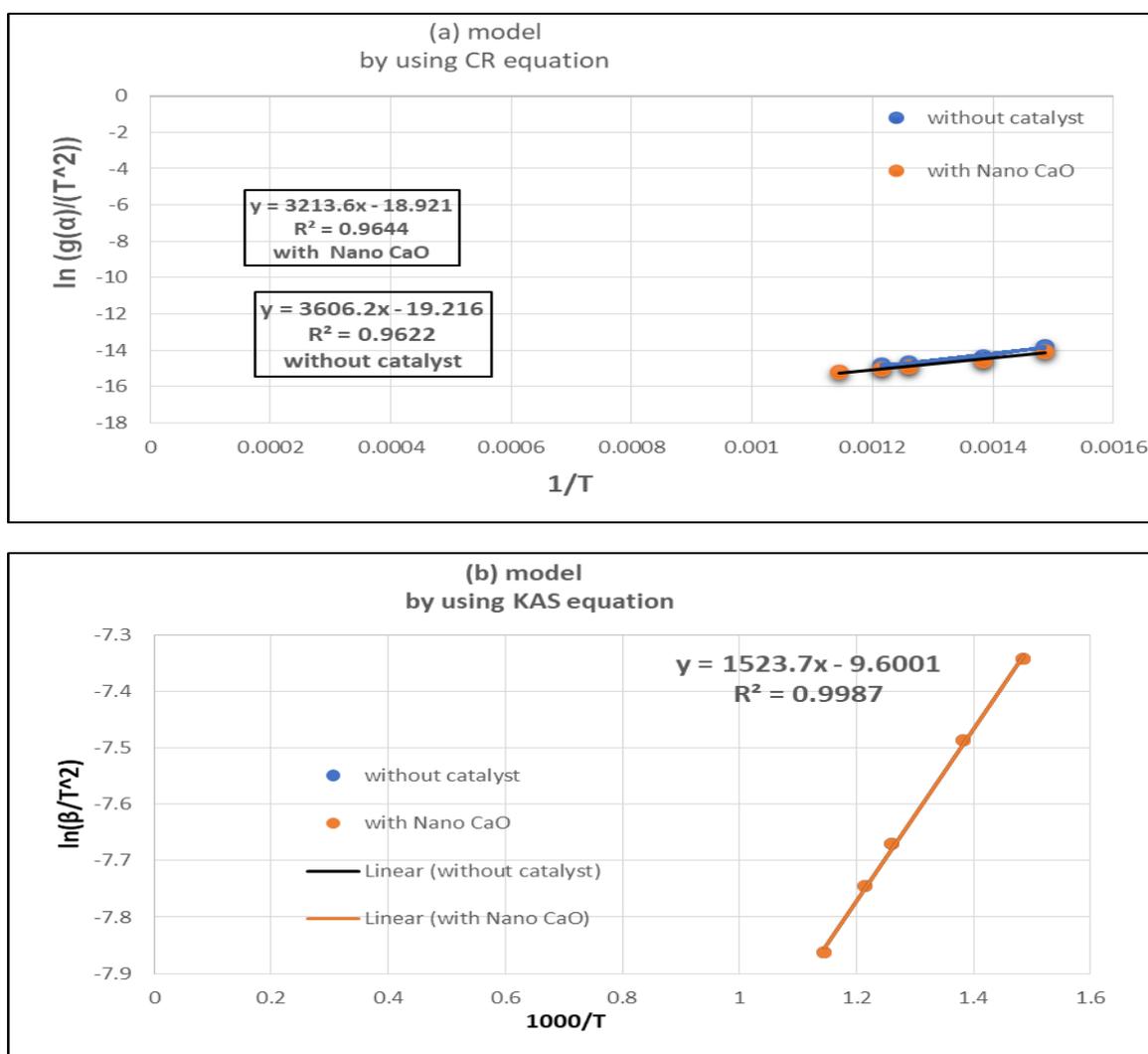


Fig. 11. (a) CR; Figure 11. (a) CR; (b) KAS plot of rice straw for different values of conversion. (b) KAS plot of rice straw for different values of conversion.

TABLE 12. The values of activation energies and pre-exponential factors of JCSO

(α) at $\beta=20^{\circ}\text{C min}^{-1}$	Kissinger $R^2 = (0.9908-0.9947)$		(KAS) $R^2 = 0.9987$		Literature [41-44]	
	Ea (kJ mol ⁻¹)	A (min) ⁻¹	Ea (kJ mol ⁻¹)	A (min) ⁻¹	Ea (kJ mol ⁻¹)	A (min) ⁻¹
0.17751			14.43	0.5934	196.81	$10^{15} \cdot 2.13$
0.21530			17.44	1.2994	200.62	$1.88 \cdot 10^{15}$
0.24115	7.25	2.85E-01			194.52	$2.21 \cdot 10^{14}$
0.30061			18.74	2.377	191.21	$10^{13} \cdot 5.85$
0.84937			4.97	4.97E-01	221.72	$4.07 \cdot 10^{14}$

Conclusions

In this study, Jatropha seed was taken as a biomass sample and the fast pyrolysis experiments was done in a fixed bed reactor under different temperature ranges of 400 – 550 °C without and with using Nano CaO catalyst derived from eggshell to obtain maximum yield of bio-oil and to compare the results and the composition of bio-oil after using the CaO catalyst. Since our aim was to maximize and enhance the produced bio-oil content at the optimum pyrolysis temperature of 500°C and to investigate a kinetic model suitable for our fast pyrolysis process of jatropha seeds.

The maximum bio-oil yield was 50% at an optimum temperature of 500°C for Jatropha seed and by using catalyst with a concentration rate of 1%wt. it reached 65%. It is very significant from the process that as the pyrolysis temperature increased, the amount of liquid product also increased up to a certain value and then it decreased because of the formation of excessive gaseous product.

The eggshell is able to be a source of CaO as catalyst in production of biodiesel from jatropha seeds by pyrolysis hence, the SEM, EDX and XRD analysis of the catalyst showed a good agreement with the literature.

FTIR analysis and GC-MS analysis showed that the bio-oils contain about 60 chemical compounds large amount of fatty acids, oxygen and nitrogen-bearing groups (carboxylic and amines). Otherwise, by using CaO Nano catalyst the level of phenols was reduced and the acids eliminated, while the formation of

cyclopentanones, hydrocarbons and several light compounds was increased.

By applying free iso-conversational methods the (KAS) equation on the experimental data of the recent study, it has given excellent prediction with a good agreement of oil yields were obtained, and The activation energies of Jatropha curcas were found in the range of (60 to 65 kJ/mol⁻¹) which was much lower than that of the literature (generally 120 to 330 kJ·mol⁻¹), indicating that the reaction rate was fast as a fast pyrolysis process already used with a correlation coefficient (R^2) = 0.9987.

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الانحلال الحراري السريع المحفز وغير المحفز لبذور الجatroفا: قياسات معملية ونمذجة

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تتمثل المشكلات الرئيسية التي تواجه العالم وخاصة البلدان النامية في النقص وارتفاع أسعار الوقود الأحفوري ، لذلك يوصى بشدة بالبحث عن مصادر الطاقة المتجددة. ولذلك ، أصبح تحويل الكتلة الحيوية إلى وقود بديل أحد مصادر جذب الطاقة المتجددة. ويتم الآن قبول الطاقة الحيوية باعتبارها لديها القدرة على توفير الجزء الأكبر من الطاقة المتجددة المتوقعة في المستقبل. وهناك العديد من عمليات التحويل مثل الانحلال الحراري في معزل عن الهواء ، والتغويز ، والاحتراق ، والتسييل. من بينها التحويل الكيميائي الحراري (الانحلال الحراري) الذي اكتسب اهتمامًا خاصًا لأنه يمكن تحويله مباشرة إلى منتجات سائلة وصلبة وغازية من خلال التحلل الحراري في غياب الأكسجين. سوف ينظر العمل الحالي إلى نبات الجatroفا كمصدر للكتلة الحيوية لأن بذور الجatroفا هي بذور عالية الطاقة في الطبيعة. ولذلك فإن الهدف من هذا العمل هو فحص عملية الانحلال الحراري السريع لبذور الجatroفا مع وبدون محفز. علاوة على ذلك ، تم اختبار نموذج باستخدام القياسات المعملية المقاسة في هذه الدراسة. تم العمل على التحلل الحراري لبذور الجatroفا ودراسة النقص في الكتلة مع الانحلال الحراري السريع في الحالتين باستخدام التحليل الحراري (TGA) بمعدل حراري قدره ٢٠ درجة مئوية / دقيقة في جو خامل مما يشير إلى مدى درجة الحرارة عند التحلل هو الحد الأقصى. كشفت نتائج بيانات TGA و DTG أن طريقة النموذج الحر للمحادثة (KAS) أعطت تنبؤات ممتازة مع توافق جيد مع الدراسات السابقة. تم الحصول على الطاقة النشطة لبذور نبات الجatroفا في حدود (٦٠ إلى ٦٥ كيلو جول / مول^{-١}) التي كانت أقل بكثير من الدراسات السابقة (١٩٦ إلى ٢٢٢ كيلو جول / مول^{-١}) وانتهت إلى أن معدل التفاعل كان سريعًا لعملية الانحلال الحراري المستخدمة بالفعل ومعامل الارتباط (R^2) هو ٠,٩٩٨٧.