# Thermogravimetric and Decomposition analysis of Jatropha, Castor and Pongamia Deoiled seed cakes

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Abstract – The utilization of biomass resources plays a significant role in minimizing the gap in global energy demand and supply due to the emerging quest for sustainable and environmentally benign alternatives to conventional fossil fuels. Several studies employed thermo gravimetric analysis (TGA) as a kinetic study on thermal degradation of biomass materials as it could shed light on the effect of process parameters on the feedstock conversion method. In this work, we have investigated the pyrolysis properties of the three non-edible seed cake residues such as *Jatropha curcas* (Jatropha), *Pongamia pinnata* (Pongamia) and *Ricinus communis* (Castor) using TGA analysis and the activation energy were found to be 108.73kJ/mol, 94.74kJ/mol and 126.91kJ/mol respectively. The biochemical characterization and comprehension of the activation energy of the non-edible seed cakes could assist in proposing blending of these residues in different ratios with higher activation energy materials for efficient pyrolysis.

Keywords - Biomass, Deoiled seed cakes, Bio fuels, TGA, Reaction Kinetics, Activation energy

# I. INTRODUCTION

Energy is a vital key element for sustainable development and attaining millennium development goals as it has an influencing role on all aspects of social, economic and environmental development. In spite of the increasing trend in overall energy demand, the level of per capita energy consumption in developing countries is far below than the developed countries as well as to the world average consumption rate [1]. The population explosion and subsequent utilisation of finite resources in the world have lead to an exponential rise in global energy consumption in the recent decades. This critical situation warrants the quest of sustainable and eco-friendly benign alternatives for conventional fossil fuels. Though solar, the wind, geothermal and hydro based technologies are available as alternatives to fossil fuels in case of power and heat generation, biomass-based technologies are the unique source of renewable carbon as they are highly appropriate for transportation fuels. The eco-friendly properties such as low sulphur and nitrogen content promote biofuels as clean energy sources as compared to conventional fossil fuels.

The energy conversion of biomass through thermochemical route depends upon the kinetics of the thermal decomposition of the biomass. The biomass can be converted into value-added products through many possible thermochemical processes. As far as the bioenergy is concerned on biomass, the thermochemical process such as pyrolysis, gasification and combustion are the most acknowledged and practical technologies that suit the need [2]. To date, around 97% of bioenergy production in the world are achieved through combustion process [3]. At present, India produces about 450–500 million tons of biomass per year [4]. This quantity of biomass is equivalent to around 250 million tons of coal and has the capability to generate 17,000 MW power [5]. Biomass based energy has a substantial role in rural livelihood in Indian context owing to its rich availability, where agriculture and forestry occupy 43.5% and 23.5% of the total land area [6].

Bioenergy could be derived through either biochemical or thermochemical routes, where pyrolysis is one of the subgroups of thermochemical processes during which the biomass can be changed into gases, liquid oils

and char. For pyrolysis process, the kinetic analysis is one of the tools for determining the process parameters and their effect on the feedstock conversion. The reaction mechanism of thermal degradation of biomass mainly depends on the mass ratio of principal components in the biomass such as hemicelluloses, cellulose and lignin [7]. Based on the difference in composition of biomass feed the thermal degradation behavior of the pyrolysis gets varied. The study on pyrolysis is as important as it is the beginning reaction step in combustion and gasification other than as a single process. During pyrolysis, the molecular biomass is broken down into volatile, tar and solid chars and their richness in calorific nature promote them as that can be utilized as fuel. The kinetic rate of pyrolysis process reaction determines the chemical transformation of solid biomass into value-added products.

Thermogravimetric analysis monitors the decomposition of biomass during pyrolysis in which the changes in mass with respect to time and temperature is measured. It is one of the most frequently used methods to study the kinetics of thermal decomposition reactions. In a non-isothermal TGA analysis, the study and understanding of the pyrolysis kinetics are normally done by subjecting the biomass heating up to 900°C at the low heating rate. The occurrence and the high number of the mixed nature of reactions result in the thermal degradation of lignocellulosic biomass into the more complex process. The kinetic study of different organic materials through differential thermogravimetry (DTG) is the measurement of weight loss or weight gain of material that is a function of temperature. The bioenergy conversion from low-density biomass materials necessitates the knowledge of thermal properties of biomaterial and the study on reaction kinetics for developing the systems technically as well as economically possible [8].

The physical and chemical characteristics of the biomass materials have an adverse impact on the energy content. The activation energy of biomass materials depends upon the heating rate, chemical composition and the atmosphere in which the thermal decomposition takes place. Keeping view in these aspects, many researchers carried out basic research in pyrolysis to determine the process yields, the effects of the process parameters such as heating rate and time with the wide, resulting array of feedstocks such as wood and agriculture residues, edible oil seeds and cakes, and non-edible oil seeds [4]. Currently, the researchers are focusing on the various advanced technologies for efficient conversion of the biomass previously considered as waste into useful secondary energy forms. For instance, the previous generation of biodiesel production technology aimed to use only vegetable oil extracted from non-edible seeds while the current generation started focusing on utilization of non-edible seed cakes too as it has resulted in the huge quantum of waste through the intensive usage of non-edible seeds [9]. The disposal of the rendered cakes as a waste raises serious environmental concerns due to the presence of heavy oil content in the non-edible seed cakes and its toxic nature. While the non-edible seed cakes as biomass hindering the utilization as cattle feed and as fertilizer for soil enrichment, but provides ample possibility for energy conversion through various biochemical and thermochemical processes. The rich nature of highly volatile matter content in de-oiled nonedible seed cakes assures the great potentiality for energy extraction by gasification process and makes the fuel more reactivity with enablement to convert the fuel into gas with less char. The low ash content eliminates the slogging problems in gasification as well as pyrolysis processes [10].

In this work, we have studied the TGA analysis of three different non-edible seed cakes such as *Jatropha curcas* (Jatropha), *Ricinus communis* (castor) and *Pongamia pinnata* (Pongamia) at a heating rate of 30°C/min to find the kinetic parameters such as activation energy (E), pre-exponential factor (A) and reaction order (n) by using Arrhenius model. The rationale for the study is because almost 65 to 70% of seed have been rendered as residues in the form of cakes during the process of oil extraction from these seeds. The outcome of the study could assist in proposing the appropriate blending of these non-edible seed cakes in varying ratios with higher activation energy materials for efficient pyrolysis.

### II. MATERIALS AND METHODS:

### A. Raw materials:

The deoiled cakes of Jatropha, Castor, and Pongamia seeds were utilized from Energy lab of Department of Mechanical Engineering of Periyar Maniammai University, India. The collected deoiled cakes were powdered using the mini household grinder to minimize the heat transfer effects during pyrolysis study. Thus, the powdered samples were air dried and desiccated before it was subjected to characterization and further analysis.



Fig. 1: Non-edible seed and de-oiled seed cakes

# B. Characterization of raw materials:

The non-edible seed cakes of Jatropha, Castor, and Pongamia were subjected to wide array of analytical process and the observation of the changes in the solid materials were noted down. The proximate and ultimate analysis provides the information on physical and chemical characteristics of the raw materials. In the case of proximate analysis, the moisture content, ash content and volatile matter of the non-edible seed cakes were determined according to ASTM D 3173, ASTM D 3174 and ASTM D 3175 protocols. The moisture content of the samples was obtained by drying till constant weight was attained using an oven. The ash content and volatile matter were determined after slow combustion of the samples in a lab furnace. The fixed carbon content of the samples was calculated finally by estimating the difference in the weight of the samples.

The biochemical composition of Jatropha, Pongamia and Castor seed cakes such as cellulose, hemicellulose, and lignin content were determined as outlined in Chutia et al., 2013 [11]. The fiber fractionation was done as per the method outlined in Goering and Vansoset, (1970) [12] and the complete biochemical analysis was done in Tamilnadu veterinary and animal sciences university, Tamilnadu, India. Lignin was calculated by estimating the ADL (Acid detergent lignin), a residue obtained after treating ADF (acid detergent fiber) with 72% H<sub>2</sub>SO<sub>4</sub>. While ADF is the residue after treating the biomass with an acid detergent solution (Cetyltrimethylammonium bromide (CTAB) in H<sub>2</sub>SO<sub>4</sub> solution) and NDF is the natural detergent fiber residue after treating the biomass that contains all the cellulose, hemicellulose, and lignin with a neutral detergent solution made up of sodium lauryl sulfate and EDTA. Once the lignin was estimated, hemicellulose was calculated by deducting the difference between NDF and ADF. Finally, cellulose were calculated by removing the sum of hemicellulose and lignin content.

The elemental composition such as Carbon (C), Hydrogen (H), Nitrogen (N) and Sulphur (S) were obtained for these non-edible seed cakes by carried out the analysis in Thermoscientific Flash 2000 organic elemental analyzer on CHNS/O mode. One gram of pelletized seed cakes were subjected to combustion studies using bomb calorimeter. The gross calorific value (GCV) and net calorific values (NCV) were determined using the Eq. 1 and Eq. 2.

$$GCV = 34.1C + 132.2H + 6.8S - 1.53A - 12.0(O + N), MJ/Kg$$
 (Eq. 1)

$$NCV = GCV \times \left(1 - \frac{w}{100}\right) - 2.44 \times \left(\frac{w}{100}\right) - 2.44 \times \left(\frac{H}{100}\right) \times 8.94 \times \left(1 - \frac{w}{100}\right), \frac{MJ}{Kg}$$
 (Eq. 2)

Where C, H, S, A, O, N and W are the weight % of carbon, hydrogen, sulfur, ash, oxygen, nitrogen and moisture content of the fuel. Further, the value of 2.44 in the Eq. 2. is the enthalpy difference between gaseous and liquid water at 25°C and 8.94 is the mass molecular ratio between water and hydrogen.

## C. Thermogravimetric analysis

The characterized raw materials of non-edible seed cakes were subjected to thermogravimetric analysis for determining the particular temperature range for efficient pyrolysis. The TGA analysis was carried out by using Shimadzu TGA 50 H instrument. This analysis was done by taking 5 to 6 mg of corresponding samples and heating up to a temperature of 1000°C in a nitrogen atmosphere at a heating rate of 30°C/min. The plotting of TGA weight loss versus temperature shows the range of temperature at which the maximum thermal degradation of samples were taken place.

### D. Estimation of activation energy

The activation energy of a substance could be calculated by modelling the pyrolysis reactions as one step global model with first order rate equation as follows:

A solids 
$$\longrightarrow$$
 B solids + C volatiles (Eq. 3)  
For first order reactions,  $\frac{dx}{dt} = k(1-x)$  (Eq. 4)  
where  $k = k_0 e^{\left(\frac{-Ea}{RT}\right)}$  (Eq. 5)

Where, k is the reaction rate constant; ko is the frequency factor (or) pre-exponential factor; Ea is the activation energy in J/mol; R is the gas constant (8.314 J/mol); T is the adiabatic temperature in deg K. By plotting the experimentally determined reaction rate constants as a function of temperature (i.e.) [ln k versus 1/T] using TGA data. As the plot can be observed as a linear and from the slope of the plot that is equal to -Ea/R, the activation energy could be obtained

## III. RESULTS AND DISCUSSION

# A. Characterization of deoiled non-edible seed cakes:

The proximate analysis exhibits the characterisation of any material through volatile matters, fixed carbon content, ash content and moisture content that aids in comprehending the fuel quality of the solid materials. The high volatile content of the solid material results more volatile nature than solid fuels whereas less loss due to the fixed carbon content during pyrolysis. The fixed carbon of Castor and Pongamia seed cakes were almost equal as to 22%, while the Jatropha has around 12%. During the initial preliminary characterization of the three cake residues, the volatile matter were almost above 60% that could strongly influences its decomposition behavior of the biomass material. It was observed from the proximate analysis that after pyrolysis in case of Jatropha and Pongamia seed cakes, the volatile matter content has been almost reduced by half from 64.50% and 65.57%. However, in the event of castor cake, the volatile matter has been observed from 60% to 39.17%.

The proximate analysis revealed that the biomass has been predominated with the voaltile matters. The mositure content could also influences the thermal properties of the biomass. The conversion efficiency of the any thermochemical process and the heating value of the biomass could be also affected by high moisture content that further triggers the loss of energy through decomposition while storage. However, in case of these three cake residues, the moisture content were less than that of 8%. During the initial stage of pyrolysis reactions, minimal weight loss of samples could be attributed due to desorption of moisture as the bound water on the surface and the micropores of the samples. Latterly the weight loss were suddenly increased after 200°C that could be attributed to the decomposition of oil cakes with subsequent release of volatile matters. The fixed carbon of the three de-oiled cake residues were above 12% and ash content were low as less than 3% suggests the potentiality of these residues as the appropriate biomass feedstocks for pyrolysis process. The inorganic component (ash content) of de-oiled cake residues affects both the handling and processing costs of overall biomass energy conversion process. The low moisture, ash content and high volatile matter suggest these de-oiled cake residues as a potential candidate for pyrolysis process.

Properties (%)	Jatropha seed cake	Castor seed cake	Pongamia seed cake
Neutral detergent fiber (NDF)	47.51	64.11	18.19
Acid detergent fiber (ADF)	36.35	44.62	11.00
C:H Molar ratio	$0.61 \pm 0.04$	$0.57 \pm 0.03$	$0.56 \pm 0.02$
C:N Molar ratio	$7.70 \pm 0.32$	$7.92 \pm 0.51$	$7.60 \pm 0.40$
Gross calorific value (MJ/Kg)	$16.95 \pm 0.68$	$15.96 \pm 0.13$	$17.62 \pm 0.87$
Net calorific value (MJ/Kg)	$14.37 \pm 0.26$	$13.27 \pm 0.40$	$14.69 \pm 0.76$
Empirical formula (on Ash free basis)	$C_8H_{13}NO_6$	$C_6H_{10}NO_4$	$C_8H_{14}NO_5$

Table. 1: Characterization of non-edible de-oiled seed cakes

The ultimate analysis of these three samples revealed that the samples were highly predominated with carbon and oxygen elements. The detailed elemental characterization of the various de-oiled cake residues were shown in Fig. 2. The molar ratio of the carbon: hydrogen and carbon: nitrogen were also calculated and listed as in Table 1. It showed that slight variations in carbon, oxygen, hydrogen and nitrogen content among these nonedible seed cakes. Based on the ultimate analysis, the gross and net calorific values of the cake residues were also calculated and listed as in Table 1. The GCV and NCV values of these de-oiled cake residues were highest in the case of Pongamia with 17.62 and 14.69 MJ/kg. The Jatropha was around 16.95 and 14.37 with Castor as lowest as to 15.96 and 13.27 MJ/kg. These GCV and NCV were in good agreement with the findings of Chutia et al., 2013 for *Mesua ferrea L*. deoiled cake with 18.68 and 16.28MJ/kg, respectively [11]. In addition, the empirical formulas for the three cake residues were also developed with the help of elemental analysis.

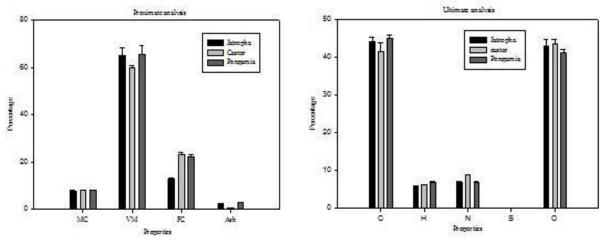


Fig. 2: Characteristics of non-edible de-oiled seed cakes: a) proximate analysis and b) ultimate analysis [MC - Moisture content; VM - Volatile Matter; FC - Fixed Carbon;

 $C-Carbon;\, H-\ Hydrogen;\ N-Nitrogen;\, O-Oxygen]$ 

# B. Thermogravimetric Analysis:

Pyrolysis is the heating of a substance in the absence of air. During pyrolysis, the varying thermal behavior could expected due to the constituents of cellulose, hemicelluloses and lignin varying with respect to the biomass materials. Further, the crystalline and amorphous nature of cellulose and hemicellulose are found mostly on cell walls of the biomass with little strength. Though lignin does not having distinct structure, it aids in binding the fibrous cellulosic particles. As the thermal degradation of these components is at different temperature range, the study on lignocellulosic compounds appropriates to pyrolysis. High cellulose and high lignin content of the biomass samples dictates the faster and slower pyrolysis rate. Among the species under investigation, Jatropha and Castor de-oiled seed cakes have high lignin content as 20%, while the Pongamia seed cakes has high cellulose content. Thus, the composition of cellulose and lignin in the biomass was one of the important parameters that affect the pyrolysis and combustion characteristics greatly.

The lignocellulosic components such as hemicellulose, cellulose and lignin also greatly influenced the thermal decomposition of the biomass and generally decompose over the temperature range of 200-400°C. As in TGA curve of Jatropha seed cake (Fig. 3 a), it has been observed that the removal of moisture and light volatiles is up to 200°C with a weight loss of 6.65%. The weight loss arose on this stage could be attributed due to the removal of physically absorbed surface water along with negligible quantum of very light volatile organic compounds. The major weight loss of this non-edible Jatropha is about between 200-600°C. Almost 62.45% of weight reduction is between 199-548°C with maximum weight reduction rate at 333°C as it has been observed from DTG curve. This particular stage is referred to as active pyrolysis zone as the rate of weight loss is very high. Beyond this zone, since the rate of weight loss is quite low as compared to previous stages denotes the passive pyrolysis zone. In case of castor seed cake, (Fig. 3 b), the TGA showed that the reduction of mass is between 200-870°C with three high removal rates. In this case, the removal of moisture and light volatiles was about 200°C with a maximum removal rate at 89°C. The rapid reduction of mass of 51% was between 210-500°C with peak reduction rates at 291°C and 357°C. In case of Pongamia seed cakes (Fig. 3 c), the removal of moisture and volatiles at about 160°C and the weight loss was between 200-670°C with 79.75% reduction of mass. From this TGA analysis of these three different non-edible seed cakes as biomass substances, 200-650°C was observed at a diverse rate of weight reduction temperature. The pyrolysis of the lignocellulosic biomass divided into three regions: moisture and light volatile component removal (<200°C) degradation of hemicelluloses (200-280°C), cellulose (280-380°C) and lignin degraded at slower rate over wide temperature range of 200-900°C. The TGA curves shows that the removal of moisture and light volatile is followed by the devolatization at 200°C for Jatropha and Pongamia whereas the castor at 225°C. The maximum devolatisation occurs in the second region of weight loss between 280-380°C.

The biomass pyrolysis can be modeled like cellulose pyrolysis, but the presence of hemicelluloses and lignin along with cellulose at different composition may vary and makes the modeling difficult [13]. The decomposition region of hemicellulose and the cellulose are in the temperature range of 220-315°C and 315-400°C. Lignin decomposes in the range of 180-900°C with unclear maximum weight loss [14]. Though lignin decomposition initiates at the lowest temperature, it might continue up to the temperature around 900°C. In the thermal degradation of wood, the most reactive compounds such as hemicelluloses decompose at a temperature in the range of 225-320°C, cellulose at 305-375°C and lignin gradually over the temperature range of 250-500°C [15]. During thermochemical energy conversion of biomass into useful products, the activation energy and pre-exponential factor are not constant throughout the decomposition process and dependent on conversion mechanism [12]. The pyrolysis temperature for most of the non-Edible de-oiled cakes lies in the range of 400-800°C. The maximum thermal degradation was found to be for grapeseed cake at 400-700°C, [16] grape bagasse around 350-690°C [17] and 200-500°C for cherry seeds [18].

### C. Kinetic study of the thermogravimetric analysis:

The kinetic study of pyrolysis enables to determine the activation energy required for reaction that brings the view of ease of occurrence of reactions. Since the activation energy determines the nature of reactions in thermal degradation, the reactions with less activation energy occurs much easier than the reactions that require high activation energy. It was observed that the Castor seed cake require more energy to initiate the combustion reactions and with increase in temperature, the activation energy was lesser than that of Jatropha and Pongamia castor seed cakes. Since the heating rate might also strongly influences the activation energy in thermogravimetric decomposition process as reported in several literatures with wide varying heating rate of 5°C/min to 40°C/min, this particular study was done at the constant heating rate of 30°C/min to understand the effect of varying biomass resources in view of species under consideration of de-oiled seed cakes.

Table 2 shows the activation energy of three non-edible seed cakes at different temperature range. Among the three non-edible seed cakes studied, the degradation of cellulose component was observed to happen between 280-380°C while the degradation of hemicellulose were occurred at the temperature ranges around 200-320°C. The castor seed cake exhibited the lowest activation energy of 28.51kJ/mol while the highest was obtained in case of Pongamia seed cakes with 51.48kJ/mol. The biocomponent lignin in the seed cakes degrading at a slower rate over the long temperature of 200-590°C required an almost equal amount of activation energy around 26kJ/mol. As the non-edible seed cakes are oil extracted residues the activation energy for thermal decomposition of Jatropha, Castor and Pongamia seed cakes are varied depending on their biochemical compositions of hemicelluloses, cellulose and lignin content. The reported activation energy values are 145 to 285kJ/mol for cellulose, 90 to 125kJ/mol for hemicellulose and 30 to 39kJ/mol for lignin [19, 20].

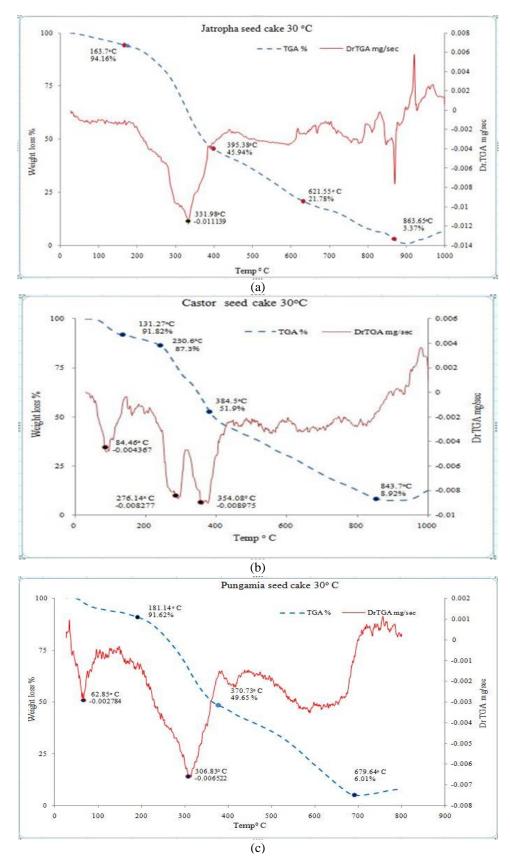


Fig. 3: TGA and DTG curve of a) Jatropha seed cake; b) Castor seed cake and c) Pongamia seed cakes

Th activation energy for the three deoiled cakes were calculated through kinetic analysis and the plots were shown in Fig. 4. In a nutshell, the investigated species has relatively high activation energy with the other reported cellulosic biomass feedstocks such as corn stalk, cotton stalk, wheat straw, tree skin, wood chip, peanut shell and seed shell, where the activation energies were around less than 100kJ/mol. For instance, the

rich cellulosic biomass feedstock such as cotton and filter paper have more than 200kJ/mol of activation energies. However, in view of utilization of waste resources rather than the fresh materials necessitates the implementation of these seed cake residues as a potential biomass feedstock.

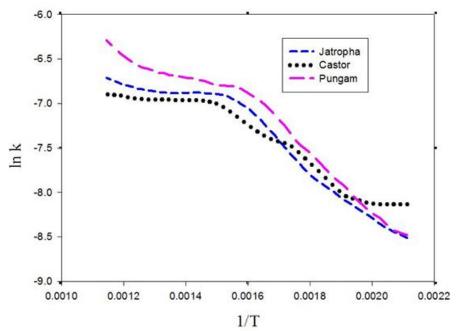


Fig 4. Kinetics analysis plot for determination of activation Energy

Table. 2: Activation energy of non-edible de-oiled seed cakes

Temperature zone (T°C)	Activation energy (E, kJ/mol)	Pre-exponential factor (A, min <sup>-1</sup> )	Reaction order (n, dimensionless)	Correlation coefficient $(R^2, dimensionless)$
Jatropha seed	d cake			
200-280	41.93	7×10 <sup>-5</sup>	1	0.97
280-380	40.05 108.73	0.18	1	0.98
240-520	26.75	0.02	1	0.91
Castor seed c	ake			
225-320	41.71	0.05	1	0.97
280-380	28.51 94.74	0.02	1	0.98
200-500	24.52	0.01	1	0.95
Pongamia see	ed cake			
200-320	51.84	0.19	1	0.99
280-380	47.44 126.91	0.23	1	0.99
200-590	26.91	0.02	1	0.93

# IV. CONCLUSION

The physicochemical and biochemical characteristics along with thermal behavior of the three non-edible seed cakes such as Jatropha, Castor and Pongamia has been investigated and the activation energy were found to be as 94.74kJ/mol, 108.73kJ/mol and 126.91kJ/mol respectively. The temperature profile, activation energy (E), pre-exponential factor (A) and reaction order (n) for the active pyrolysis zone of the three species has been studied for the constant heating rates using Arrhenius reactions model. Pongamia seed cake was found to be having faster pyrolysis rate due to the higher quantum of cellulose content with fourfold lesser content of lignin content advocates the de-oiled seed cake residues as a potential biomass feedstock for thermochemical

conversion. The kinetic analysis provides information on activation energy and explains the thermal degradation nature of thermo chemical conversion of energy could assists in optimizing the pyrolysis regimes in design of conversion systems and contributes for the advancement in co-gasification technology.

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