



# Investigation of Thermal Stability of Bakelite by Isoconversional Thermal Analysis

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**ABSTRACT:** Bakelite or polyoxybenzylmethyleneglycolanhydride is the world's first fully synthetic plastic. This research work reports on thermal degradation profile of Bakelite using isoconversional thermal analysis as per the recommendation of International Confederation for Thermal Analysis and Calorimetry (ICTAC). For this purpose, thermal degradation of Bakelite was recorded on a SDT Q600 thermal analyzer at heating rates of 5,10,15 and 20°C/min in the range between 20 to 1000°C. We observed that the degradation of Bakelite occurred in three steps but the first and third steps are insignificant with respect to mass loss and kinetics. The initial and final degradation temperatures (Td<sub>i</sub>, Tdf) of Bakelite were recorded between  $205 - 228^{\circ}$ C and  $378 - 427^{\circ}$ C, respectively at different heating rates. The mass loss associated with this phase was calculated about 59%. The<sup>Td</sup>d<sub>m</sub> values were found to be  $283 - 325^{\circ}$ C. Flynn-Wall-Ozawa (FWO) and Kissinger models were applied on the data to find the kinetic parameters such as activation energy (E a) and order of reaction (n). The Ea value was found to be 102.73 and 104.31 kJ/mol as per FWO and Kissinger models while order of reaction was 1.09. Thermodynamic parameters were also found and the value of  $\Delta H, \Delta S$  and  $\Delta G$  were found 97.89 kJ/mol, -141.60 kJ/K and 180.46 kJ/mol, receptively. This study explains that Bakelite is a thermally stable compound.

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### I. INTRODUCTION

Plastics are a wide range of synthetic, semi-synthetic or natural materials. The main ingredient of plastics is organic polymers. They have the property of plasticity, which means that they can be pressed molded or extruded into various shapes of solid objects. Plastics are widely used due to its plasticity and various other properties such as durability, flexibility, being lightweight and inexpensive to produce<sup>1</sup>. Plastics are mainly produced by human industrial systems. Mostly plastics are made from fossil fuel-based chemicals such as petroleum or natural gas but recently industries use renewable materials like cotton and corn derivatives<sup>2,3</sup>. In more developed countries about one third of plastic is used in packaging and the same amount of plastic is used in building materials like plumbing, piping etc<sup>4</sup>. Up to 20% of plastic consumption is in automobiles, toys and furniture. In the developing countries, the usage of plastic may differ<sup>5</sup>.

Bakelite or polyoxybenzylmethyleneglycolanhydride is the world's first fully synthetic plastic. Its chemical formula is  $(C_6H_6O.CH_2O_n)$ . Its density is 1.3 g/cm<sup>3</sup>. It is infusible, hard and chemically resistant plastic. Bakelite is a trade name, which was given to phenolformaldehyde resin. It is a type of thermoplastic polymer, which is dimensionally stable. It is a cross-linked polymer produced by the chemical reaction of phenol and formaldehyde either in basic media or in acidic media<sup>6</sup>. Bakelite is a major commercial product. It is

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important and common polymer used for making various objects. Major uses of Bakelite are in electrical systems for making machine parts and electrical switches because Bakelite has high heat resistant and low electrical conductivity. Bakelite has adhesive and binding properties so they are used in making gums. Bakelite is used for protective purposes in coating industry<sup>7</sup>. Handlings of variety of utensils are made of Bakelite. Various objects are made up of Bakelite such as clocks, toys, kitchenware, washing machine impeller, buttons etc. Bakelite can be prepared in different colors so it is widely used in making jewelry (bracelets, earrings and bangles). Artificial jewelry made of metals can cause skin irritation. Bakelite jewelry is safe to wear and is more abundant in market<sup>8</sup>. Bakelite is used in telephone fixtures, fountain pens, castanets, pipe stems, billiard balls, radiator caps etc<sup>9</sup>. The use of Bakelite in nowadays is less as compared to previous years, but it is still effective  $^{10,11}$ . There are many other cheap products in the market, which have reduced the usage of Bakelite. Bakelite was produced in the twentieth century but it was the main subject of study until 21<sup>st</sup> century. Bakelite was extensively used in commercial products due to its chemical, physical, thermal and electrical properties.

On a global basis, 175,000 tonnes of bakelite are manufactured annually and used in 15,000 different products<sup>12</sup>. The widespread use of synthetic bakelite in various products, including electronic components, polymer metals, dangerous chemicals, phone cases, household items, plastic containers, and pipe stems, has led to a significant amount of waste, contributing to pollution<sup>13</sup>. Literature reports that waste bakelite could be exploited as a source of carbon and oil using pyrolysis. But the pyrolysis of such thermosetting plastic material (bakelite), is not a easy task due to cross-linking and hardening at high heating  $1^{14}$ . The objective of the study is to investigate thermal degradation parameters as per recommendations of ICTAC that will not only help to establish its performance parameters but also may help researchers to develop a recycling method for this thermosetting synthetic polymer which is also an environmental threat as a pollutant. Moreover, it can be hypothesized that recycling of Bakelite waste can become an alternative carbon resource for steel industry. So investigation of thermal degradation parameters of bakelite are necessary to convert this bulk waste into rich source of carbon.

#### II. MATERIAL AND METHODS

#### A. MATERIAL

Bakelite was purchased from Taian Guangyuan International Trade Co., Ltd (Shandong, China).

#### **B. MEASUREMENTS**

SDT Q-600 thermal analyzer (TA Instruments, USA) was used to record the thermogravimetric data of Bakelite under  $N_2(100 \text{ mL/min})$  in ramp mode. Data was acquired on four different heating rates of 10, 15, 20 and  $30^{\circ}$ C/min. Universal Analysis 2000 (TA instruments, USA) was used to process the data.

# C. Isoconversionai Thermal Analysis and Degradation Kinetics

The objective of studying the kinetics of a reaction is to determine the kinetic parameters. Thermal analysis has demonstrated significant efficacy, particularly in the context of solidstate reactions. The process includes the degradation of a sample by subjecting it to both isothermal and nonisothermal conditions<sup>15</sup>. Isoconversional or nonisothermal methods are preferred over isothermal approaches and being utilized more frequently to study the kinetics of solid-state reactions. Several kinetic models are available for analyzing thermal degradation data, including ASTM standard, Coats-Redfern, and isoconversional methods, each with distinct features, suitability, advantages, and limitations. In nonisothermal or isoconversional methods, it is assumed that the energy of activation of a degradation reaction remains same in a range of heating rates.

These methods enable the determination of activation energy  $(E_a)$  without requiring the assumption of a specific reaction model. Known for the complex degradation mechanism, which may involve multiple competing reactions. This model-free approach is advantageous as it minimizes potential errors associated with predefined reaction orders in contrast to the alternative approaches such as the Coats-Redfern method and ASTM standard kinetic models as reported in literature. The Coats-Redfern method assumes a single reaction model, which may not accurately capture the complexity of polymer decomposition. Similarly, while ASTM methods are widely accepted, they are typically limited to a single heating rate, which restricts their applicability for thorough kinetic analysis.

The attraction of isoconversion methods, the Flynn-Wall-Ozawa analysis<sup>6,16,17</sup> stems from the possibility of being able to determine the Ea for certain prerequisites,

without knowing the kinetic model of the reaction mechanism. In contrast to the above, the traditional singlecurve analyses such as those of Borchardt and Daniels<sup>18</sup> or Coats and Redfern<sup>19</sup> also supply totally inaccurate values of the activation energy when a false model has been chosen, even when the quality of fitting is very high<sup>20,21</sup>. This shortcoming is caused by the lack of information content of a single non-isothermal experiment so that the simultaneous determination of the correct kinetic model and the parameters is not possible.

By using TG and DTG curves the initial, maximum and final degradation temperatures (represented by Tdi, Tdm, and Tdf respectively) of each thermal degradation step was estimated. Different isoconversional approaches such as Kissinger and FWO were used for the kinetic examination of thermal data.

FWO method is based on the fact that for a given rate of conversion ( $\alpha$ ) the rate of thermal degradation response depends purely on temperature. When a graph is plotted between  $\ln\beta$  and 1/T for given value of  $\alpha$  and for different heating rates, a straight line is obtained which gives the value of activation energy ( $E_a$ ).

For the calculation, the slope of graph is needed.

$$\ln\beta = \frac{AE_a}{Rg(\alpha)} - 5.331 - 1.052\frac{E_a}{RT} \tag{1}$$

where A is the pre-exponential factor, R is the general gas constant, T is absolute temperature and  $\beta$  represents the heating rate. In the above equation,  $\alpha$  is calculated by given relation Eq. 2.

$$\alpha = \frac{m_0 - m_t}{m_0 - m_f} \tag{2}$$

In Kissinger's method, Tdm in DTG curve was considered as temperature with maximum reaction rate. This method uses following equation.

$$\ln \frac{\beta}{T_m^2} = -\frac{E_a}{RT_m} + C \tag{3}$$

For a constant conversion,  $\ln (\beta/T_m^2)$  is plotted against 1000/T<sub>m</sub> which gives the value of  $E_a$  at that conversion. Kissinger also developed a method to estimate order of reaction (n) value by following equation.

$$S = \frac{\left[ \left( \frac{d^2 \alpha}{dt^2} \right) \right]_L}{\left[ \left( \frac{d^2 \alpha}{dt^2} \right) \right]_R} \tag{4}$$

2DTG curves are used for the determination of values of  $(d^2\alpha/dt^2)_R$  and  $(d^2\alpha/dt^2)_L$  where subscripts " R " and " L " represent the data points on the right and left sides of the 2DTG peaks. The value of " S " helps in the determination of n using equation:

$$n = 1.88 \text{ S} \quad (S \ge 0.45) n = 1.26 \text{ S}^{0.5} \quad (S \le 0.45)$$
(5)

For the investigation of thermodynamic parameters (entropy (S), enthalpy (H) and Gibbs free energy (G)) Eyring-Polanyi equation was used.

$$k = \frac{k_a T}{h} e^{\frac{\Delta G}{RT}} \tag{6}$$

Where h is the Planks constant,  $\Delta G$  is the Gibbs free energy and  $K_{\rm B}$  is the Boltzmann constant.

$$\ln\frac{K}{T} = -\frac{\Delta H}{R} \times \frac{1}{T} + \ln\frac{K_a}{h} + \frac{\Delta S}{R}$$
(7)

In this equation K is the rate constant,  $\Delta H$  is the enthalpy of reaction, T is the absolute temperature,  $\Delta S$  is the entropy of reaction,  $K_{\rm B}$  is the Boltzmann constant, R is the general gas constant and h is the Planks constant. When we plot  $\ln \frac{K}{T}$  against  $\frac{1}{T}$ , it gives a straight line. The slope of straight line gives the enthalpy of reaction while intercept gives the entropy of the given thermal degradation reaction.

By using the approach provided by Doyle, the complete index of integral procedural decomposition temperature (IPDT) and intrinsic thermal stability (ITS) were estimated. The values of IPTD and ITS for all four heating rates were calculated by using TG curves and the average values are presented.

#### **III. RESULTS AND DISCUSSION**

#### A. THERMOGRAVIMETRIC ANALYSIS

Thermal stability of bakelite was determined by thermogravimatric analysis. For the investigation of thermal degradation behavior of bakelite, multiple heating rates were used in the range of 20 to 1000°C. TGA curve (Fig. 1) showed a little dent up to 130°C indicating some moisture loss. Then two stages of degradation were observed. But the second stage appeared as a step with major mass loss of 59.11%. The initial and final and maximum degradation temperatures (Td<sub>i</sub>, Td<sub>f</sub> and

Method	Type	Suitability for Degradation	Advantages	Limitations
	Non-Isothermal	Highly suitable	Activation energy	
	Model-free	for complex	at multiple	Requires multiple
FWO	method	degradation	conversion points	heating rates
	Non-Isothermal	Highly suitable	Simple analysis	Lacks detailed
	Model-free	for complex	widely used	mechanistic
Kissinger	method	degradation	for peaks	information
	Isothermal method (specific	Limited for multi-step	Relatively simple	May misrepresent complex
Coats-Redfern	mechanism)	degradation	to apply	degradation
	Reaction model-	Limited for	Industry-	
	based method	$\operatorname{multi-step}$	standard	Single heating
ASTM Standard	(specific order)	degradation	acceptance	rate limitation

TABLE I: A comparison of kinetic models for analyzing thermal degradation.

 $Td_m$ , respectively) for main considerable stage of thermal degradation were found to be 214,387 and 293°C, respectively. The char yield of bakelite was recorded as 1.72% at almost 1000°C.  $Td_j$ ,  $Td_f$  and  $Td_m$  values recorded at different heating rates (Fig. 2) are shown in Table I. The pattern of TG and DTG curves showed that bakelite is a thermally stable material which can with stand a significantly high temperature but it will undergo structural decomposition at almost  $235 - 250^{\circ}C$ and will start to degrade. The remaining mass loss (28.08%) occurred between 410 - 1000°C in a linear manner showing gradual conversion of the material into char.

The decomposition temperature (Td) values recorded at different heat rates are also shown in Table II. These values indicate the interesting factor that Td values increase by increasing heating rate in accordance with literature reported elsewhere.

Heating rate emerges as one of the most critical parameters in this study since materials, when subjected to different heating rates, need varied residence time that manifests itself in the form of variations in thermal degradation temperature ranges and kinetics. So a proper analysis of thermal decomposition provides an insight into the mechanism governing the thermochemical conversion process when various heating rates are applied. Increasing heating rate leads to a corresponding shift in the reaction zone to higher temperatures for MS biomass. This behavior can be deciphered based on residence time. A sample subjected to low heating rates has a high residence time due to which the thermal gradients penetrate up to the inner core of particles. On the other hand, high heating rates lead to a lowering in residence time. The thermal gradients get enough time to spread out evenly throughout the particle, thus showing a high peak temperature and less dominant peaks in the DTG curve.

When we plot a graph between  $\alpha$  and T curves obtained at different heating rates for the significant stage of thermal degradation of bakelite, identical degradation patterns was observed as shown in Fig. 3.

#### B. Degradation Kinetics

Thermal degradation may result in molecular deterioration of a polymer depending upon their physical and chemical composition. Different mechanisms exist for thermal degradation. The most common one is the unzipping or breaking of bond between polymer molecules with release of oligomers and monomer units. Cross-linking, elimination, and cyclization reactions also contribute breaking down of original polymer into its monomer or other modified end products.

FWO and Kissinger isoconversional methods were used to assess the different kinetic parameters like activation energy (Ea) and frequency factor (A). For the determination of order (n) of degradation reactions, Kissinger's method of shape index was used while for the determination of breakdown at different transformations ( $\alpha = 0.1 - 0.9$  with 0.1 increment), FWO the most reliable approach is used.

Both ways rely on different approximation techniques for getting the temperature integral. Also,  $R^2$  values close to unity imply that isoconversional models better mimic the experimental data to estimate kinetics. Also, activation energy, Ea which signifies energy barriers during chemical reactions is significant for thorogh understanding of kinetics.

The average value of Ea for Bakelite's major mass degradation step was  $102.73 \text{ kJ mol}^{-1}$  (Table II). The



FIG. 2: DTG curves of Bakelite at multiple heating rates.

TABLE II: Thermal decomposition temperatures, mass loss (%) and char yield (%) of bakelite recorded at multiple heating rates.

Sample	В	Td (°C)	$\mathbf{Td}_{m}$ (°C)	Td $_f$ (°C)	Wt. loss (%) at $T_{\rm f}$	Char yield mass $(\%)$
Bakelite	$5^{\circ}C/min$	205	283	378	57.66	$0.87\%$ at $999^{\circ}C$
	$10^{\circ}C/min$	214	293	387	59.11	$1.72\%$ at $999^{\circ}C$
	15°C/min	220	302	413	59.86	$1.06\%$ at $999^{\circ}C$
	$20^{\circ}C/min$	228	325	427	60.31	8.47% at 999°C



FIG. 3:  $f'(\eta)$  in opposition to  $\frac{a}{c}$  for  $\phi_2$ .

FWO plots of  $\ln\beta$  vs. 1000/T at different  $\alpha$  degrees of conversion for thermal degradation step of bakelite, are shown in Fig. 4. The Ea value estimated in this study confirms an inverse relation with the rate of reaction. As the activation energy increases, the rate of reaction decreases. energy barrier. This factors is also evidenced by higher weight loss per each °C increment as shown in Fig. 2 (DTG curve) for major mass loss step that has been reported here.

In the Kissinger technique,  ${\rm Td_m}$  is measured at each heating rate. The values of activation energy is evaluated by straight line graph that is plotted between  $1000/T_{\rm m}$  and  $\ln\left(\beta/T_{\rm m}^2\right)$  as shown in Fig. 2b . Ea for the degradation of Bakelite was 104.31 kJ mol^{-1}. The values of Ea evaluated by FWO and Kissinger method were very similar. In each thermogram, Bakelite showed first order kinetics which was predicted by Kissinger method.

The experimental findings also indicate multi-step degradation kinetics for bakelite highlighting the necessity for model-free methods such as FWO and Kissinger for thorough analysis.

#### C. Thermodynamic Analysis

Thermodynamic parameters like change in entropy  $(\Delta S)$ , change in enthalpy  $(\Delta H)$  and change in Gibbs free energy  $(\Delta G)$  were measured by using TG data for Bakelite (Fig. 1a). TG curves is used to estimate inte-

gral procedural decomposition temperature (IPDT) and intrinsic thermal stability (ITS). For Bakelite, the mean IPDT value was found to be 323°C while ITS value was found to be 0.47. Substantially higher IPDT and ITS values suggested that Bakelite is thermally stable.

# IV. CONCLUSION

Present study reports investigation of thermal degradation of bakelite using isoconversional thermal analysis which revealed three step degradations, among which the first step was removal of moisture, second step was the major thermal decomposition and third step was the gradual conversion of the polymer into char like material. Bakelite showed high thermal stability. Similarly, other thermal stability parameters also establish bakelite as thermally stable material, which can be used to prepare appliances that operate at higher temperatures. Additionally, an understanding of degradation behavior can enhance predictive maintenance strategies, ensure regulatory compliance, and optimize recycling methods for bakelite-based components. Moreover, the degradation temperatures also enable to develop a method for recycling or converting spent bakelite into carbon for use in steel industry. The determination of kinetic parameters and analysis of product distribution will be useful in the utilization of bakelite waste as an energy source on a commercial scale.



FIG. 4: Flynn-Wall-Ozawa (FWO) plot for the calculation of Ea of major degradation step of Bakelite at several  $\alpha$  values.



FIG. 5: Kissinger plot for the calculation of Ea of major degradation step of Bakelite at several  $\beta$  values.

# DECLARATION OF COMPETING INTER-EST

The authors have no conflicts to disclose.

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Sample	Method	Step	$\boldsymbol{n}$	$E_a \ (kJ/mol)$	$\Delta H \ (kJ/mol)$	$\Delta S$ ( kJ/K )	$\Delta G \; (kJ/mol)$	$\Delta H \; (kJ/mol)$
Bakelite ·	FWO	II	-	102.73	97.89	-141.60	180.46	97.89
	Kissinger	II	1.1	104.31	99.46	-140.56	181.42	99.46

TABLE III: Thermal kinetic and thermodynamic parameters of bakelite.

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