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Thermogravimetric analysis and Chemical kinetics of Waste Tires

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ABSTRACT

The focus of this paper is to analyse the pyrolysis reaction of waste tires. The pyrolysis was studied experimentally using thermal and chemical kinetics. The tires were pyrolyzed using thermogravimetry at a temperature range of 200 to 600°C, with heating rates of 10, 20, and 50°C/min. The activating energy was observed to increase with increased temperature. The highest activating energy was 101.02 kJ/mol at a heating rate of 50°C/min.

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INTRODUCTION

Uncontrolled disposal of waste tires is causing environmental threats globally (Ferronato and Torretta, 2019). Due to their high toxicity and flammability, as well as their persistence and difficulty in storing or processing, these tires pose significant environmental challenge. Approximately 1.4 billion tires waste are produced annually, and they generally end up either in landfills or being burned, which worsens environmental issues (Rogachuk and Okolie, 2023). However, tire stockpiles constitute a potential fire hazard, which once ignited takes long time to extinguish. At the same time, they generate oily residue and emit acrid smoke, which contaminates soil surface, water, and air, which is expensive to handle (Abdallah et al., 2020). Since waste tires are bulky, they occupy greater space in open air dumping and fill the landfill faster (Lu et al., 2023). Also, these methods ignore the potential energy recovery from waste tires.

Scrap tire has High Heating Value (HHV) of 35.75 MJ/kg (Frigo et al., 2014), which is higher than bituminous coal which has 24.19 MJ/kg (Pan et al., 2021). Therefore, the energy recovery from scrap tires could be an excellent method. A direct combustion of waste tire is one of the energy recovery methods that is used, but many studies are against it, because of the emission of toxic solid particles and Palyomatic Hydrocarbons (PAHs) generated are carcinogenic (Carmo-Calado et al., 2020).

Pyrolysis is one of the methods that are accepted by many researchers (Martínez et al., 2013, Hofman and Pietrzak, 2011, Aylón et al., 2010, Kaminsky et al., 2009, Islam et al., 2008,). Besides pyrolysis being appropriate technology for energy recovery, it is energy intensive (Oliveira et al., 2019). Therefore, it requires further study on optimization of energy input and pyrolytic oil production. One interesting phenomenon about pyrolysis is that the pyrolytic oil produced has high fuel to feedstock ratio (e.g. tire Lower Heating Value is 24 MJ/kg whilst its oil produced has 30 MJ/kg). There is the expectation of production of oil and gas from waste tires. The amount of oil and gas produced from the process corresponds with the volatile matter present in the waste tire sample.

Scrap tires' fast pyrolysis requires a short residence time for their complete decomposition, hence the comprehensive study of their reaction kinetics regime is essential. Thermal parameters are also necessary for heat balance formulation during pyrolysis reactions. Various degradation equations have been developed by several researchers from literature. These equations can predict and influence pyrolysis yield. Many of these models were developed based on experimental results and have been proved to be realistic in predicting intermediate products from primary devolitalisation (Torretta, 2019). Relationship between individual tire components that devolitalize either independently or parallel and the heating rate will improve our knowledge of optimizing oil production on tire Pyrolysis process. Tire particle weight loss referred to as conversion and changes of product distribution that occur simultaneously during all tire conversion processes has been found difficult to model. However, it is important to find a general or multipurpose simple model which can be understood mathematically.

Various researchers have studied thermal degradation kinetics of Natural rubber (NR), polybutadiene rubber (PBR) and styrene- butadiene rubber (SBR). Pyrolysed NR and reported that its degradation started at 300°C and its peak mass loss was at 400°C. Their study was done using TGA in nitrogen environment (Lopez et al., 2019). Pyrolysed PBR using TGA and stated that the maximum decomposition rates were at two stages one being 370°C and the other at 470°C (Demirbas, 2017) reported that SBR and PBR degradation were at maximum temperature of about 463°C. Product transformation of constituent components has not been fully exhausted. These components has great influence in the products outcome and distribution of char, condensable volatile and non-condensable volatile (Galvagno et al., 2007). They also play a role for viability of the pyrolysis technology and chemical production. Due to that this paper is going to determine the characteristics and decomposition kinetics of waste tires from Tanzania.

METHODS AND MATERIALS

Scrap tire material was collected from Temeke open dumping site in Dar es Salaam. It was then cleaned and cutting into small pieces of about 8 mm3 by using slicing machine.

Proximate and ultimate analysis study

The amount of moisture, primary volatile matter, fixed carbon, and ash are determined from the study of proximate analysis. The ultimate analysis is the determination of chemical composition of waste.

Moisture content determination

Determination of the maximum amount of moisture content was according to ASTM E1756-01. Waste tire sample was placed into an oven at 105°C to constant mass. The sample was placed in the desiccators, cooled down and reweighed. The difference in weight represented the percentage of moisture content of the sample.

Volatile matter determination

The waste tire fuel Volatile matter is the condensable and non-condensable vapour released from a hydrocarbon material when heated. The process was done as defined by ASTM standard E-872. The equation used for calculating Volatile Matter (VM) is as shown in Equation 1.

$$VM = 100\%\left(\frac{w_i - w_f}{w_i - w_c}\right) \qquad (1)$$

Where: w_c = weight of crucible, w_i = initial mass of waste tyre, w_f = final mass of waste tyre.

The sample used for moisture content determination was heated in a covered crucible to avoid contact with air during devolatilization. The covered crucible was placed into a furnace at 450°C for three hours. The crucible was then taken out and cooled in desiccators. The weight difference after decomposition was referred as volatile matter.

Ash content determination

Ash is the inorganic solid residue left after the fuel is completely burned. The procedure used to determine ash was using ASTM D1102 (ASTM, 1984). The remaining sample from volatile matter calculation was placed in the furnace at 575°C for an hour for combustion. All carbon was burnt, and the remaining ashes were reweighted.

d) Fixed carbon value

Fixed carbon is the material (other than ash) that does not vaporize when heated without oxygen. The value of fixed carbon in the waste tyre is given as the difference from the moisture, volatile matter, and ash contents equation (2);

$$FC = 100 - M - VM - ASH \qquad (2)$$

where: FC = Fixed carbon, M= Moisture, VM = Volatile matter and ASH = remaining ash.

Fixed carbon represents the solid carbon in the tire waste that remains in the char after devolatilization process.

The Calorific value determination

The calorific value is the standard measure of energy or heat of combustion contained in any fuel (Demirbas, 2015). The calorific value were determined as per ASTM D240 (ASTM, 2012). In this experiment, it was determined by using bomb calorimeter type Wagtech Gallenkamp Auto bomb. The tyres were dried and grinded. Bomb was assembled and filled oxygen to a pressure of 30 bars. The bomb was submerged in water and was fired. The temperature changes were recorded.

Thermal-Gravimetric Analysis and DTG experiments of global weight loss rate

The used tyre sample was heated in an inert Atmosphere of a Nitrogen gas starting from room temperature up to 600°C. Initial temperature (Ti) was taken at 5% weight loss. Initial sample masses were heated at heating rates of 10, 20 and 50oC/min the room was air conditioned.

Three replications were taken for each tested condition with different prepared samples. The result found from TG and DTG curves of these experiments could not have much diversion, and then only one result for each condition was taken to represent others. At the end of this experiment the heat supply was switched off, the purge gas (N_2) had to be left until the temperature of the reactor was about 90°C.

Determination of chemical kinetic study

The pyrolysis kinetics was studied by using thermogrametric analyser at different heating rates (i.e., 10°C/min, 20°C/min, and 50°C/min). The devolatilisation reactions rate of the waste tire which is assumed to decompose as the sum of three main components decomposing individually (Carmo-Calado, 2020). The importance of estimating kinetic parameters activating energy (E_a) and Frequency factor (A). The data from mass-loss history curves are obtained from fundamental equations (Equation 3).

$$\ln\left[\frac{F(X)}{T^2}\right] = \ln\frac{AR}{\xi E} - \frac{E}{RT} \tag{3}$$

Using Coats and Redfern method to calculate the frequency factor and activation energy (Mkhize & Ben., 2019). This method has been used and supported by various researchers with satisfactory results (Tsamba et al., 2017). Similarly, if left hand side is plotted against (1/T) after linearizing equation (3) the slope will be proportional to activating energy of the sample. This approach of approximation has been used successfully by many researchers (Pan, Jiang, & Guo, 2021). Coats and Red-fern method for numerical determination of pyrolysis from TG analysis is given in Equation (4).

$$ln\left[\frac{-\,ln(1-X)^{1-n}-1}{(n-1)T^2}\right] = ln\left[\frac{AR}{\xi E}\left(1-\frac{2RT}{E}\right)\right] - \frac{E}{RT} \tag{4}$$

For materials when n ≠ 1

$$ln\left[\frac{-\,ln(1-X)}{T^2}\right] = ln\left[\frac{AR}{\xi E}\left(1-\frac{2RT}{E}\right)\right] - \frac{E}{RT} \tag{5}$$

Equation (5) gives a straight line of slope E/R and $\ln\frac{AR}{\xi E}$ its Y intercept when the left-hand side $\ln\left[\frac{F(X)}{T^2}\right]$ of the Equation is plotted against 1/T. The value, dx/dt could be traced from the TG curve.

Therefore, the kinetic parameters, i.e., the apparent activation energy E and the pre-exponential factor A, could be found from the expression given by Equation (15) above. The correct approach used in this study was to assume that every compositional component of tire rubber undergoes independent irreversible degradation of first order. The mathematical analysis that adopted Coats and Red-fern method while evaluating kinetic parameters separately was to locate three consecutive and remarkable phases namely (I) Oil, chemicals and other additives (II) Natural Rubber breakdown and (III) breakdown of the SBR and PBR.

RESULTS AND DISCUSSION

Proximate and ultimate analysis results

Proximate and ultimate analysis was used in the characterisation of the composition and properties of tire waste that yielded the results on calorific values, moisture content, volatile matter, ash content, fixed carbon and chemical composition.

Proximate analysis Results

The results were tabulated and summarised in Table 1 and 2. Table 1 shows the comparison of proximate analysis with other researchers, while Table 2 shows the comparison of proximate analysis of different brand of tyres from different countries. In this study the sample contained 62.16% mass of volatile, 37.87% char which produced 32.79% fixed carbon and 5.05% ash.

Table 1: Proximate analysis of scrap tires

Materials					**Source**
Moisture Contents	**Volatile matter**	**Fixed carbon**	**Ash**	**HHV (kJ/kg)**	
0.80	68.80	27.20	3.30	36,463.00	(Dai *et al.*, 2015)
1.40	66.10	27.50	5.00	33,400.00	(Ucar *et al.*, 2016)
1.72	61.61	22.66	14.01	-	(Juma *et al.*, 2013)
1.53	62.20	29.40	2.41	40,000.00	(Abdallah & Manzano-agugliaro, 2020)
0.70	65.00	29.70	4.60	35,750.00	(Frigo *et al.*, 2017)
1.31	62.16	31.54	5.05	39,354.00	This Study (UDSM)

Table 2: Proximate Analysis of the Different Tire Brands (Kyari *et al.*, 2005)

s/n	Tire Brand	Country of origin	Moisture contents	Ash	Volatile	Fixed Carbon
1	Dunlop	Japan	0.51	4.81	66.79	27.89
2	Courier	Italy	1.31	3.39	65.04	30.26
3	Goodyear	South Africa	0.57	4.98	67.96	26.49
4	Michelin	Italy	1.76	2.39	67.13	28.72
5	Continental	Great Britain	0.47	6.23	67.44	25.86

Ultimate analysis results

The same materials were investigated for elemental analysis and the results are shown in Table 3. The result of carbon content from this study is 85.88%, which agrees with the results of other researchers. Similar agreement was indicated for hydrogen and nitrogen content. However, Ucar et al. (2019) reported that oxygen content in tires to be 15.89%, while this study showed oxygen content of 2.22%.

Table 3: Elemental composition value

C (w %)	H (w %)	N (w %)	S (w %)	O (w %)	Ashes	Sources
85.88	7.72	0.4	1.48	2.22	2.94	This study (UDSM)
83.2	8.9	0.3	1.6	6	-	(Demirbas, 2015)
74.3	7.2	0.9	1.71	15.89	-	(Ucar *et al.*, 2019)
86.7	8.1	0.4	1.4	1.3	2.9	(González *et al.*, 2020)
82.8	7.6	0.5	1.3	4.5	3.3	(Dai *et al.*, 2018)
85.8	8	0.4	1	2.3	2.4	(Torretta, 2019)
86.7	6.9	0.3	1.9	1	3.3	(Mkhize & Ben., 2019)
81.24	7.36	0.49	1.99	8.92	-	(Juma *et al.*, 2016)

The Calorific study results

The calorific value was determined using bomb and the values were tabulated in Table 1. The values show that calorific value ranges between 33,400.00 and 40,000.00 kJ/kg of waste tire. This study shows the calorific value of waste tire was 39,354.00 kJ/kg.

Thermogravimetric analysis results

The kinetic parameters were estimated from information retrieved in TG and DTG experiments. Nevertheless, this instrument does not readily give individual constituent reactions (Koreňová et al., 2018), so the overall mass loss was considered. TG and DTG results were illustrated in Figure 1 and Figure 2 respectively. The total weight loss which is attributed to volatile matter is 64.8% and the remaining percent of the initial mass is char composed by carbon black and ash.

Figure 2 shows the derivative thermogravimetric analysis (DTG) curves of waste tire at different heating rates (10, 20 and 50 degrees C/min). The curves reveal the degradation range of the samples as being approximately 200-525°C.

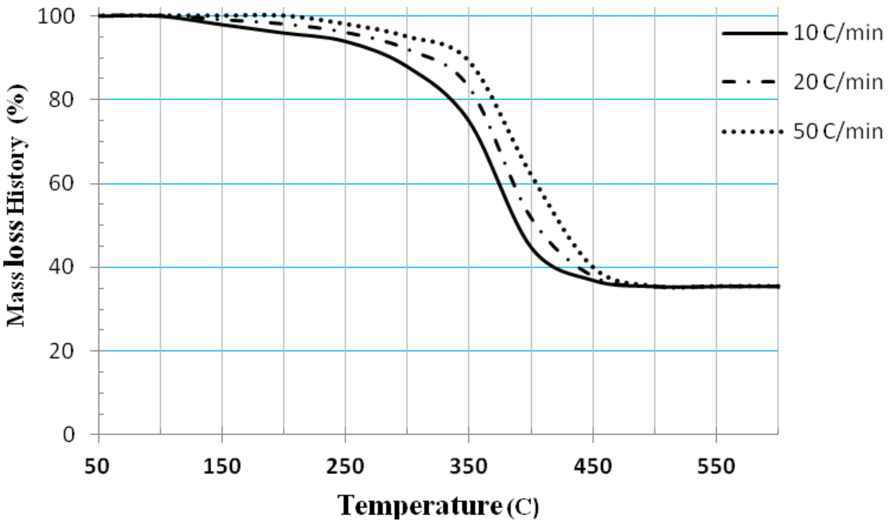

Figure 1: TG Curves.

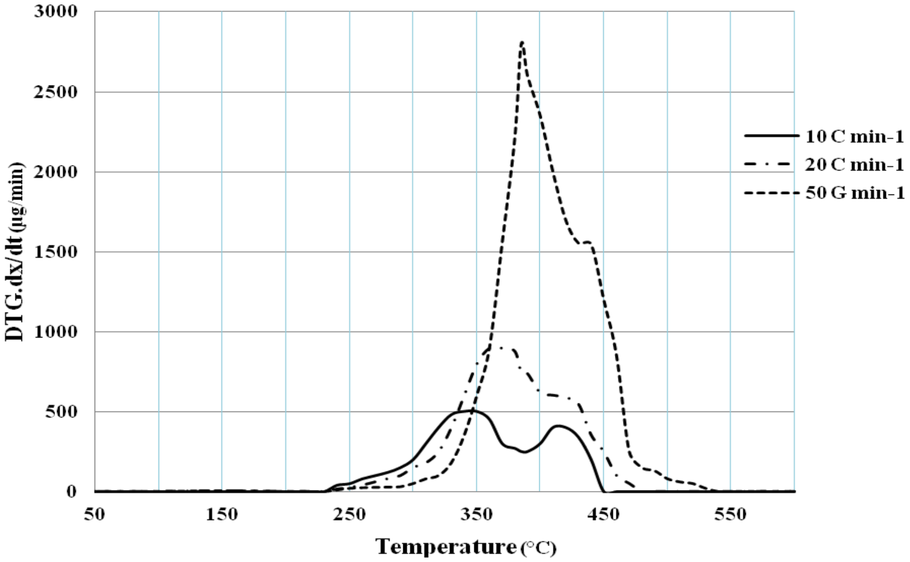

Figure 2: DTG Curve.

Decomposition time and heating rate

All three heating rates (10, 20 and 50 °C/min) from TGA were taken and compared. The results obtained from this analysis indicated that initial decomposition moves towards higher temperatures as the heating rate increases. An interesting phenomenon however, indicated that decomposition time taken for pyrolysis process completion was smaller the higher the heating rate. Which was under agreement of the study done by (Unapumnuk et al., 2015) shown in Figure 3.

In order for waste tire fast pyrolysis to perform well, it is necessary to immediately expose the materials to their optimal reaction temperatures. This reduces char deposition experienced when time is prolonged hence, avoiding secondary reaction due to vapour solid interaction. The reason for this occurrence is that when the heating rate increases the time taken to reach the maximum peak becomes smaller thus surpassed the early started slow heating rate. Therefore, the higher decomposition heating rate seems to lag behind.

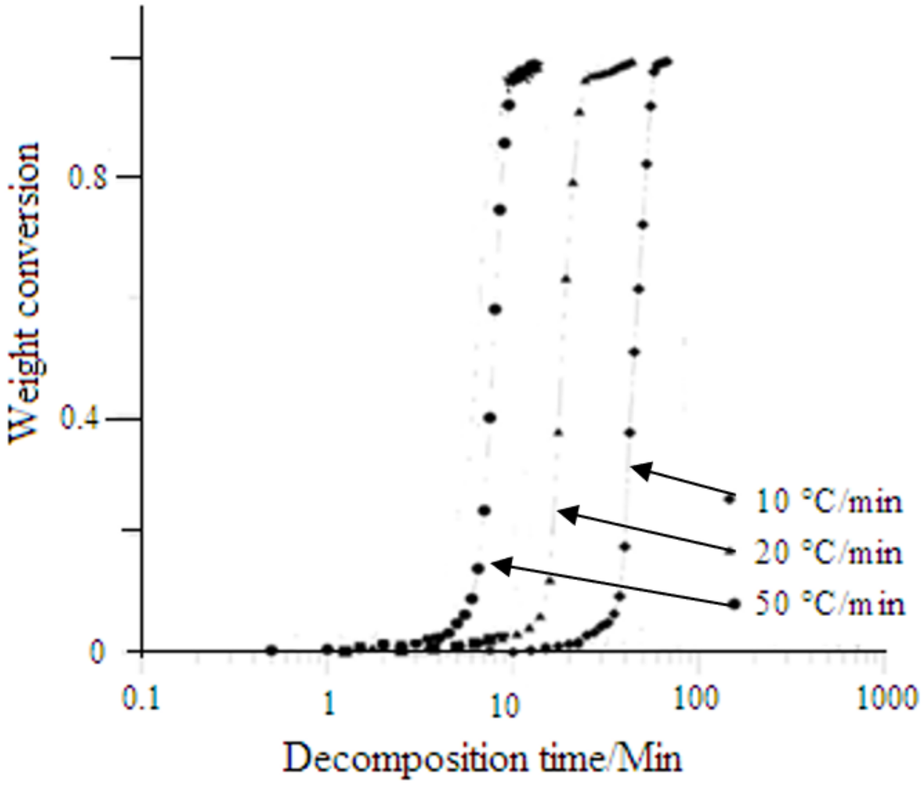

Figure 3: Decomposition time curve source: (Unapumnuk et al., 2015).

Chemical kinetic study results

A pyrolysis mechanism has variety of reactions, with some leading to the release of volatile matter, while others primarily result in alterations of mechanical properties (Unapumnuk, 2015). In the current work, the reaction causing weight loss has been the one considered in thermal gravimetric analysis. Since the determination of detailed components separately is complex, the reactions are taken as an overall reaction. Another problem pointed out by Seneca et al. (2019) arising from considering rubber components separately is that they are non-charring materials, they also found an increase in ultimate char yield 37 to 40% instead of 31% original content of carbon black filler. However, this study used constant heating rate as suggested by Heinzel et al. (2017) The value of activation energy obtained ranging from 11.27 kJ/mol. to 101.02 kJ/mol.

The variation of activation energy shows that the activation energy increases as heating rate increases as shown in Table 4. Even in small temperature range, the activating energy increases as the heating rate increases.

The values of kinetic parameters agreed with other researchers such as Haydary et al. (2018), but these values disagree to those obtained by Lah et al. (2020), who obtained 116 kJ/mol for NR and 113 kJ/mol for PBR at heating rate of 20°C/min,, while in this study the values of activation energy were 73.460 kJ/mol and 58.708 kJ/mol respectively. This variation is due to different methods used to estimate kinetic parameters.

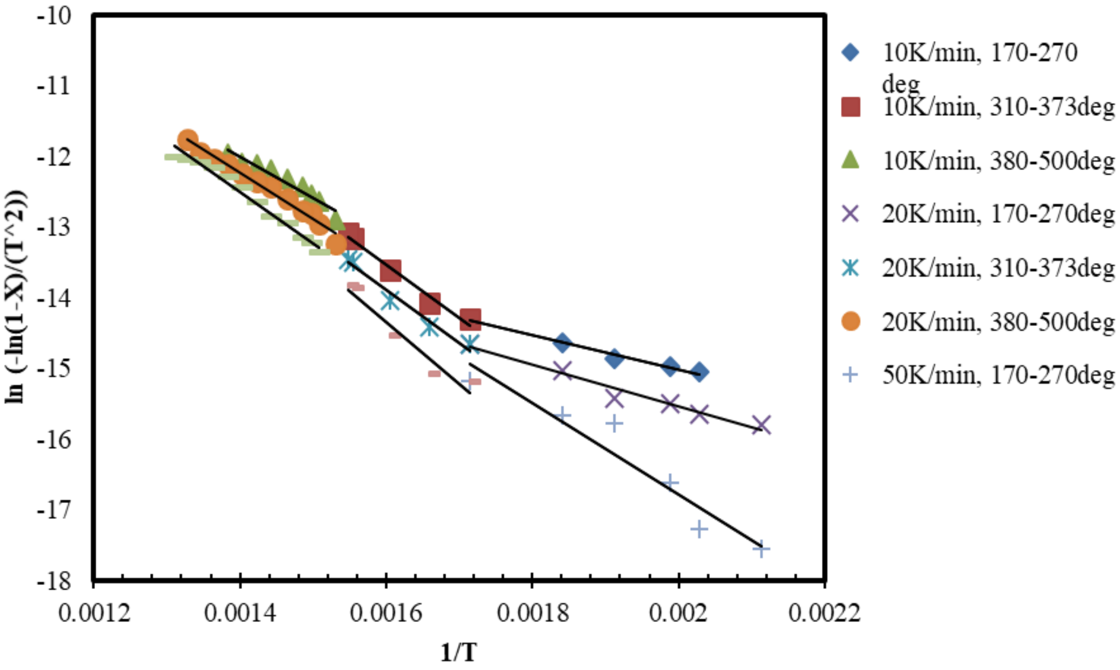

Figure 4: Determination of Kinetic Parameters; ξ = 10, 20 and 50oC/min.

Table 4: Summary of the results in determination of kinetics parameters

Material	Heating rate (°C/min^{-1})	Temp. Range (°C)	E (kJ/mol)	A (sec^{-1})
Extender oil and additives	10	150-300	11.268	1.098×10^{-3}
	20	150-330	20.332	2.0517×10^{-2}
	50	210-350	47.732	2.425×10^{1}
NR	10	300-370	62.157	2.610×10^{2}
	20	330-380	73.460	3.638×10^{3}
	50	350-390	101.015	1.507×10^{6}
PBR and SBR	10	370-450	51.348	2.873×10^{4}
	20	380-450	58.708	2.341×10^{2}
	50	390-500	62.481	8.893×10^{2}

The highest activating energy in this study is at temperature ranging from 300 to 390 °C. This is due to the devolitization of natural rubber (NR) and synthetic rubber (BR and SBR).

CONCLUSION AND RECOMMENDATION

The characteristics of waste tires obtained from Tanzania agreed with characteristics of other waste tires analysed by other researchers. During thermal decomposition three components were observed, these are additives and chemicals, which were obtained at low temperature, the second component was natural rubber and the third component was polybutadiene rubber which was obtained at higher temperature.

Kinetic properties showed that the activation energy of the additives ranges between 11 and 47 kJ/mol, natural rubber is between 62 and 101 kJ/mol, and polybutadiene is between 51 and 62 kJ/mol, these were determined at heating rate of 10, 20 and 50 °C/min.

Reaction kinetic knowledge obtained from tire rubber decomposition implies that the yield of products can be obtained even at lower temperature but the composition of the products depend heating rate applied.

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