Thermal Degradation Kinetics of Modified Fe_3O_4 With Poly(Vinyl Chloride) via Click Chemistry

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Abstract: The PVC-g-poly(POHMAC-co-VTM)-g-Fe_3O_4 and its precursors were characterized based on thermal properties comprising the initial decomposition and final decomposition temperatures and the maximum decomposition rate temperature were studied by TG/DTG and the decomposition steps were investigated. Then, the thermal characteristics of the final product formed via click chemistry, PVC-g-poly(POHMAC-co-VTM)-g-Fe_3O_4 were evaluated through thermogravimetric analysis (TGA) under argon atmosphere at 5, 10, and 15 °C/min. The studied temperature selection is 0 to 800 °C. Centered on the iso-conversional method, the thermal degradation kinetic parameter of the energy of activation was computed by the free model methods of the Flynn-Wall-Ozawa method (FWO) and Kissinger-Akahira-Sinuse method (KAS). The predicted activation energy values ranged from 41.40 to 117.83 KJ/mol meanwhile the average energy of activation values were computed as 85.75 KJ/mol and 72.31 KJ/mol respectively for the FWO method and KAS method. In addition, a saturation magnetization of 33.7 emu/g was recorded using vibration sample magnetometer (VSM).

Keywords: Click chemistry, Fe_3O_4, Kinetics, PVC, Thermal degradation.

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1. INTRODUCTION

The property known as the ability of a polymeric material to resist the action of heat and to maintain properties such as strength and elasticity at a given temperature is defined as the thermal stability of polymers (1, 2). The thermal stability of polymers is usually done by thermogravimetric analysis (TGA). In recent years, researchers have attempted to analyze polymeric materials by combining TGA with FTIR or mass spectrometry (3). In the case of polymeric materials, changes in mass can involve multiple steps. In general, the incorporation of the doping material into a polymer matrix increases the thermal stability of the pure polymer (4). The thermal stability of polymers varies depending on the degree of crystallinity, molecular weight, and chemical structure. While it is known that effects such as aromatic structures and cross-linking of polymers in the polymer chain improve the thermal stability of polymers, the presence of oxygen or double bonds in the main chain reduces the thermal stability (5, 6).

Currently, immense importance is being paid to the stability of the polymeric material with precise qualities of resistance to the high temperature called thermal stability. The thermal stability of polymeric materials can be determined based on initial, half, and final temperatures of decomposition via several thermal methods. Additionally, the stability of polymeric materials by thermal means can be substantiated by some kinetic parameters. Thermal degradation behavior of different types of polymeric materials has been extensively studied by thermo-gravimetric analysis (TGA), differential scanning calorimetry (DSC), differential thermal gravimetry (DTG), differential thermal analysis (DTA), thermo-mechanical analysis (TMA), and other techniques. Currently, thermal and thermo-oxidative degradation of polymeric materials, composites, and modified polymer materials has been studied by thermo-analytical methods (7-10). Moreover, methods of thermal analysis were applied to a great extent in analyzing the thermal aspect of polymers, composites, and polymers modified with nanoparticles and also, arrangements of complexes and polymer chelate compounds of transition metal ions (10-12).

It is accustomed to studying the thermal degradation of this modified polymer compound to get hold of valuable data on the polymer-magnetic nanoparticle bonds and stability course. TGA is the utmost prevalent technique for computing thermal decomposition owing to the speedy and remarkable facts which allow the thorough analysis of the mass fragments and the determination of the kinetic parameters (13, 14). Thermal degradation and kinetics of PVC have been discussed in detail (15-18). Thermal degradation and kinetics of PVC modified with different nanoparticles have been argued thoroughly. The degradation kinetics behavior of PVC and its blend with ZnO leads to the decline in activation energy of the PVC due to the physical impact of volatilization on the whole mass loss (19). Other works of literature include (20-22).
In this communication, we focused on thermal characteristics and kinetic studies of Fe_3O_4 modified with poly(vinyl chloride) via click chemistry and compared the thermal properties of the modified polymer and its precursors under the flow of argon at a heating rate of 10 °C/min. The kinetics from the results of the acquired TGA data at diverse heating rates under non-isothermal conditions were ascertained by the model-free methods, FWO and KAS. The activation energy values were computed via the aforementioned methods. The current work evaluated the kinetic parameter such as activation energy (E_a) and thermal characteristics from TGA/DTG data obtained. The results allowed us to study the thermal stability values for the modified polymer and its precursors (Figure 1).

2. METHODS

2.1. Synthesis and Technique

The novel PVC-modified Fe_3O_4 (PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4) and its precursors were synthesized, characterized, and published elsewhere (23). The pathway of the synthesis is shown in Figure 1. Thermal analysis was put through by TGA/DTG at a heating rate of 10°C/min under argon flow and the magnetic property was measured using the vibration sample magnetometer (VSM) at 300 K.

2.2. Kinetics Method

Non-Isothermal thermogravimetry analysis (TGA) of novel modified Fe_3O_4 was carried out by PerkinElmer instruments Pyris Diamond. TGA experiments were conducted from 0 to 800 °C at diverse heating rates of 5, 10, and 15 °C/min under an argon atmosphere. About 5 mg of samples were used for all the TG measurements. Figure 2 depicts the non-isothermal TG curves of novel PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4.

3. RESULTS AND DISCUSSION

3.1. Thermal Stability of Modified Magnetic Nanoparticle

The thermal degradation behavior of the modified magnetic nanoparticle was deduced and deliberated. The thermal degradation (TGA/DTG) curves of modified Fe_3O_4 were shown in Figure 2 and evaluated in Table 1. The initial decomposition temperature, percentage mass losses, and residue in the thermal decomposition of modified Fe_3O_4 have been examined through TG analysis at a heating rate of 10 °C/min under an atmosphere of argon. TG analyses were performed for weight loss in milligrams of modified Fe_3O_4 for raising the temperature. Peak, initial, and final decomposition temperatures of modified Fe_3O_4 were pinpointed by DTG investigation. The thermographs are depicted in Figure 2. The thermal stability properties of modified Fe_3O_4 were assessed by using TGA/DTG data over the temperature range 0-800 °C and outcomes obtained showed good thermal stabilities for the whole modified Fe_3O_4 compounds.

As seen from Figure 3, the thermograms of poly(POHMAC-*co*-VTM) show a single-step weight loss at a peak (T_{max}) of 424 °C while for poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 and PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4, both show two steps weight loss over a wide temperature range of 334 °C-413 °C and 334 °C-787 °C, respectively. The initial temperatures of decomposition (T_i) of poly(POHMAC-*co*-VTM), poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4, and PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 recorded from the thermograms are 266, 280 and 275 °C correspondingly which infers that adding of Fe_3O_4 to the copolymer augment the thermal stability of the copolymer meaningfully. Afterward the click reaction of azide PVC with the copolymer having Fe_3O_4, there was an infinitesimal drop in thermal stability which was foreseen to be an outcome of the materialization of a 1,2,3-triazole ring. At 330 °C, 20% of poly(POHMAC-*co*-VTM) have undergone thermal decomposition but poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 and PVC-*g*-Poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 reaches the same percentage decomposition respectively at a temperature of 770 °C and 795 °C. This implies that at a 20% decomposition state, PVC-*g*-Poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 is more stable than it is precursors. The % residue at T_f for poly(POHMAC-*co*-VTM) recorded is 21 which was lesser than that of poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4, indicating the bonding of the Fe_3O_4 to the copolymer. The 20% for PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 was most likely a result of a click reaction with azide PVC.

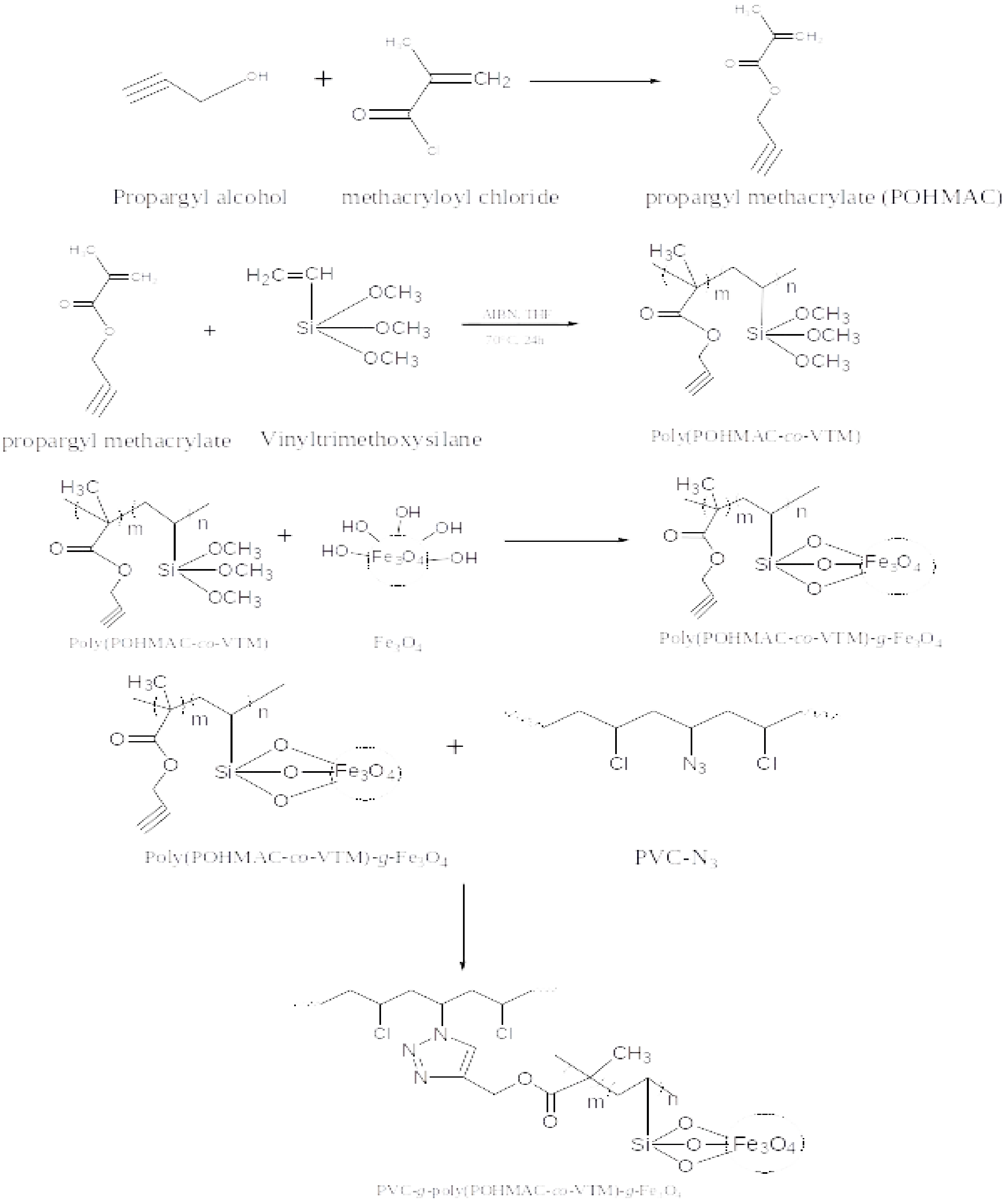

Figure 1: Synthetic pathway and structure of PVC-g-poly(POHMAC-co-VTM)-g-Fe_3O_4.

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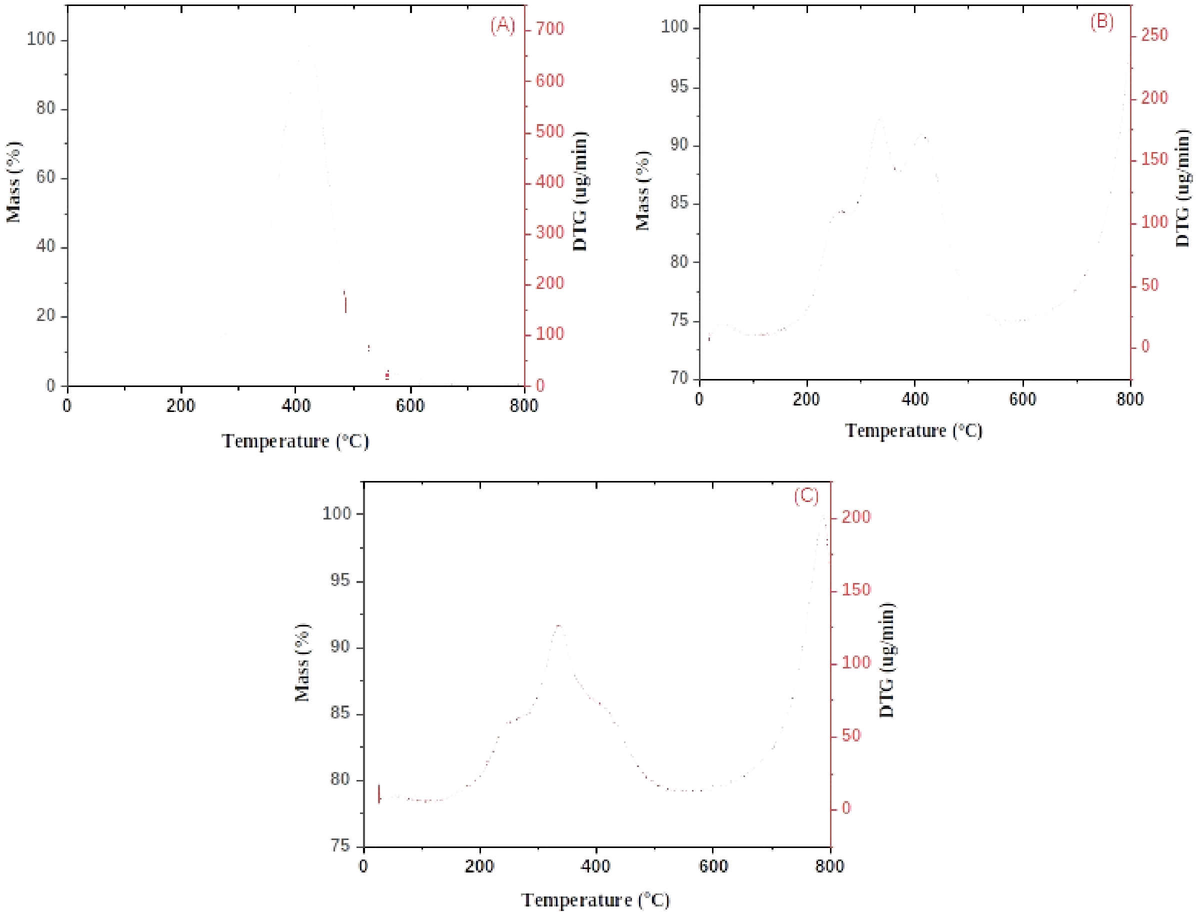

Figure 2: TGA/DTG of (A) poly(POHMAC-*co*-VTM) (B) poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 and (C) PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4.

Table 1: TGA/DTG evaluation of PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4.

Polymer	T_i (°C)	$T_{\%20}$	Residue (%) at T_f	Decomposition Stage	T_{max} (°C)
Poly(POHMAC-*co*-VTM)	266	330	21	1	424
Poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4	280	770	23	2	334, 413
PVC-*g*-Poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4	275	795	20	2	334,787

T_i Initial decomposition temperature
$T_{\%20}$ Temperature at 20% of decomposition
T_{max} Temperature at a maximum rate of decomposition
T_f final decomposition temperature

3.2. Thermal Decomposition Kinetics Based on Thermo-gravimetric Measurements

Non-isothermal thermo-gravimetric (TG) analyses of novel PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 formed as a result of click reaction were carried out by PerkinElmer instruments Pyris Diamond. The modified Fe_3O_4 was subjected to heating from 0 to 800 °C at a diverse heating rate of 5, 10, and 15 °C/min under an argon atmosphere. All the TG assessments were followed through with approximately 5 mg of the sample.

There exist methods, which can be detached into two various ways such as model-fitting and model-free for evaluating non-isothermal solid-state kinetic information from TGA analysis that has been effectively used for researching the kinetics of various substances (24). The model-free methods originated through the belief of reaction rate reliance on temperature and conversion degree simply devoid of creating whichever premise about the reaction function and reaction order evading the possibility of finding erroneous kinetic parameters. In other to examine the consequence of thermal activation on non-isothermal decomposition kinetics of novel PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 depending on thermo-gravimetric analysis, thirteen conversion values from 1 to 13% by way of an addition of 1% are practiced for the kinetic studies. Mainly, FWO and KAS analysis methods are extensively proven to be appropriate for model-free methods (25-28).

Various collections of kinetic information founded on thermal analysis and calorimetry (ICTAC) commendations have been documented in the literary works. One of the commendations was the report of α-temperature curves at diverse heating rates under an inert gas atmosphere (29). For this purpose, the TGA curve of novel PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 obtained under argon flow at diverse heating rates (5, 10, and 15 °C/min) was shown in Figure 3.

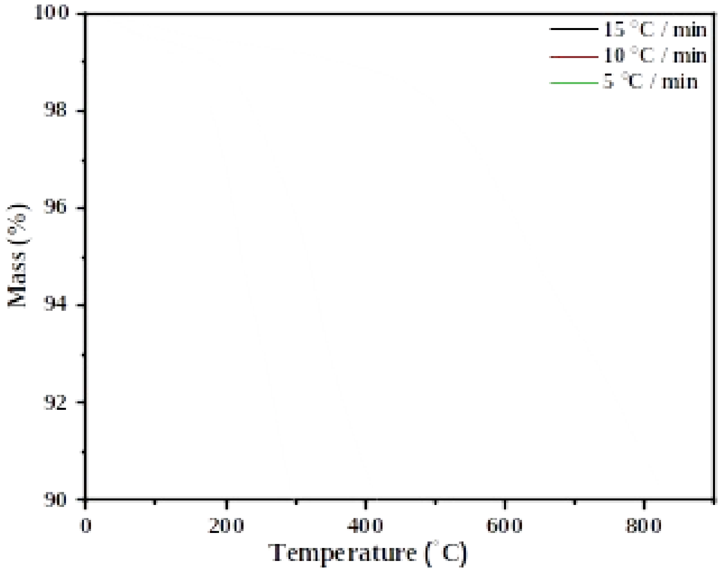

Figure 3: TGA curves of novel PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 obtained under argon flow at different heating rates (5, 10, and 15 °C/min).

The Flynn-Wall-Ozawa (FWO) method is gotten from the integral isoconversional way that Flynn, Wall (30), and Ozawa (31) suggested for the computation of the energy of activation by Doyle's approximation of the temperature integral (32) as shown in Equation (1). As for Kissinger-Akahira-Sunose (KAS) method, the Coats-Redfern approximation is used for the temperature integration (26) as presented in Equation (2).

$$\ln(\beta) = \ln\left(\frac{AEa}{Rg(\alpha)}\right) - 5.331 - 1.052\frac{Ea}{RT} \quad (1)$$

$$\ln\left(\frac{\beta}{T^2}\right) = \ln\left[\frac{AEa}{Rg(\alpha)}\right] - \frac{Ea}{RT} \quad (2)$$

The lines of best fit of ln(β) against 1/T for the FWO method and ln(β/T^2) against 1/T for the KAS method which is achieved conditional on thermograms determined at different heating rates, should be a linear graph whose slope can be practiced to calculate the energy of activation. In this research, the TGA was controlled under the temperature heating rates of 5, 10, and 15 °C/min, and the lines of best fit centered on the FWO and KAS methods are presented in Figure 4(a)(b) correspondingly. It can be noticed from these figures that the lines of best fit have a linear relationship designating the activation energy at the varied conversions following a distinct mechanism or unification of multiple reaction mechanisms. The slope, activation energy (E_a), and average E_a are listed in Table 2.

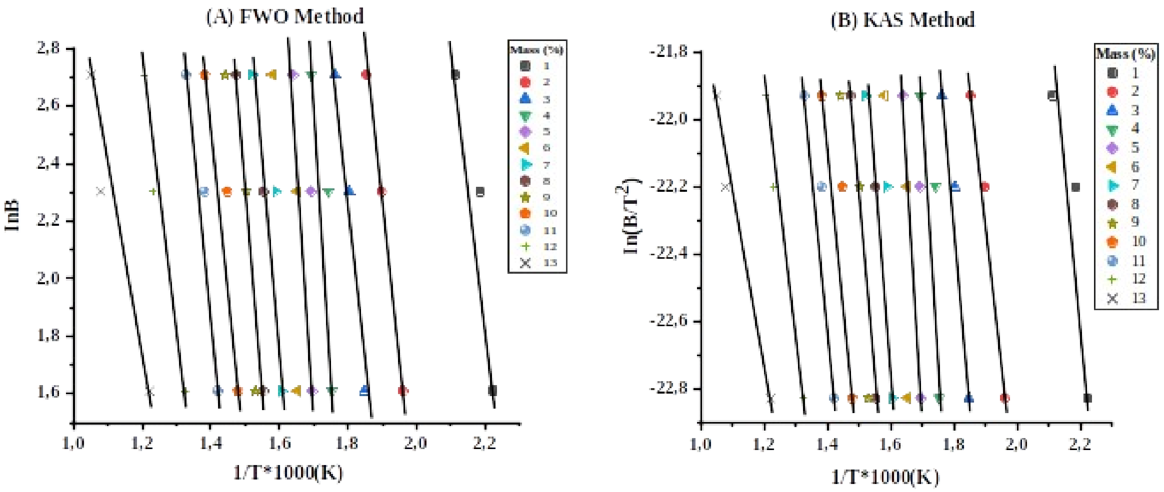

Figure 4: Regression lines to thermal decomposition of PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 based on (A) FWO method and (B) KAS method at 5, 10, and 15 °C/min.

As data demonstrated in Table 2, it is remarkable to notice there is not much major change in the activation energy achieved amid the two varied methods. It is verified that not only one mathematical method could be applied to evaluate the activation energy in as much as they are accurately chosen, and yet the value may differ to an assertive degree. It can be accessed that the activation energy for PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 thermal degradation is in the range of 41.40-117.83 KJ/mol with changeable conversion (1% ≤ α ≤ 13%). The average value is ascertained as 85.72 KJ/mol and 72.31 KJ/mol for FWO and KAS respectively. With the thermal degradation proceeding, the activation energy gets increased moderately and reaches its peak at 4% conversion for both methods. Thus far, as the conversion is repositioned from 3% to 4%, the equivalent activation energy is altered from 101.63 KJ/mol to 117.83 KJ/mol for the FWO method and 87.38 KJ/mol to 97.94 KJ/mol for KAS method, which is increased by 15.94% and 12.09% respectively. An increase in activation energy is noticed as the conversion is shifted from 6% to 7%, 8% to 9%, and 10% to 11%. All at once, the activation energy starts to drop when the conversion value surpasses 11%. At the conversion of 13%, the activation energies distinctly decline to 46.87 KJ/mol for FWO and 41.40 KJ/mol for the KAS method.

Table 2: Slope, E_a, and average E_a deduced from FWO and KAS methods.

α/%	FWO method		KAS method	
	Slope	**E_a/KJ/mol**	**Slope**	**E_a/KJ/mol**
1	9.41	74.37	7.58	63.02
2	10.32	81.56	8.48	70.50
3	12.86	101.63	10.51	87.38
4	14.91	117.83	11.78	97.94
5	14.11	111.51	11.01	91.54
6	10.49	82.90	8.16	67.84
7	11.04	87.25	8.75	72.75
8	9.64	76.19	7.48	62.19
9	11.49	90.81	9.21	76.57
10	10.58	83.61	8.49	70.59
11	11.56	91.36	9.36	77.82
12	8.71	68.84	7.27	60.44
13	5.93	46.87	4.98	41.40
Average		85.75		72.31

Furthermore, E_a-α curves of PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 obtained at diverse heating rates of 5, 10, and 15 °C/min under argon atmosphere have been shown in Figure 5. In this condition, the activation energies are found to be 74.37 KJ/mol for FWO and 63.02 KJ/mol for KAS at a conversion of 1%. The highest activation energy value is on the FWO curve at a conversion of 4%. There is also a corresponding increase in the activation energy after a slight decrease at conversion values of 7%, 9%, and 11% for both the FWO and KAS curves.

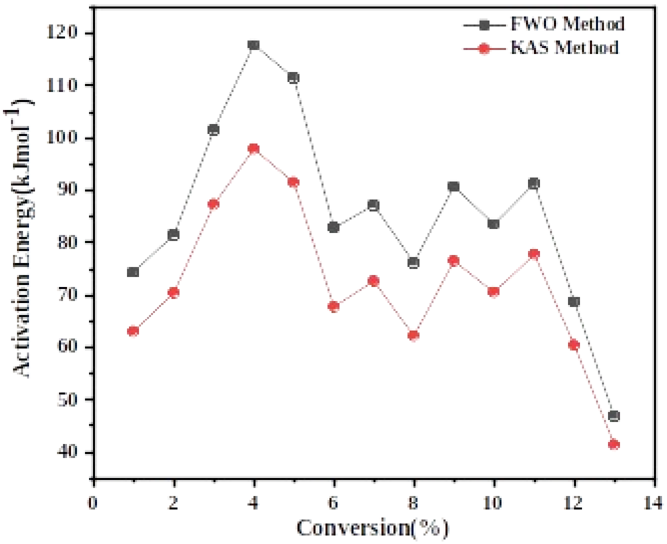

Figure 5: E_a-α curves of PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 were obtained at different heating rates of 5, 10, and 15 °C/min.

3.3. Magnetic Property

Figure 6 depicts the magnetization curve acquired from the vibration sample magnetometry (VSM) of novel PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 at 300 K for routine magnetic measurements (33). The VSM plot is vital evidence of the formation of PVC-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4. From the plot, the PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 displayed a saturation magnetization (M_s) of 33.72 emu/g which was less than saturation magnetization (M_s) values stated by various literature work of pure Fe_3O_4 nanoparticle. Several literary works were 53.24 emu/g (23), 65.0 emu/g (34), and 75.30 emu/g (35). The lesser value acquired for PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 than the given kinds of literature was purely due to the bonding of the PVC to the copolymer bearing Fe_3O_4. A study by Tukur et al. (36) has described that the modification of Fe_3O_4 with polyvinyl chloride via click chemistry shows a saturation magnetization value of 41.55 emu/g.

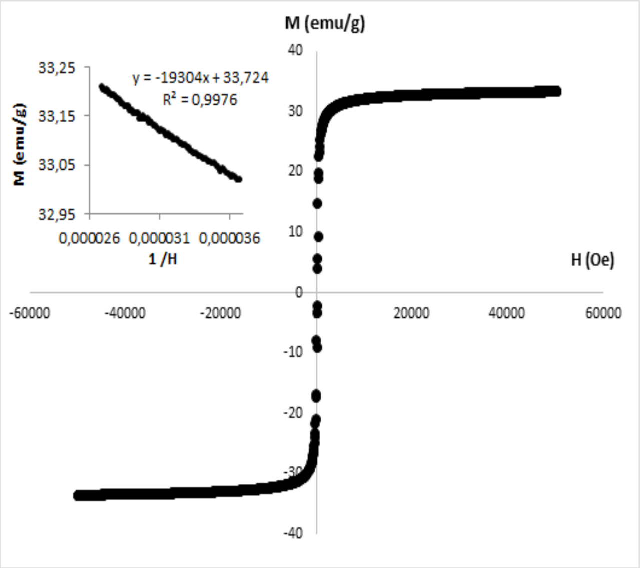

Figure 6. VSM plot of PVC-*g*-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4.

4. CONCLUSION

In this communication, a slight highlighting was put on the thermal characterization of novel PVC-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 and its precursors at a heating rate of 10 °C/min under argon flow. Based on TGA-DTG data and kinetics calculations were computed at several heating rates of 5, 10, and 15

°C/min for PVC-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 under the study. Two descriptive model-free approaches, FWO and KAS methods were designated to compute the activation energy and the average values inferred from the two methods are respectively 85.75 KJ/mol and 72.31 KJ/mol. The utmost activation energy was realized at 4% conversion. A saturation magnetization (M_s) value of 33.7 emu/g was recorded using vibration sample magnetometry (VSM). This research is anticipated to boost the basic data for industrial application of PVC-poly(POHMAC-*co*-VTM)-*g*-Fe_3O_4 thermal degradation.

5. CONFLICT OF INTEREST

The authors declare no conflict of interest.

6. ACKNOWLEDGMENTS

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