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Effect Of Zeolite On The Thermal Properties Of Conducting Polymer Polypyrrole

Emma Ziezie Mohd Tarmizi
Physics Unit, Centre of Foundation Studies For Agricultural Science, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia.
**E-mail: emma@upm.edu.my

Zainal Abidin Talib
Physics Department, Faculty of Science, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia.
E-mail: zainalat@upm.edu.my

Halimah Mohamed Kamari,
Physics Department, Faculty of Science, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia.
E-mail: halimahmk@upm.edu.my
**corresponding author

Ikhwan Syafiq Mohd Noor
Physics Unit, Centre of Foundation Studies For Agricultural Science, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia.
E-mail: imnoor@upm.edu.my

***Abstract*—This Polypyrrole and Polypyrrole/Zeolite conjugated system were successfully synthesized through the chemical oxidation polymerization method. The structural, morphological and thermal properties of the synthesized compound were characterized by X-ray diffraction (XRD) analysis, Field Emission Scanning Electron Microscope (FESEM), Thermogravimetry Analysis (TGA) and Laser Flash Measurement. XRD confirmed the amorphous characteristic peak structure of Polypyrrole conjugated system at two theta ~25^o. The incorporation of Zeolite resulted in the presence of sharp peak which implies that Polypyrrole/Zeolite conjugated system has some degree of crystallinity and more ordered arrangement than those Polypyrrole conjugated system. The FESEM morphology showed the formation of Polypyrrole and Polypyrrole/Zeolite conjugated system revealing a globular and coiled structure images. Thermal transport property of thermal diffusivity has been measured ranging from room temperature up to 420 K while thermal stability through thermogravitmetric analysis is measured from room temperature to 1273 K. The incorporation of Zeolite in conducting Polypyrrole shows an enhancement of thermal stability through laser flash measurement measurement and thermogravimetric analysis. As Zeolite content increases in the Polypyrrole/Zeolite samples, the conjugation length increased hence improved the thermal diffusivity and the thermal stability of the sample.**

Keywords—polypyrrole; zeolite; conducting polymer; conjugated system; XRD; FESEM; thermal

I. INTRODUCTION

Polyypyrrole (PPY) ranks among the most studied conducting polymers because of its high electrical conductivity, facile synthesis and good environmental stability under ambient conditions. Many papers have concentrated on the improvement of morphology and thermal properties of the Polypyrrole which includes carbon nanotubes, metal halides, chitosan etc but less attention is paid on Polypyrrole conjugated system based microporous material [1-6]. Incorporation of polymer system into microporous material is one of the recent and promising research directions in enhancing the thermal properties of a material. Zeolite is one of the promising host materials owing to their highly ordered pore systems of different dimensions and shapes and their surfaces is negatively charge-balanced with exchangeable cations [7]. Their porous structure is expected to protect polymer from degradation, improving the polymer alignment and decelerate aging of the system [8]. Moreover, Polypyrrole/Zeolite composites have the advantage of fast electronic mobility of Polypyrrole and the capability of Zeolite to incorporate cations into its structure with excellent adhesion [9-10].

The study of thermal properties of conducting polymer such as Polypyrrole is crucial for their use in practical applications. Their long-term thermal stability is very important and hence led to this work in investigating the influences of Zeolite on the thermal properties of Polypyrrole and Polypyrrole/Zeolite with different Zeolite content. Their morphological and structural characterizations were analyzed using X-ray diffraction (XRD) analysis and Field Emission Scanning Electron Microscopy (FESEM). The ability of Polypyrrole and Polypyrrole/Zeolite to transit heat was measured using Laser Flash technique ranging from room temperature until 420 K with interval of 20 K. The study of the thermal degradation has been used to determine the thermal stability of Polypyrrole and Polypyrrole/Zeolite and is mainly studied by TGA.

II. MATERIALS AND SYNTHESES

In this work, Polypyrrole and Polypyrrole/Zeolite conjugated system were prepared using chemical oxidation polymerization technique. Pyrrole (ACROSS) was used as the monomer for the synthesis of PPy. Prior to use, pyrrole was double distilled at a temperature of 131 ^oC at atmospheric pressure stored at 4 ^oC avoiding air. Iron (III) Chloride Hexahydrate ($FeCl_{3.}6H_2O$) (HAMBURG) is used as primary doping agent while commercial Zeolite (Fluka, product no. 96096) is introduced in conducting polymer Polypyrrole as secondary doping agent. Iron (III) Chloride Hexahydrate and Zeolite were used without any further purification except for Pyrrole. Distilled water was used in this work. Pyyrole of 0.2 mole concentrations was mixed with 0.2 mole $FeCl_3$ as primary doping agent together with Zeolite dissolved in water and stirred for 6 hours at room temperature. Zeolite was added to the Polypyrrole solutions in the proper quantity of 5, 10, 15 and 20% w/w content of Polypyrrole. The resulting powder were then filtered and washed thoroughly with distilled water to wash the adhered Fe^{3+} and then dried under vacuum at 50°C for 36 hours. These steps were repeated for various mole ratio of oxidant/Pyrrole. The sample was then ground into fine powder and press into pellet at 7 ton/cm² pressure using a Carver Hydraulic Presses and were polished by using fine 2000 grit sandpaper to make parallel surfaces and to remove any impurities.

III. EQUIPMENTS

The X-ray diffraction patterns of the samples in this work were recorded in a computer controlled X-ray diffractometer, (PANalytical X'Pert PRO PW3040 XRD System) with a conjunction of programmable control unit. CuK_α radiation source (λ= 1.5418Å) was generated at an accelerating potential of 40 kV and a tube current of 30 mA. The scanning was carried out for 2θ ranging from 5-40° at a step width of 0.03°.

For this analysis, scanning on a particular area was performed using FESEM (Model Nova NanoSEM 30 series and JEOL SM-7600F) with an accelerating voltage of 10kV. Magnification is adjustable depending on the study/analysis resolution requirement. For the cross-sectional scanning, edge-cracked pellets were positioned vertically using plasticine on a standard aluminium stub.

The thermal diffusivity value of 1 MR Polypyrrole and Polypyrrole conducting polymer with the presence of Zeolite from 5 to 20 % was obtained directly through Laser Flash apparatus model NETZSCH LFA 457 MicroFlash™ – 1100 °C version ranging from room temperature until 420 K with interval of 20 K. Thermal diffusivity α, thermal conductivity λ, can be obtained by measuring the thermal diffusivity α, specific heat C_p and density ρ as a function of temperature and the equation as follow:

$a(T) = \frac{\lambda(T)}{C_p(T).\rho(T)}$ (1) The thermogravimetric (TGA) analysis was performed in a flow of nitrogen by using TGS/SDTA 851^e (Mettler Toledo) thermal analyzer. A minute amount powder around 1-4 mg was placed in an alumina crucible and heated from room temperature to 1273 K with heating rate of 10K/min.

IV. RESULTS AND DISCUSSION

Morphological and structural Characterizations

The structure of Polypyrrole doped with various concentration levels of primary agent, iron (III) chloride hexahydrate, $FeCl_3$ and Zeolite have been analyzed by XRD technique in the 2θ range of 4 to 40°. Figure 1 shows the effect of Zeolite on 1 MR Polypyrrole. It is observed that one broad halo for all samples and it shows reflection maxima at around the 2θ value of 25°. The appearance of the broad halo in the region $15^o < 2\theta < 30^o$ represents a broad separation of polymer chain [11]. It like corresponds to highly disordered region and gives evidence that the samples formed in amorphous structure. Such broad peak usually indicates short range arrangement of chains [12]. In case for 1 MR Polypyrrole, the presence of primary doping agent shows no crystallinity or chain arrangement in these two conjugated systems. This agrees well with the structure reported in literature [13-16].

Alternatively, secondary doping agents of Zeolite has been impregnated in Polypyrrole conjugated system which is believed the presence of those additive can further enhances the thermal properties of the system by several orders of magnitude, perhaps. Figure 1 shows the XRD spectra of Zeolite in 1 MR Polypyrrole conjugated systems. In Figure 1, the presence of sharp peaks are observed in the conjugated systems which reveals that the polymer has some degree of crystallinity. As can be observed from the figure, pure Zeolite revealed more than 10 peaks in the spectrum. The XRD pattern of semicrystalline polymer of Polypyrrole/Zeolite showing superposition of crystalline reflections on an amorphous halo which appears at $2\theta = 25^o$ range in the XRD diffractogram of the Polypyrrole. Polymer exist both in crystalline and amorphous forms. The polymer chains are not aligned with each other over their whole length and the alignment is limited only to small crystalline region. A superposition peak of Zeolite which overlapped the broad halo indicating higher crystallinity of Polypyrrole/Zeolite compared to Polypyrrole. It is believed that the effect of higher cyrstallinity polymer is very important as the polymer tends to be much stiffer, harder and denser compared to amorphous polymer.

The morphology of the Polypyrrole and Polypyrrole/Zeolite conjugated systems were examined on the cross-sectional region of the specimens. The surface morphology of these specimens gives globular or nodule morphology similar to that bulk and film as reported in literature [1,6,17-18]. As can be seen in Figure 2(a), the bulk polymer tends to aggregate in the form of globules and coiled regularly to give rise to a three dimensional structure. The globules did not have any particular direction and appeared to be connected to each other at variant angles.

Field emission scanning electron micrograph (FESEM) revealed some interesting morphological difference between Polypyrrole and Polypyrrole/Zeolite.It has been found that Zeolite exhibits a dense and more compact morphology in Polypyrrole compare to solid Polypyrrole. The globular structure still can be seen distributed throughout the Zeolite external surface as seen in Figure 2(b). It is believed that the polymer composite are well dispersed inside the pore and on the external surface of the Zeolite which resulted in a more uniform composite structure. It can be concluded that Zeolite has served as a backbone improving the alignment of its chain provided by the cavity structure of Zeolite. This finding is in opposition with work reported by Enzel and Bein [19] and coworkers as in their work, they did not observe any significant amount of Polypyrrole forms on the external Zeolite surface [20]. This is expected to decelerate the aging, enhancing the mechanical properties and increase the electrical conductivity [8].

V. THERMAL PROPERTIES

Using the laser flash technique mentioned earlier, the influence of Zeolite on the ability to transit heat in Polypyrrole conjugated system is investigated. Figure 3 shows the variation of thermal diffusivity with temperature of 1 MR Polypyrrole and Polypyrrole/Zeolite with the presence of 5 to 20 % of Zeolite from 300 K to 420 K. The values of thermal diffusivity observed for all samples are of the expected values and falls in the same range available in literature [1,2, 8, 21-22]. All values in Figure 3 show that there is a common trend concerning the thermal diffusivity dependence with temperature. For each sample there are two temperature regions were observed where the first region denoted as T_1 which shows slight increase of thermal diffusivity with increasing temperature. Another region is marked as T_2, shows a fall-off of thermal diffusivity with increasing temperature.

It is observed that, thermal diffusivity of samples of 1 MR Polypyrrole and Polypyrrole/Zeolite with the presence of 5 to 20% of Zeolite, increases to specific maximum temperature of 380 K before started to decrease with further increase of temperature up to 420 K. Thermal diffusivity sensitively reflects the chemical, molecular and supramolecular structure of the polymer and is believed to have an effect and relation from several properties such as molecular weight, molecular orientation pressure and degree of crystallinity [23].

In conducting polymer, the diffusion of heat mechanism depends on the conjugation chain length in order to allow the free movement of electron. The heat energy is carried by electron which migrates along the polymer chain and then transferred from one chain to neighboring chain. Thus, the dopant plays important role on the conjugation length. In this work, it is found that the primary doping agent doesn't affect much the thermal diffusivity. The increment of thermal diffusivity can be seen obviously after the introduction of secondary doping agent that is Zeolite. It is believed that as Zeolite concentration is increased in Polypyrrole conjugated system, the conjugation length increased simultaneously and provides more through-space pathways for electron to migrate [24]. At this state, the electrons wander easily along the polymer chain and freely move to another chain. Hence, the rate of energy transfer is increased [2].

The transition temperature observed in this work is around 380 K which is common for most polymers and is known to have syndiotactic conformation. Syndiotactic is a polymer whose monomer units are oriented alternately dextro and levo or a polymer structure in which a monomer unit attached to the polymer backbone a-b-a-b fashion. In case of phonon that has been discussed above and will below, it characterized the process of transformation from longitudinal to transverse phonon [25]. The transition temperature observed in the work also could be related to the change of stiffness which has something to do with the change of conformation of internal rotation of skeletal bonds [26]. Under tensile stress the conformation is transferred into other conformation by internal rotation of skeletal bonds. This rotation which requires certain amount of energy is temperature independent as mentioned above. In this case of rotation a certain free volume must available to permit such displacement. The total free volume of the polymer increases with increasing temperature as a result of thermal expansion [27].

Crystallinity also is believed has a considerable effect on unsteady-state heat conduction. Largest molecular weight is believed to have higher degree of crystallinity. Although it is difficult to derive the relationship between thermal diffusivity and degree of crystallinity quantitatively, much believed it has a strong relation through work reported in literature [28]. Systems of higher thermal diffusivity values also is believed to have a stronger interaction of chain molecules compared to system with lower thermal diffusivity which is more rigid.

As the temperature rise, in the temperature region T_2, which is beyond transition temperature, the polymer change to rubbery through leathery state, gradually, individual units, atomic groups and small chain segments undergo intensive thermal motion and large torsional rotations, and sliding of chain segments starts to play dominant role in governing variation properties with temperature.The decrease of thermal diffusivity above transition temperature indicates a change in stiffness of the polymer. The mechanism of heat diffusion is depending on the medium state of the polymer itself [2]. As an addition, thermal diffusivity has been relates to Debye equation of:

$$\lambda = \frac{1}{3} C v l \quad (2)$$

Where λ is thermal conductivity, C is the specific heat capacity per unit volume, □ is the average phonon velocity, and l is the phonon mean free path. In Equation 1, specific heat capacity at constant temperature, C_p and density, δ is considered related to C in Equation 2. Thus, thermal diffusivity is expected to be influenced by υ. It is believed that above transition temperature, the distance of polymer become large and was considered to decrease the velocity of phonon (□) and this results in decreasing of thermal diffusivity [29].

From the study, it is observed that the introduction of Zeolite in Polypyrrole conjugated system has enhanced the thermal diffusivity of the conducting polymer. It is suggested that the increment of thermal diffusivity can be due to the interaction between the framework of Zeolite and Polypyrrole thus increases the phonon velocity. The phonon transport can be assumed to occur preferably through these materials due to higher number of phonon vibrational modes and the higher length of path in Zeolite compared to Polypyrrole conjugated system [1]. As mentioned earlier, crystallinity is believed to have impact on the value of thermal diffusivity. It is known that the introduction of Zeolite in Polypyrrole conjugated system have a higher crystallinity compared to Polypyrrole conjugated system which has been discussed in morphological and structural characterizations earlier. Higher crystallinity shows a larger value of thermal diffusivity compare to lower crystallinity material [23] which agreeable with the reported finding in this work.

VI. THERMAL STABILITY

Thermogravimetric analysis measurements were used to investigate the influence of Zeolite on the thermal degradation/decomposition and stability of the conducting polymers system, and the results are shown in Figure 4. Generally there are three-stages weight losses were observed in this work where each transition corresponds to endothermic decomposition. Zeolite was comparatively more stable and did not show dramatic decomposition until it reach 427 K and recorded only one stage weight loss total of 12.590 % between 293 K to 1173 K. In case of solid Polypyrrole 1 MR, the first significant weight loss of 5.416% already occurs at temperature 326 K. It is known that Polypyrrole is hygroscopic and at this first stage, the mechanism is characterized by the expulsion of absorbed water from the polymer samples, surface and physical (since it is difficult to remove the moisture in the samples completely) [30-31]. It is believed also at this stage, the weight loss starts eliminating bound water [32-33].

As Zeolite was impregnated inside the Polypyrrole, a relatively higher decomposition temperature is observed only up to 15% of Zeolite content. At higher intercalation, Zeolite of 20% in Polypyrrole has shown a slight shift to lower temperature but with a weight loss of 3.747 %. According to reported literature, sample that shows highest decomposition temperature indicating better thermal stability and may be due to longer conjugation length chain segments in Polypyrrole polymer units [34].

All the samples of Polypyrrole and Polypyrrole/Zeolite undergo second stage weight loss occurring between 404 K up to 663 K are believed due to loss of dopants in Polypyrrole and the degradation of host polymer, Polypyrrole [35]. Solid Polypyrrole of 1 M. As 5% and 10% of Zeolite were impregnated in Polypyrrole, the sample decomposed at higher temperature of 471 K and 558 K pronounced a weight loss of 9.104% and 10.508%. This definitely justified the enhancement of thermal stability of the system with the intercalation of Zeolite in host Polypyrrole. The intercalation of Zeolite beyond 10% recorded a major weight loss of 25.589% and 22.593% for 15% abd 20% of Zeolite intercalation in Polypyrrole conjugated system.

The third stage at about 664 K to 1173 K was characterized by breaking of the hydrocarbon chain, combustion of remaining organic species and is believed leaving only the relatively less volatile, metal oxide (Al, Si). It is also characterized by dehydroxylation and water loss aasociated with condensation of silanol group [6]. Prior to final stage of decomposition, only pristine Polypyrrole of 1 MR exhibits a major weight loss of 38.942%. This final stage shows a lower weight loss with 5% and 10% intercalation in host polymer compare to pristine Polypyrrole.

The observation has been made on the total weight loss of pristine Polypyrrole and Polypyrrole/Zeolite samples and it is found that the lowest weight loss is recorded by a Polypyrrole with 20% of Zeolite intercalation. Zeolite is believed has served as a backbone in Polypyrrole improving its alignment of its chain provided by the cavity structure of Zeolite. It is also deemed that with the presence of Zeolite, the conjugated system has been properly oriented and with a less contamination in sample, thermal stability of the sample has been improved.

VII. CONCLUSION

In this work, the effect of Zeolite on thermal properties of Polypyrrole conjugated system has been investigated. It is found that Polypyrrole/Zeolite conjugated system has some degree of crystallinity and more ordered arrangement than Polypyrrole conjugated system. A higher crystallinity result from introduction of Zeolite is belived has increased the conjugation length and providing more through-space pathways for electron to migrate and thus increased the thermal diffusivity. Zeolite is assumed has served as a backbone improving the alignment of polymer chain, introduce a more porosity and more compact morphology of the system and was found to improve the thermal stability of the Polypyrrole conjugated system.

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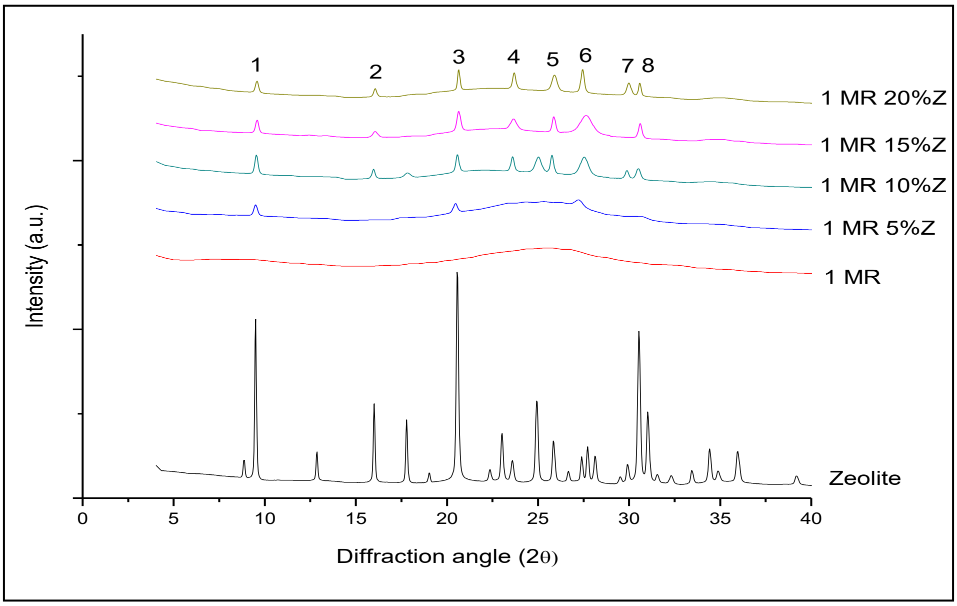

Fig. 1. *The effect of Zeolite on 1 MR Polypyrrole.*

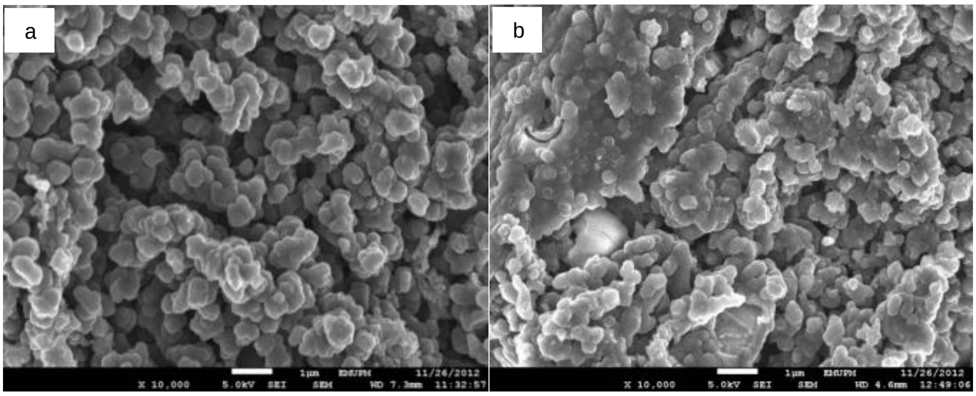

Fig. 2. *FESEM image of 1 MR Polypyrrole and 1 MR Polypyrrole with 20% of Zeolite intercalation (magnification 10 X).*

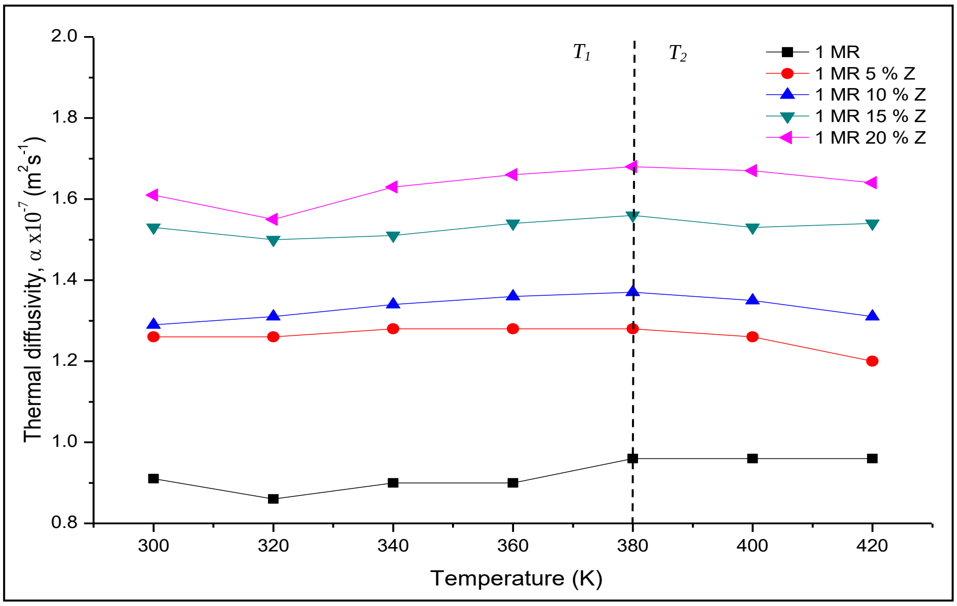

Fig. 3. *Thermal diffusivity of 1 MR Polypyrrole as a function of Zeolite concentration at different temperature.*

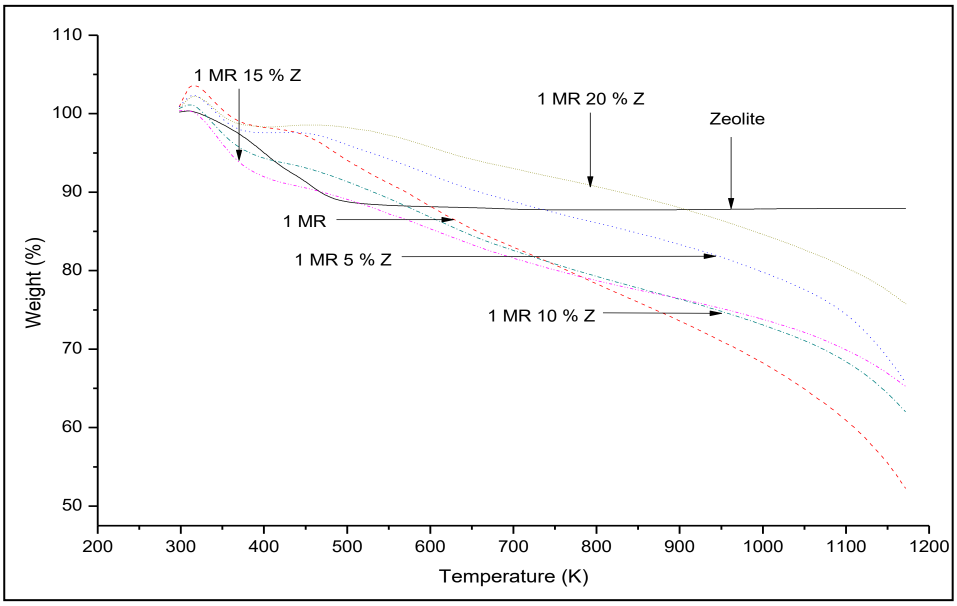

Fig. 4. *TGA thermogram of 1 MR Polypyrrole and Polypyrrole/Zeolite conjugated system.*