



## THERMAL PROPERTIES OF POLYIMIDE FILM AND POLYIMIDE-IRON COMPOSITE FILM

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**ABSTRACT:** *This study is on the thermogravimetric and the derivative weight loss of the Pi and Pi + Fe composite films. The weight loss resulted from the decomposition and burning. The peaks were observed and indicated the complete combustion of the Pi and Pi + Fe composite films. The prominent exothermic peak was observed at the Pi polyimide film at 550°C and the Pi + Fe composite film at 530°C, indicating the complete combustion of the films. The enhanced thermal stability of the polyimide synthesized with iron can be attributed to the inherent thermal stability of these materials and the robust chemical bonding interactions between the polyimide and iron.*

**KEYWORDS:** Thermogravimetric, composite films, derivative weight.



## INTRODUCTION

Polyamides are a type of high-performance polymer noted for their outstanding thermal and mechanical qualities. They have a distinct set of features, including high glass transition temperatures (TG), superior chemical resistance, low dielectric constants, and exceptional dimensional stability. Due to these properties, polyamides find applications in various industries, including aerospace, electronics, automotive, and microelectronics. Iron can alter the thermal breakdown behavior of polyimide composites. The addition of iron particles or iron oxide nanoparticles in the polyimide matrix has the potential to impact the composite's breakdown kinetics and thermal stability. Iron can operate as a catalyst, increasing degrading processes and boosting the development of char residues that increase the composite's flame retardancy. The particular effects of iron in polyimide composites vary depending on aspects such as iron particle concentration and dispersion, iron-polymer interaction, and thermal processing conditions.

Thermal degradation of polyamides refers to the breakdown of the polymer's structure and properties when exposed to elevated temperatures. Chain scission, cross-linking, oxidation, and volatilization are common degradation mechanisms. The extent and pace of thermal deterioration are determined by temperature, exposure period, environment, and the polyimide's chemical structure. Thermogravimetric analysis (TGA) is an effective method for determining a material's thermal behavior across a wide temperature range. This type of analysis is commonly used to assess the stability, purity, and temperature tolerance of various materials, including polymers, fuels, and fibers. As a result, TGA is useful for analyzing novel biogases materials and products and comparing them to the alternatives (biogases and non-biogases) that currently exist in the marketplace. Aromatic polyimide (PI) resins have gained popularity in the creation of carbon materials in recent years due to their potential in the fabrication of C-C composites [1,2], carbon films with high crystalline [3,4], and carbon molecular sieve films [5], as well as in other applications [6,7].

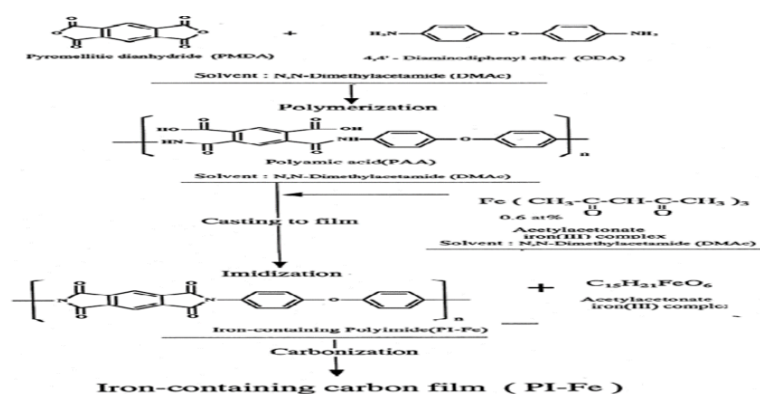
Transition metal particles in organic or carbon materials have a significant catalytic influence on carbonization and graphitization, making them useful for producing carbon films and annotating [8–10, 11–13]. Fine metal particles are known to have unique ferromagnetic characteristics, such as high coercive forces [14]. Carbon materials containing transition metal clusters are interesting not only from scientific aspects but also from technological points of view because of the retained properties of the fine metal clusters due to protective carbon coating. Simple processes for the preparation of aromatic polyimide (PI) films and their carbonization suggest an application to doping transition metals into carbon. Recently, we prepared a Kapton-type PI film by mixing an acetylacetonate iron (III) complex (AAFeIII) with polyamic acid in solution and by following imidization, and changes of the structure, texture and magnetic properties of the film by carbonization were investigated [23–25]. Fine iron particles with nano and submicron sizes were found to be homogeneously dispersed in the carbonized film, promoting the expansion and stacking of layers of matrix carbon. The magnetization changed from super paramagnetic to ferromagnetic when the film was carbonized due to the formation of iron particles, and a distinctive behavior of the magnetization due to tiny iron particles was observed. According to research on the carbonization of PI films with various chemical structures, the planarity and orientation of the repeating unit are significant in the formation of carbon films from polyimide. In theory, it possesses the highest planarity when compared to other polyimide systems and is expected to

produce high-performance carbon films.

We studied in the first research: The PI films used were synthesized from poly-biphenyl dianhydride (BPDA) and para-phenylenediamine (PDA). Therefore, the purpose of this study is to determine the influence of the structure and the catalyst effect on the carbonization behavior of the PI films with changes in the chemical structures. The PI films used in this work were synthesized from pyromellitic dianhydride (PMDA) and 4,4'-oxydianiline(ODA). In the present study, polyimide/Fe composite films were prepared from poly(amic acid) (PAA) based on PMDA and ODA with Iron (III) 2,4-pentanedionate (Fe(acac)) source via solution process. The prepared composite films were characterized using thermogravimetric analysis (TGA), (DTA). Thermal degradation and kinetic parameters, such as activation energy of thermal degradation processes, were also investigated through dynamic thermogravimetric analysis at different heating rates.

## PREPARATION OF PI-FE COMPOSITE

The synthesis procedure for the preparation of PI, PI-Fe composite films is shown in Fig. 1. In a typical process, PMDA was first dissolved into DMAc and then ODA was gradually added into the solution under an ice water bath to prepare PAA solution. This was followed by the introduction of a solution of Ferrum (III) acetylacetonate (AAI) in DMAc. The content of Ferrum, according to the previous literature<sup>[18]</sup> was chosen as 0.6 at % (atomic percentage) in the precursor mixture (PAA and Ferrum (III) acetylacetonate). After continuous stirring for at least 30 minutes, the mixture was cast on a clear glass plate. Then the composite film was heated in an air atmosphere at 80°C for 20 min and at 100°C, 200°C, 300°C, and 320°C, each for 1 h, yielding PI film containing Ferrum, during which thermally oxidized Fe<sup>3+</sup> ions transferred to Fe<sub>3</sub>O<sub>4</sub>. The thickness of the resultant composite film was approximately 40 μm and the color was dark brown. In contrast, pure polyimide film without Ferrum was also prepared.



**Figure 1:** The synthesis procedure for the preparation of carbon films derived from PI, PI-Fe.



## ANALYSIS OF THERMAL PROPERTIES OF POLYIMIDE / FE COMPOSITE FILMS

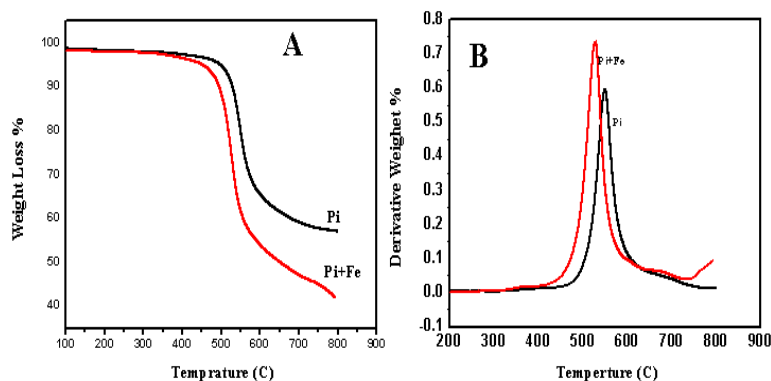
TGA and DTA will test the thermal stability of the prepared polyimide/iron compound films. TG curves display polyimide/iron composite films of varying iron content at a heating rate of 5°C/min as weight loss is due to the discharge of unstable gases during polymer backbone degradation. Fig. 2a shows the behavior of thermal degradation of PI films in the nitrogen environment. Clearly shown, the two PI films experienced a three-step weight loss in the temperature ranges of 50–500, 500–700, and 700–900°C. These three stages of weight loss appear to exemplify the chemical decomposition reaction, carbonization and removal of retained water. Additionally, these films obtained a char yield of over 40 wt% at the temperature above 800°C. It is not possible that the PI-Fe film showed an abundant weight loss than the PI pure film at temperatures lower than 900°C. This finding indicated that the PMDA / ODA film had better thermal stability and greater char performance than the (PMDA / ODA) + Fe film. The PMDA / ODA film is easier to decompose thermally at high temperatures because of the ether group with bonding capacity. Because of the high thermal stability of PI films from the TGA experiment, PI films were further treated thermally to gain carbon film from 600 to 1600°C with heating rate of 5°C/min in a high-purity N<sub>2</sub> environment. The carbon yields can be obtained.

As for the (PMDA / ODA)+Fe film, a larger weight loss occurs in a small temperature range of 500 to 700°C, resulting in a lower carbon yield, which means that the residual weight of the polyimide/iron composite film after thermal decomposition is higher than that of the polyimide film above 700°C. The increase in the weight of the remains of over 700°C shows the successful incorporation of greater amounts of iron in the polyimide/iron film composite and increases the ultimate in thermal stability. Fig. 2b shows the results of the thermogravimetric curve and the derivative weight loss curve of the Pi and Pi + Fe composite films. At the Pi + Fe composite film curve, a weight loss resulted from the decomposition and burning. The peaks were observed and indicated the complete combustion of the Pi and Pi + Fe composite films. The prominent exothermic peak was observed at the Pi polyimide film at 550°C and the PI + Fe composite film at 530°C indicating the complete combustion of the films. Improved thermal stability of the polyimide prepared with iron can be based on the fact that these materials have good thermal stability by nature and also because of the bonding interaction between strong chemicals between the polyimide and iron.

### TGA ANALYSIS

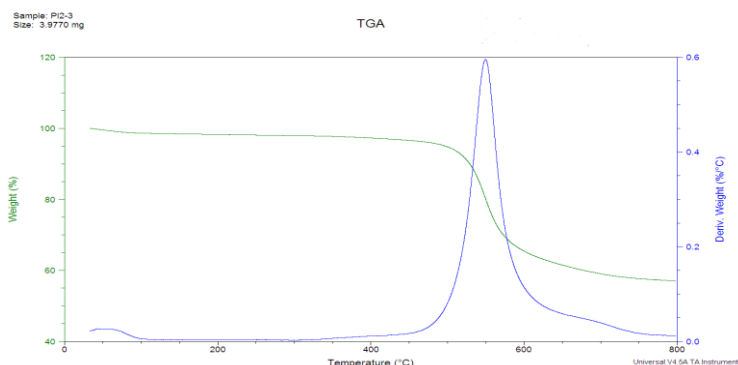
PI resin film changed obviously during pyrolysis: Its colour became bright black from transparent brown, and its texture became brittle. The TGA and DTG curves of PI resin film are shown in Figure 2. It indicated that a total weight loss of about 40% during pyrolysis in N<sub>2</sub> was observed. The weight loss during carbonization comprised three phases. The first was a slight weight loss before 530°C corresponding to the removal of retained water, which also indicated the good heat resistance of PI resin film. And the weight decreased continuously from 530 to 700°C, especially at 605.5°C because most chemical decomposition reactions of the material took place at this temperature. This step of weight loss is mostly due to the conversion of four carbonyl groups (C<sup>1</sup>/<sub>4</sub>O) in the imide part to ether CO or CO<sub>2</sub>. Above 700°C, weight loss began to decrease. That was because the volatilizing of CO and CO<sub>2</sub> tended to decrease, CH<sub>4</sub> and H<sub>2</sub> started to evolve above 605.5°C, and the total quantity of gas formed became

smaller.

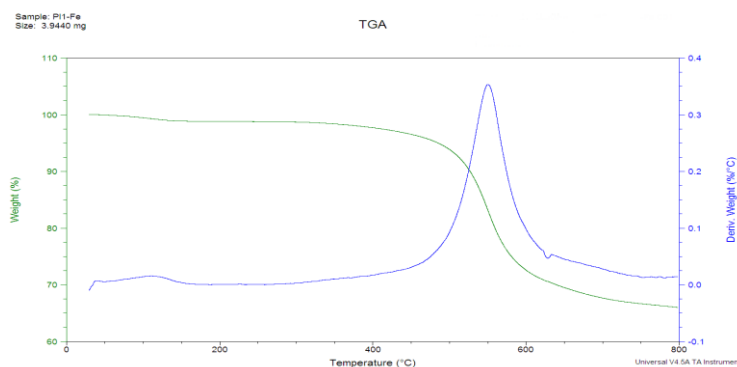


**Figure 2:** (a) TGA and (b) DTA thermograms of the polyimide/Fe composite films

The chemical rearrangement reactions and cyclization took place and a graphite-like structure was formed gradually in this phase. Until 800°C, the gas evolution was almost finished except for H<sub>2</sub>. Above 850°C, N<sub>2</sub> started to evolve.



**Figure 3:** (a) TGA and DTA thermograms of the polyimide film



**Figure 3:** (a) TGA and DTA thermograms of the polyimide film



## CONCLUSION

The results of the thermogravimetric curve and the derivative weight loss curve of the Pi and Pi + Fe composite films show that at the Pi + Fe composite film curve, a weight loss resulted from the decomposition and burning. The peaks were observed and indicated the complete combustion of the Pi and Pi + Fe composite films. The prominent exothermic peak was observed at the Pi polyimide film at 550°C and the PI + Fe composite film at 530°C, indicating the complete combustion of the films. Improved thermal stability of the polyimide prepared with iron can be based on the fact that these materials have good thermal stability by nature and also because of the bonding interaction between strong chemicals, between the polyimide and iron.

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