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# Study of the fire decomposition behavior of recycled polyethylene terephthalate (RPET) with the addition of MCA additive retardant

**G Prada Botia[1], C Acevedo Peñalosa[2] and S Orjuela Abril[3]**
[1] Grupo de Investigación GIINGPRO, Universidad Francisco de Paula Santander, San José de Cúcuta, Colombia
[2] Grupo de Investigación GIDIMA, Universidad Francisco de Paula Santander, San José de Cúcuta, Colombia
[3] Grupo de Investigación GEDES, Universidad Francisco de Paula Santander, San José de Cúcuta, Colombia

E-mail: gaudycarolinapb@ufps.edu.co

**Abstract**. Polyethylene terephthalate (PET) currently occupies an important space in the manufacture of bottles for carbonated beverages and mineral water, however, its use has become another pollution problem in the world, due to the fact that most of them end up in landfills, increasing the volume of plastics waste. The present investigation focuses on the analysis of recycled polyethylene terephthalate (RPET) analyzing whether it is possible to modify the thermal behavior of this plastic, by adding flame retardant, with the aim of improving its thermal properties. The thermal properties of the mixture and the degradation were evaluated by thermogravimetric analysis (TGA) and Dynamic Thermal Analysis (DTA), characterizing its decomposition process in increase of temperature, showing the little variation of the behavior of the mixture of RPET and % of MCA as a flame retardant additive compared to virgin PET.

## 1. Introduction
Polyethylene terephthalate, better known as PET, is one of the materials commonly used in the beverage bottling industry. These bottles occupy unnecessary spaces in landfills, have a very long life, and if they are not recycled, become pollutants [1]. Currently, PET has become a global environmental problem, so it has been decided to recycle it; however, there are few applications for less purified grades of recycled PET [2]. Recycling PET is to reintegrate this plastic into the production cycle as a raw material to manufacture various products such as packaging material, thermoforming sheets, plastic wood, pallets, and polyester fiber, among others [3]. The objective of this study is to obtain improvements in the thermal properties of RPET for the manufacture of electrical products.

In Colombia, on August 30, 2013, the Ministry of Mines and Energy issued the RETIE [4] Technical Regulation for Electrical Installations, which regulates the manufacture of electrical equipment and products, by means of Resolution 90708. The resolution requires the materials to be evaluated by Glow-Wire Flammability Index (GWFI) test method.

Similarly, the Colombian Technical Standard NTC 5534 of 2015 [5], defines the test for by Glow-Wire Flammability Index for materials (GWFI), determining the maximum temperature at which the tested material will not ignite or, if it does, the flames extinguish 30 seconds after removing the incandescent wire and have not been fully consumed; for this analysis a sample of RPET obtained from beverage bottles in the city of Cúcuta with flame-retardant additives was used.

The design of modified polymers with some additives is an alternative for improving their properties, in this case the use of flame retardant additives is an innovation for the industrial sector of the new materials [6]. The flame retardant concept, also called flame retardants, applies to a variety of compounds or mixtures of chemical compounds incorporated in plastics, textiles, electronic circuits, etc. designed to reduce the flammability of a material or to delay the propagation of flames along and across its surface. Flame retardants are responsible for generating a change in the pyrolysis reaction in polymers to reduce their accelerated combustion and prevent or reduce the level of risk these materials pose when exposed to flame [7].

The thermogravimetric analysis records the loss of weight of a sample as the temperature increases, up to temperatures of 1200ºC, under controlled conditions of heating speed and different reaction atmospheres; obtaining in this way the curves called TG or thermographs and the curves of differential thermogravimetric analysis (DTG) [8].

In this work RPET was mixed with MCA as a flame retardant in order to minimize the possibility of taking flame to this material being exposed to high temperature. The objective of the research was to evaluate the effect of flame retardant on the thermal properties of RPET. Thermal degradation was evaluated by thermogravimetric analysis (TGA). The aim of this work is to contribute to the knowledge of PET waste recovery by adding additives that control the effects of exposure to high temperatures of recycled plastic without impairing its thermal properties, and thus contribute to the innovation of materials and the recycling of plastics.

## 2. Methodology

The thermal characteristics of RPET with a flame retardant additive were evaluated, analyzing its behavior when it is subjected to temperature increases by means of laboratory tests that identify the values that form its thermal history such as melting temperatures ($T_m$), degradation temperatures, glass transition temperatures ($T_g$). To determine the change in thermal behavior, TGA and DTA tests were performed. These were carried out using the NETZSCH STA 409 PC/PG analyzer, taking simultaneous measurements of Differential Thermal Analysis (DTA) and Thermogravimetric Analysis (TGA) to the mixture of RPET and a flame retardant.

### *2.1. Data collection*

*2.1.1. General properties of virgin PET polyethylene terephthalate*. It is theoretically determined that PET Polyethylene Terephthalate exhibits the following general properties [9] listed in Table 1.

**Table 1.** General properties of Polyethylene Terephthalate (PET).

| Properties of PET | |
|---|---|
| $T_g$ (°C) | 80 |
| $T_m$ (°C) | 265 |
| LOI (%) | 21 |
| Tg | 420 |
| Density (g/cm³) | 1.37 |
| Thermal conductivity (W/m*K) | 0.24 |

The melting temperature of Polyethylene Terephthalate is 265°C one of the highest in relation to other recyclable thermoplastic polymers, followed by Polystyrene at 235°C, Polyvinyl chloride at 205°C and Polypropylene at 160°C [9]. The RPET in turn has a melting temperature of 252.1°C and its degradation showing loss of mass at 380°C [10].

Since the objective is to possibly use this material for the manufacture of electrical materials and these require high fire resistance, it is concluded that PET is a good choice. As shown in Table 1, PET has a LOI of 0.21 which classifies it as a moderately combustible material. With the addition of flame retardant additives, the thermal properties of RPET to the combustion reaction are expected to remain stable.

*2.1.2. Selection of flame retardant compound*. There are more than 175 different types of flame retardants, which are generally divided into classes including organic halogenated (usually brominated or chlorinated), phosphorous-containing, nitrogen-containing, and inorganic flame retardants. The most environmentally friendly are the containing nitrogen [11]. For the present investigation, retardant additives were used due to the fact that they were worked with a thermoplastic material and are the ones that present greater compatibility with this type of plastic. The alternatives offered by the industry in terms of flame retardants were evaluated and it was determined that Melamine Cyanurate (MCA) was used, which is a nitrogen-based retardant additive, its main characteristics are low solid state toxicity, in case of fire no dioxins or toxic substances are produced and little smoke generation. It belongs to the group of melamine salts, it is marketed as Budit 314/ Budit 315 [12]. Through the supplier of the retardant, a GWIT glow-wire flammability test was performed at 650°C and a GWFI at 920°C of Budit 315 with a sample of RPET recovered in the city of Cucuta, which determine the ignition resistance of the plastics, defining percentages of the most optimal mixture of 85% RPET plus 15% Budit 315 (MCA). With these values of the mixture the TGA and DTA were realized.

## 3. Results

The TGA was carried out taking into account that the sample analyzed was a mixture of 85% Recycled Polyethylene Terephthalate (RPET) plus 15% Melamine Cyanurate, with a heating rate of 5°C/min, from 25°C to 700°C and in an air atmosphere of 60mL/min. First, the components of the mixture were analyzed separately, obtaining results observed in Figure 1 for the RPET. In zone A RPET loses 1.7% mass due to the release of volatile substances, water, gaseous products from material decomposition, plasticizer.

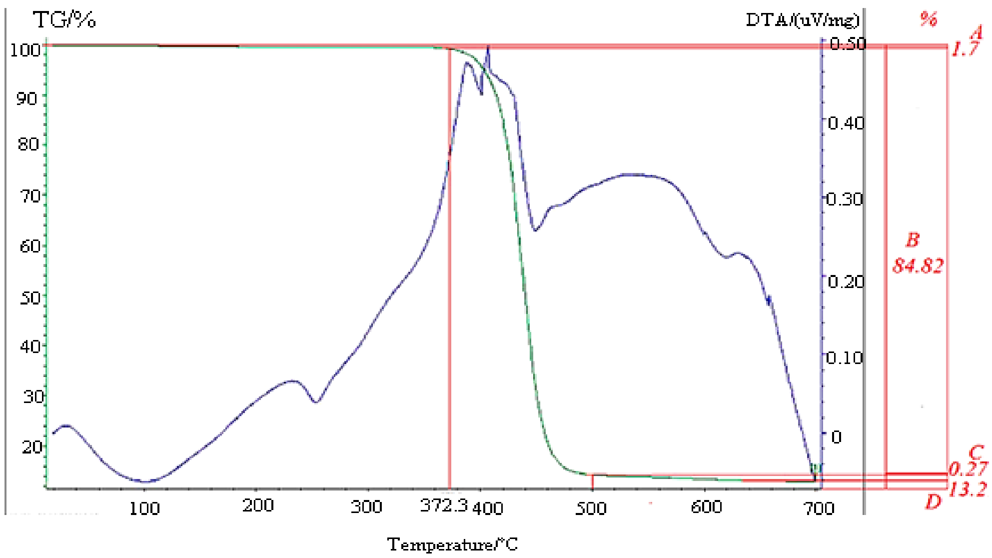


**Figure 1.** TGA diagram for the RPET.

Likewise, in zone B, thermal degradation of 84.82% of the RPET between 372.3°C and 500°C occurs. Finally, in zone C the combustion of the organic fraction takes place. In zone D, 13.21% of inorganic loads are present, which remain as waste after the degradation of the plastic, since, in general, they resist high temperatures.

Finally, it is concluded that the temperature at which RPET degradation begins is 372.3°C.

Similarly, TGA was performed for the fire retardant additive with the results shown in Figure 2. In zone A, Melamine Cyanurate loses 1.6% mass due to the dissociation of the Melamine salt and hydrocyanic acid in an endothermic way, which consumes the heat of the combustion preventing it from continuing and spreading.

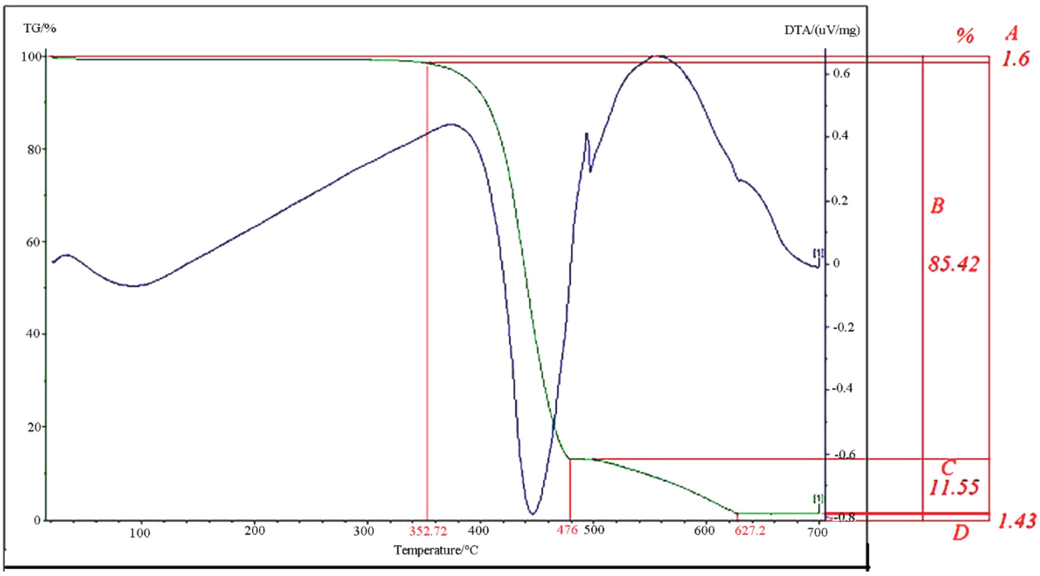


**Figure 2.** TGA diagram for melamine cyanurate.

While in zone B, thermal degradation of 85.42% of melamine cyanurate occurs between 352.72°C and 476°C. In this area there is an endothermic sublimation of melamine which aims to reduce the heat available for the combustion reaction. Finally, in zone C, 11.55% combustion occurs as a result of the decomposition of the melamine vapors in an endothermic process. In zone D, 1.43% of inorganic loads are observed as a result of degradation.

Figure 3 shows the endothermic reactions of Melamine Cyanurate from 360°C to 500°C. The temperature at which degradation of Melamine Cyanurate begins is 352.72°C. Table 2 shows the % loss in mass of the mixture during the TGA test.

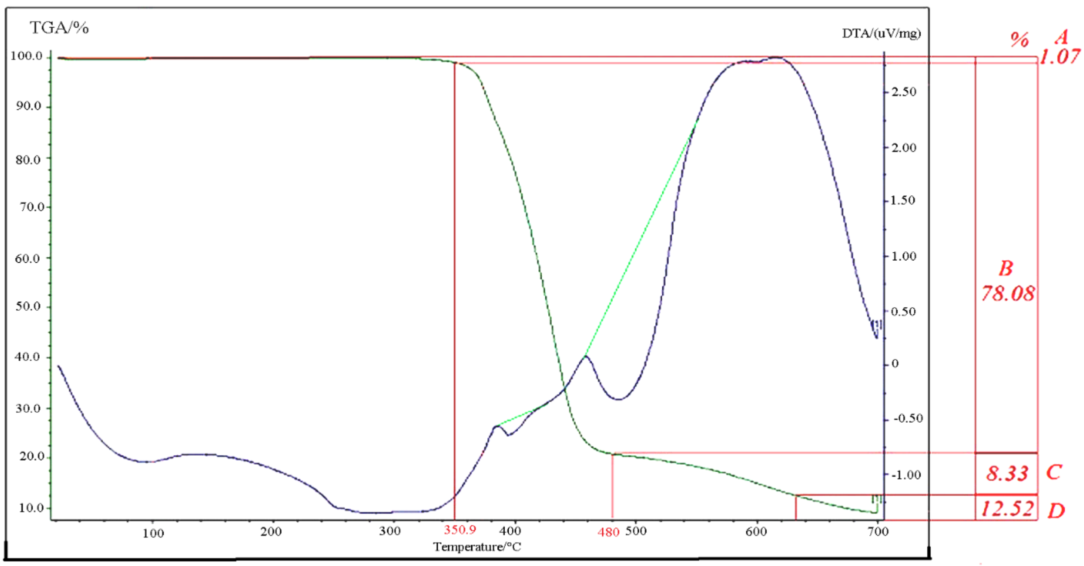


**Figure 3.** TGA diagram for 85% RPET plus 15% additive and DMA mixture.

It is observed that in zone A of Figure 3 the mixture loses 1.6% mass, it is expected that in this zone the dissociation of the salt of Melamine and the hydrocyanic acid begins in an endothermic way so that it consumes the heat of the combustion preventing it from continuing and spreading. In addition, the polymer must lose volatile substances and water. In zone B, thermal degradation of 78.08% of the mixture begins between 352.72°C and 480°C. In this area there is an endothermic sublimation of melamine which aims to reduce the heat available for the combustion reaction. Finally, in zone C, 8.33% of the combustion is produced as a result of the decomposition of the Melamine vapors in an endothermic process. In zone D, the presence of 12.52% of inorganic loads that remain as waste after degradation is observed, this zone goes from 630°C to 700°C. In turn, Table 2 shows that the mixture begins to degrade at 380°C where significant weight loss is identified.

**Table 2.** % Mass loss of the mixture per TGA.

| Temperature (°C) | % Mass | % Mass Loss | Temperature (°C) | % Mass | % Mass Loss |
|---|---|---|---|---|---|
| 347 | 99.06 | 0.94 | 425 | 52.50 | 47.50 |
| 350 | 98.93 | 1.07 | 427 | 49.63 | 50.37 |
| 352 | 98.80 | 1.20 | 430 | 46.68 | 53.32 |
| 377 | 91.42 | 8.58 | 442 | 32.29 | 67.71 |
| 380 | 89.83 | 10.17 | 445 | 29.97 | 70.03 |
| 382 | 88.34 | 11.16 | 447 | 28.12 | 71.88 |
| 395 | 80.59 | 19.41 | 507 | 20.03 | 79.97 |
| 397 | 78.83 | 21.17 | 510 | 19.96 | 80.04 |
| 400 | 76.93 | 23.07 | 512 | 19.85 | 80.15 |
| 407 | 70.48 | 29.52 | 627 | 12.90 | 87.10 |
| 410 | 68.41 | 31.89 | 630 | 12.66 | 87.54 |
| 412 | 65.69 | 34.31 | 632 | 12.62 | 87.48 |

## 4. Conclusions

Polyethylene Terephthalate PET has the highest LOI oxygen limit index among thermoplastics, adding its high strength and its melting point is the highest of thermoplastics, making it a viable option for use as a material for the manufacture of electrical products.

The percentage used to evaluate RPET under the action of fire with the incandescent wire test was 15% MCA and 85% recycled polymer, this percentage of mixture gave acceptable thermal results that were evidenced in the TGA test.

The Polyethylene Terephthalate RPET recovered in the city of Cúcuta, has a degradation temperature of 372.3°C and presents 1.7% of volatile compounds and 13.21% of inorganic loads.

Melamine Cyanurate presents degradation reactions of endothermic nature in a temperature range of 352.72°C to 500°C that confirm its nature as a flame retardant additive.

The mixture begins its degradation at 350.9°C, indicating the beginning of the endothermic degradation of the melamine and the decomposition of the retardant vapors. The DTA diagram shows endothermic reactions in the range 380°C to 550°C, confirming the retarding nature of the additive selected for the manufacture of electrical products.

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