

# Study of the Conversion of Postconsumer Polystyrene on CeO<sub>2</sub>/HZSM-5 Type Materials

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## Abstract

The catalytic conversion of polystyrene (PS) was studied in the presence of the materials type HZSM-5, CeO<sub>2</sub>, 10% CeO<sub>2</sub>/HZSM-5 and 20% CeO<sub>2</sub>/HZSM-5, which were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD) and nitrogen adsorption at 77K. The catalytic tests were performed via thermogravimetric analysis (TG) at heating rates of 5, 10 and 20°C min<sup>-1</sup> in a temperature range from 30°C to 900°C. For the tests, a ratio of 30% by mass of each catalytic material mixed with PS was used and the activation energy of the degradation process was determined by the Vyazovkin method. The obtained results showed that the addition of the catalyst to the PS in general reduced its degradation temperature. The 10% CeO<sub>2</sub>/HZSM-5 catalyst showed greater efficiency, as it resulted in lower activation energy for PS degradation. Thus, the combination of CeO<sub>2</sub> with HZSM-5 resulted in materials with potential for application in the catalytic degradation of polystyrene and the results indicate that the production of a composite material can be a good strategy to generate an increase in catalytic activity and a decrease in energy process activation.

## Keywords

HZSM-5 Zeolite, Cerium Oxide, Polystyrene, Pyrolysis

## 1. Introduction

Plastic was created in the 20th century and is currently one of the most used materials on the global market, due to its low cost and good structural properties. Currently, many advances have been made in plastic recycling and these processes facilitate the development of products that are beneficial to society in many ways [1]. With the exponential growth of the world population comes the

need for new technologies and development. Plastic production has been growing as these new demands emerge [1]. According to a report released by the Brazilian Association of Plastic Industries (ABIPLAST), in 2020, 367 million tons of thermoplastic resins and 373 million tons of processed plastics were produced worldwide, of which 3.9% is produced in Brazil [2]. Polystyrene, obtained through the polymerization of styrene (vinyl benzene), can be found in the most diverse industries and segments, such as cosmetics, food, household appliances, among others. Its wide range of applicability is due to its properties, which add values such as malleability, resistance to temperature variations and its ease of molding [3].

The main thermoplastic resins consumed in Brazil in 2020 were Polypropylene (PP), polyvinyl chloride (PVC), high-density polyethylene (HDPE), linear low-density polyethylene (LDPE), polystyrene (PS) and expanded polystyrene (PSE). Polystyrene corresponds to 7.5% of the amount of thermoplastic resin consumed in Brazil in 2020 [2]. The plastic generated has negatively impacted the environment and human health. In 2010, the National Solid Waste Policy (PNRS) came into force in Brazil, established by Law 12,305, which establishes strategies for sustainable development. Strategies to reduce the disposal of plastic waste in landfills and landfills are recommended by the PNRS [1].

According to ABIPLAST, only 23.1% of the plastic material produced was recycled in Brazil in 2020 [2]. Mechanical recycling is a viable alternative for reusing plastic, it consists of converting plastic waste into granules that can be reused in the production of other products. However, recycling becomes more complex and costly, due to the difficult and expensive separation of different types of plastics and the high degree of contamination of organic matter in solid waste [3]. Another recycling option is chemical, carried out through several chemical reactions, including the depolymerization of plastic [4] [5]. Through the thermal degradation process, it is possible to reduce the amount of plastic waste discarded into the environment. However, thermal degradation requires high temperatures and the quality of the products obtained is still reduced, which ends up limiting the viability of the process. One solution is to improve the process with the presence of appropriate catalysts, such as zeolites, thus reducing the polymer degradation temperature [6].

For the catalytic degradation process to be efficient, it is necessary to take into account the polymer/catalyst ratio, heating rates and reaction time, as these are variables that influence the composition of the product [7]. The textural, morphological and structural properties of the catalyst play a significant role in the distribution of the final product, therefore the choice of the catalytic system used must be of paramount importance [8].

Various types of zeolites have been applied as catalysts for catalytic pyrolysis processes due to their characteristics of strong surface acidity and ability to break down hydrocarbons, significantly reducing the temperature and reaction time of the process. These characteristics provide products with better quality and applicability for a wide variety of polymers [9].

In this study, HZSM-5 zeolite was used, due to its high thermal stability, intrinsic acidity, high surface area and porosity [10]. Some studies in the literature have used oxides to increase the catalytic activity and stability of catalysts. The HZSM-5 zeolite was impregnated with cerium oxide ( $\text{CeO}_2$ ) in different proportions, as cerium compounds have enormous potential for application in various technological areas, mainly due to some of their properties, such as redox potential range and high mobility of oxygen in the crystal lattice [11]. Therefore, the present work aims to study the catalytic conversion of post-consumer polystyrene (PS) over  $\text{CeO}_2$ /HZSM-5 type catalysts, aiming to obtain more active catalysts with lower energy costs for the catalytic pyrolysis process.

## 2. Experimental

### 2.1. Obtaining the Catalysts

The preparation of the catalysts was carried out using commercial ZSM-5 zeolite (Zeolyst International), in its ammoniac form. The ammoniac zeolite was calcined in an air atmosphere at  $450^\circ\text{C}$  in a muffle furnace with a heating rate of  $10^\circ\text{C}/\text{min}$ , after reaching  $450^\circ\text{C}$ , the system was maintained under these conditions for 3 hours, according to a procedure adapted from the literature [12].

During the calcination stage, the decomposition of  $\text{NH}_4^+$  ions occurs, which are compensating charges within the zeolite structure, releasing  $\text{NH}_3$ , and obtaining the acidic form HZSM-5 (Equation (1)). This proton originating from the decomposition of the ammonium ion binds to the oxygen close to the structural aluminum, generating an acid Brönsted site, making the surface acidic. The procedure for obtaining zeolite was adapted from previous works in the literature [13] [14]. Cerium oxide ( $\text{CeO}_2$ ) was calcined in an air atmosphere at  $400^\circ\text{C}$  in a muffle furnace with a heating rate of  $10^\circ\text{C}/\text{min}$  for 2 hours.



The zeolite obtained in its acid form was impregnated with cerium oxide through the mechanic synthesis process according to a procedure adapted from Santos [13]. Impregnation was carried out by adding 10% and 20% by mass of cerium oxide to the zeolite. These percentages were made in relation to the total mass of the catalyst to be obtained, which is 1 gram. With the aid of a pestle and mortar, the phases were homogenized, each sample had a mixing time of 5 minutes. From this process, 10%  $\text{CeO}_2$ /HZSM-5 and 20%  $\text{CeO}_2$ /HZSM-5 catalysts were obtained.

### 2.2. Characterization of the Catalysts

The catalysts were characterized morphologically, structurally and texturally, respectively, by scanning electron microscopy (SEM), X-ray diffractometry (XRD) and nitrogen adsorption and desorption at 77K.

The morphology of the particles that make up the materials in this study was evaluated based on scanning electron microscopy analysis. The micrographs

were obtained on Hitachi model TM3000 equipment with 1000x magnification, using a sample holder using a thin copper tape to which the samples were adhered. This method was used to morphologically evaluate calcined CeO<sub>2</sub>, HZSM-5, 10% CeO<sub>2</sub>/HZSM-5 and 20% CeO<sub>2</sub>/HZSM-5.

XRD analyzes were carried out on a Panalytical Empyrean diffractometer, model TCU 1000N, with a radiation source Cu K $\alpha$  = 1.5418 Å, nickel filter, with a pitch of 0.026°, current of 40 mA, voltage of 40 kV, speed of scan of 2° min<sup>-1</sup> at an angular range ( $2\theta$ ) of 5° to 60°.

The textural properties of the material were determined through nitrogen adsorption analysis at 77K. The analyses were carried out on the NOVA 1200 equipment. For the analysis, the catalysts were previously degassed for 2 hours under vacuum at 300°C.

### 2.3. Sample Preparation

The polystyrene used for the pyrolysis tests comes from a commercial plastic container (plastic cup). It was pulverized with the help of a file, generating fine particles and after this procedure they underwent rapid homogenization for 5 minutes in a mortar and pestle.

To prepare the samples for the catalytic pyrolysis, calcined CeO<sub>2</sub>, HZSM-5, 10% CeO<sub>2</sub>/HZSM-5, 20% CeO<sub>2</sub>/HZSM-5 and polystyrene (PS) were used. The mixture of the catalyst with the PS was done with the aid of a pestle and mortar over a period of 5 minutes, per sample. For each sample, 0.03 grams of calcined CeO<sub>2</sub>, HZSM-5, 10% CeO<sub>2</sub>/HZSM-5, 20% CeO<sub>2</sub>/HZSM-5 were weighed for every 0.07 grams of PS. Therefore, each sample was composed of 70% polystyrene and 30% catalyst.

### 2.4. Pyrolysis Tests

The thermal and catalytic pyrolysis tests were carried out in a TG thermal analysis equipment model TGA - 5000 from TA Instruments in an inert nitrogen atmosphere, with a flow of 60 mL/min, at different heating rates (5, 10 and 20°C/min), ranging from room temperature to 900°C, using a platinum crucible with around 3 mg of sample. Each sample was composed of 30% catalyst and 70% PS. The method of Vyazovkin and Goryachko [15] was used to estimate the activation energy of the thermal and catalytic pyrolysis tests.

This model is based on two assumptions: the activation energy is only constant for a certain conversion  $\alpha$  and the validity of the Arrhenius expression for the temperature dependence of the coefficient rate stays. The equation of this model is called Dynamic Equation (Equation (2)), being used to determine  $E_a$  through linear regression of graphs of  $\ln(\beta/T^2)$  versus  $1/T$ , obtaining a straight line with slope, for all the stipulated conversion values [15]. Where  $E_a$  is the activation energy,  $\alpha$  is conversion,  $\beta$  is the heating rate,  $A$  is the pre-exponential Arrhenius factor,  $g(\alpha)$  is the function of the reaction model  $R$  universal gas constant and  $T$  the temperature.

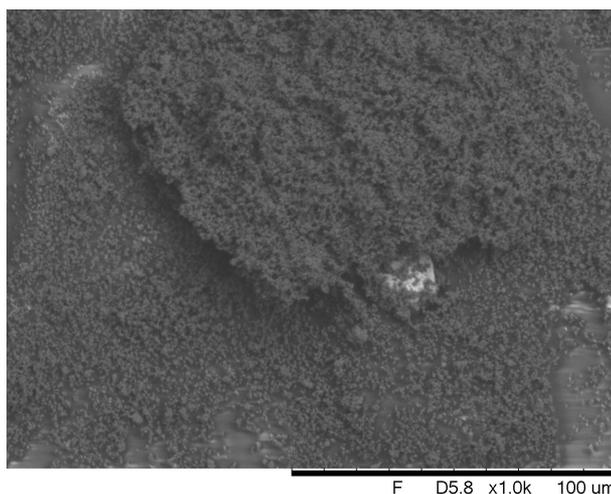
$$\ln\left(\frac{\beta}{T^2}\right) = \ln\left(\frac{RA}{E_{\alpha}g(\alpha)}\right) - \frac{E_{\alpha}}{RT} \quad (2)$$

### 3. Results and Discussion

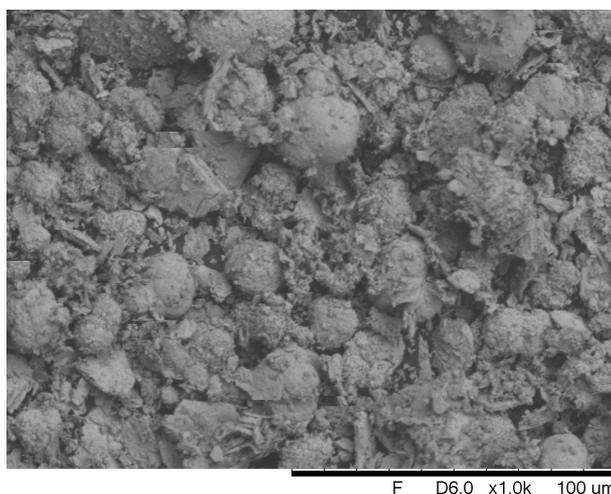
#### 3.1. Characterization of the Materials

**Figure 1** shows scanning electron micrographs (at 1000x magnification) of the HZSM-5, CeO<sub>2</sub>, 10% CeO<sub>2</sub>/HZSM-5 and 20% CeO<sub>2</sub>/HZSM-5 catalysts. In **Figure 1(a)** and **Figure 1(b)**, the HZSM-5 and CeO<sub>2</sub> zeolite crystals can be seen. In **Figure 1(c)** and **Figure 1(d)**, samples of 10% CeO<sub>2</sub>/HZSM-5 and 20% CeO<sub>2</sub>/HZSM-5 can be seen.

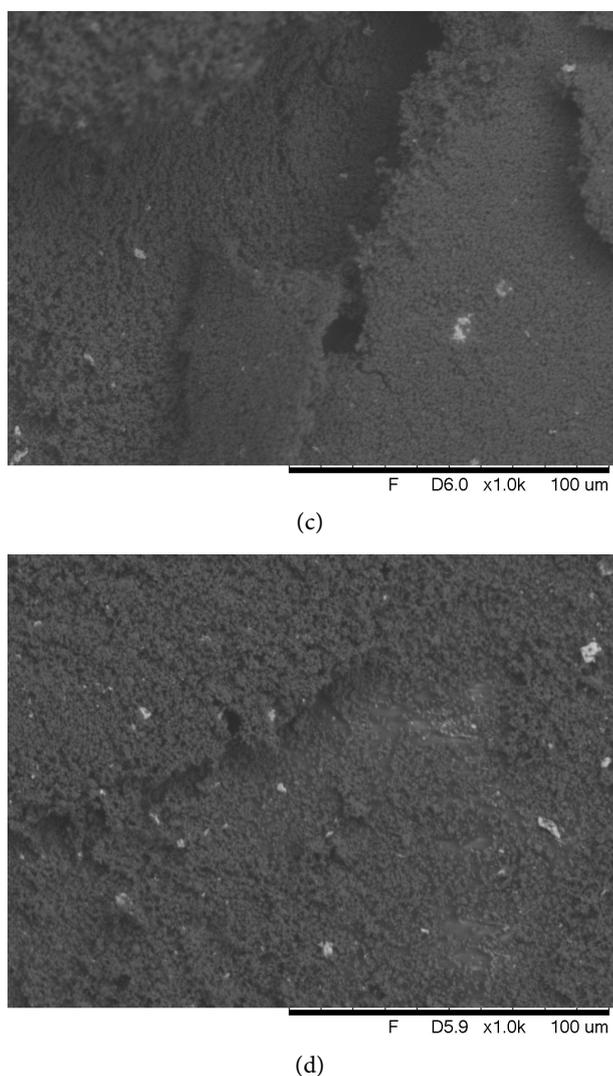
The image of HZSM-5 zeolite shows very small and relatively uniform particles with agglomeration. The CeO<sub>2</sub> image shows particles with an approximately irregular shape of different sizes, and particles with an undefined shape, all with agglomeration, but due to small magnification more details cannot be visualized. The SEM images of the catalysts containing 10% CeO<sub>2</sub>/HZSM-5 and



(a)



(b)

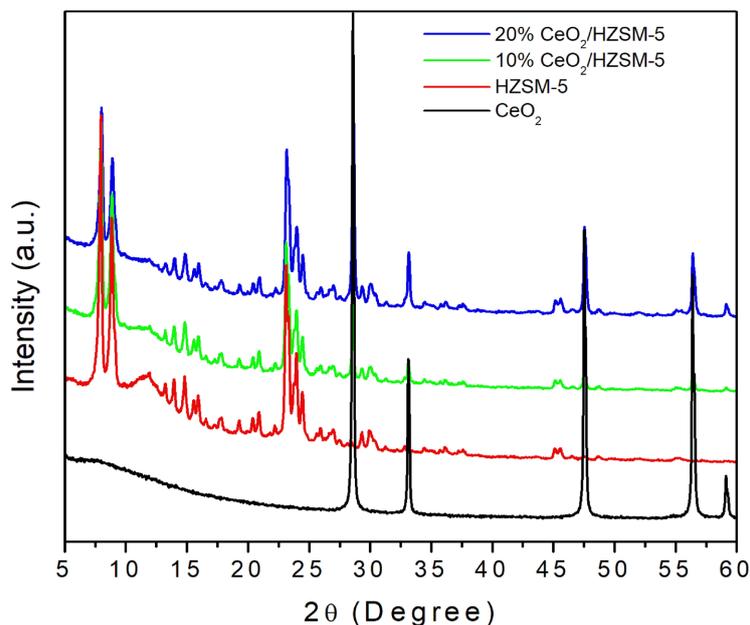


**Figure 1.** Scanning electron micrographs for HZSM-5 (a), CeO<sub>2</sub> (b), 10% CeO<sub>2</sub>/HZSM-5 (c) and 20% CeO<sub>2</sub>/HZSM-5 (d).

20% CeO<sub>2</sub>/HZSM-5 predominantly show the profile of the ZSM-5 zeolite, with emphasis on the scattered small light spots, probably related to the CeO<sub>2</sub> phase. From the micrographs, the typical morphology of the zeolite and also the presence of aggregated CeO<sub>2</sub> were evident.

**Figure 2** shows the X-ray diffractograms of the catalysts HZSM-5, CeO<sub>2</sub>, 10% CeO<sub>2</sub>/HZSM-5 and 20% CeO<sub>2</sub>/HZSM-5. In the diffractograms, diffraction peaks characteristic of the crystalline structure of the HZSM-5 zeolite, as observed in [16] and of cerium oxide, as reported by [17] can be observed. For pure HZSM-5, the largest peaks at  $2\theta$  were observed at  $2\theta = 7.95^\circ; 8.42^\circ; 22.21^\circ; 23.14^\circ$  and  $24.44^\circ$ . The main peaks of CeO<sub>2</sub> were at  $2\theta = 28.59^\circ; 33.13^\circ; 47.52^\circ; 56.38^\circ$  and  $59.12^\circ$ .

The peaks present in the diffractograms of the 10% CeO<sub>2</sub>/HZSM-5 and 20% CeO<sub>2</sub>/HZSM-5 catalysts reflect the characteristics of both the zeolite and the cerium oxide. It is noted that the characteristic peaks of zeolite and cerium oxide



**Figure 2.** X-ray diffraction patterns of HZSM-5, CeO<sub>2</sub>, 10% CeO<sub>2</sub>/HZSM-5 and 20% CeO<sub>2</sub>/HZSM-5 catalysts.

appear in the samples of the 10% CeO<sub>2</sub>/HZSM-5 and 20% CeO<sub>2</sub>/HZSM-5 catalysts, where the high intensity peaks are preserved. In the 10% CeO<sub>2</sub>/HZSM-5 catalyst sample, the main peaks of HZSM-5 were at  $2\theta = 7.97^\circ$ ;  $8.87^\circ$ ;  $23.11^\circ$ ;  $23.97^\circ$  and  $24.47^\circ$ , for the 20% CeO<sub>2</sub>/HZSM-5 sample the main peaks of HZSM-5 were at  $2\theta = 8.00^\circ$ ;  $8.89^\circ$ ;  $23.16^\circ$ ;  $23.98^\circ$  and  $24.48^\circ$ . The peaks related to CeO<sub>2</sub> are maintained in the 10% CeO<sub>2</sub>/HZSM-5 and 20% CeO<sub>2</sub>/HZSM-5 materials, although the zeolite presents some low intensity peaks in these positions. The results confirm that after the mechanic synthesis process, the structure of the ZSM-5 zeolite and CeO<sub>2</sub> are maintained in the final catalyst.

Based on the adsorption points obtained, they were correlated using the BET (Brunauer-Emmett-Teller) and  $t$ -plot methods to estimate specific surface areas (m<sup>2</sup>/g), micropore volume (cm<sup>3</sup>/g) and internal and external (m<sup>2</sup>/g). The results are presented in **Table 1**. The specific surface area that was obtained by the BET method for the HZSM-5 zeolite was close to the reference values found in the literature [18]. It was observed that the sample containing 10% cerium oxide had a gain in its specific surface area, with an increase in the internal surface area and microporous volume, while for the sample containing 20% cerium oxide there was a loss in area specific surface area and a small increase in internal surface area and micropore volume. Suggesting that smaller amounts of cerium oxide do not block the zeolite pores and channels, however when used in greater quantities it can obstruct the zeolite pores, thus reducing its specific surface area.

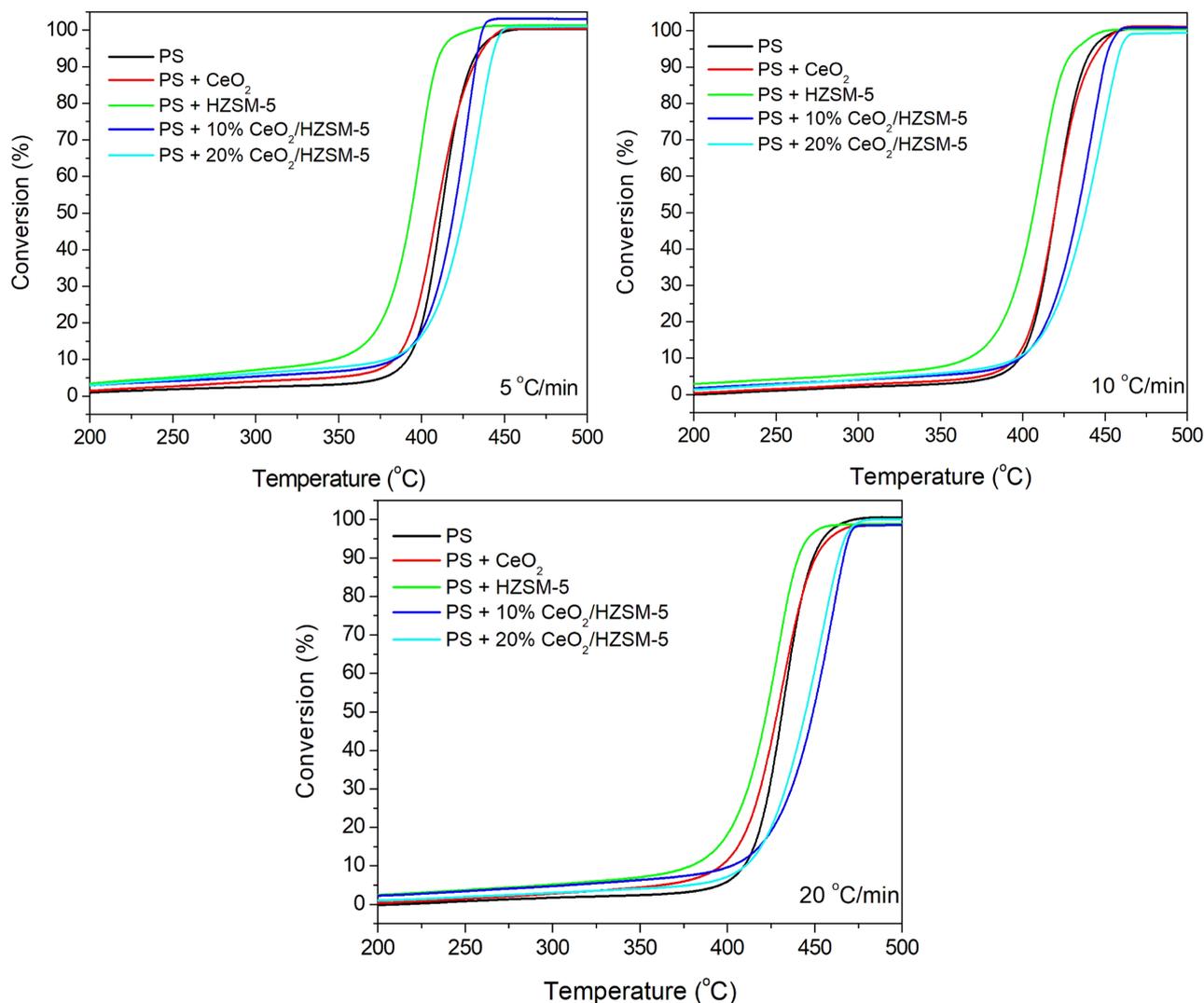
### 3.2. Pyrolysis Tests

The conversion curves and kinetic data for the thermal and catalytic degradation process were obtained from thermogravimetric data. **Figure 3** shows the thermal

**Table 1.** Textural properties of catalytic materials.

Sample	$S_{\text{BET}}$ ( $\text{m}^2\cdot\text{g}^{-1}$ )	$S_{\text{INT}}$ ( $\text{m}^2\cdot\text{g}^{-1}$ )	$S_{\text{EXT}}$ ( $\text{m}^2\cdot\text{g}^{-1}$ )	$V_{\text{MIC}}$ ( $\text{cm}^3\cdot\text{g}^{-1}$ )
HZSM-5	294	238	56	0.109
10% $\text{CeO}_2/\text{HZSM-5}$	321	266	55	0.120
20% $\text{CeO}_2/\text{HZSM-5}$	263	248	15	0.113

Where:  $S_{\text{BET}}$  = specific surface area by the BET method,  $S_{\text{INT}}$  = internal surface area by the  $t$ -Plot method,  $S_{\text{EXT}}$  = external surface area by the  $t$ -Plot method and  $V_{\text{MIC}}$  = microporous volume by the  $t$ -Plot method.

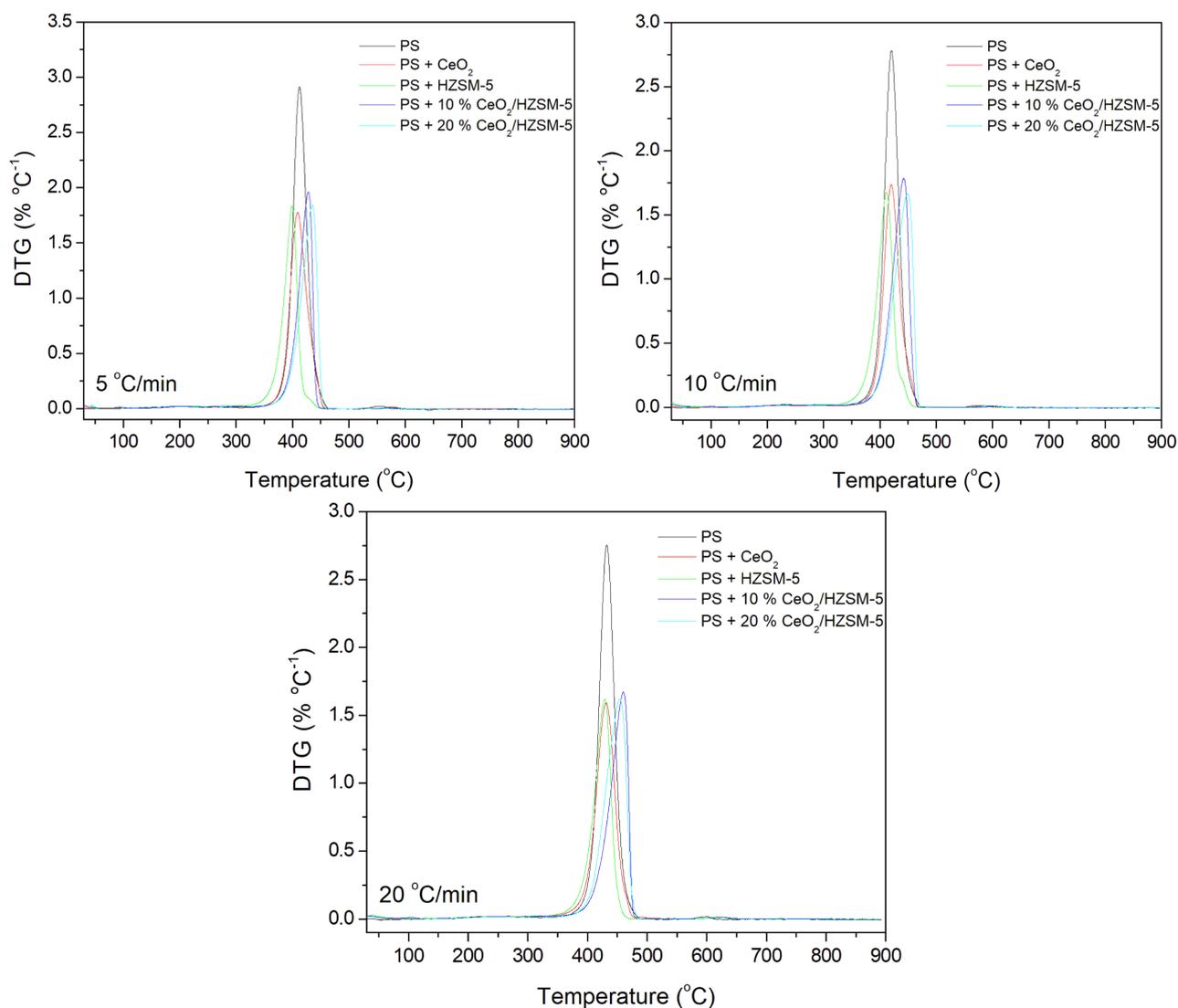
**Figure 3.** PS thermal and PS catalytic conversion curves over different catalysts at heating rates of 5, 10 and 20 °C/min.

and catalytic conversion curves of polystyrene at different heating rates. The action of the HZSM-5 zeolite can be observed in the thermal profiles found. It can be observed that in general, the presence of the zeolite shifts the conversion temperatures to lower temperatures, when compared to thermal pyrolysis.

For catalysts zeolytic impregnated with cerium oxide, conversions begin to

occur more quickly than in thermal pyrolysis, especially at the beginning of the process, but at higher temperatures an inverse change occurs, requiring temperatures higher than those of thermal pyrolysis to achieve the same degree of conversion and this may be occurring in the range of experimental conditions studied due to diffusional limitations to heat and mass transfer. Regarding the addition of  $\text{CeO}_2$  to PS can be observed in the thermal and catalytic curves at all heating rates that the conversion curves profiles of pure PS and PS +  $\text{CeO}_2$  are close with only a small difference in temperature when the oxide is added to the PS. Through the DTG curves, it was possible to observe the small temperature differences between thermal and catalytic pyrolysis, present in **Figure 4**. The kinetic parameters are obtained from mathematical methods that are applied to data from the TG and DTG experiments [19].

The kinetic model proposed by Vyazovkin and Goryachko [15] (Equation (1)) was chosen for the present work. The model is based on isoconversional techniques



**Figure 4.** DTG curves of thermal and catalytic pyrolysis of PS over different catalysts at heating rates of 5, 10 and 20 °C/min.

for calculating apparent activation energy ( $E_a$ ) as a function of conversion ( $\alpha$ ) [20] [21]. One of the main advantages of this method is the possibility of isolating the function  $g(\alpha)$  from the linear coefficient. The determination of this function in complex processes is difficult to determine [22]. Thus, based on the  $T_a$  observed in the DTG curves (Figure 4), it was possible to estimate the activation energy for the thermal and catalytic pyrolysis reactions of PS as shown in Table 2.

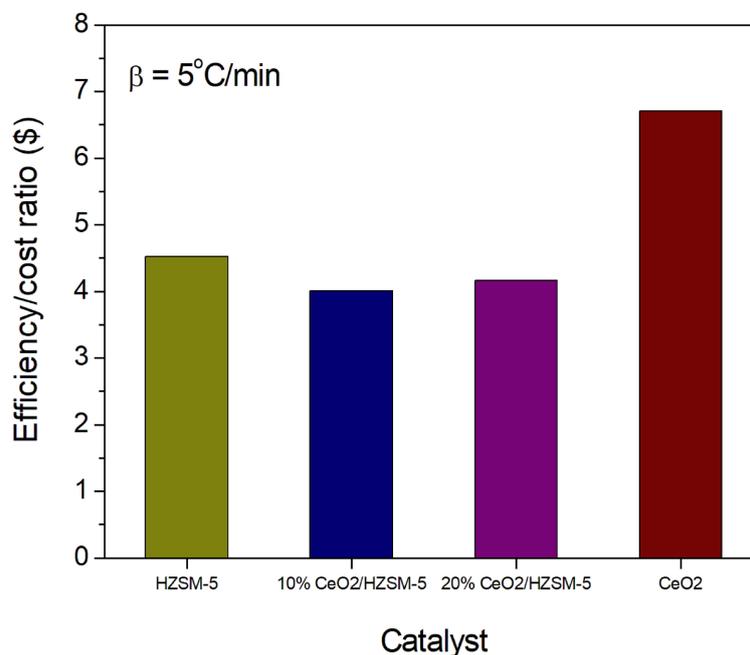
When observing the values of temperatures ( $T_a$ ) at conversions ( $\alpha$ ) of 50% of the samples at  $\beta = 5^\circ\text{C}/\text{min}$ , it is possible to notice that there is a temperature difference of approximately  $12.5^\circ\text{C}$  between the decomposition temperatures of pure PS and PS + HZSM-5, making the role of the catalyst evident in the process. The activation energy for PS degradation found by the Vyazovikin method was 274 kJ/mol and the lowest activation energy for PS degradation was 173 kJ/mol, this being when the 10%  $\text{CeO}_2/\text{HZSM-5}$  catalyst was used, thus resulting in a reduction of ca. 100 kJ/mol (corresponding to a 37% reduction). The activation energies for PS degradation in decreasing order were: PS + 20%  $\text{CeO}_2/\text{HZSM-5}$  > PS > PS +  $\text{CeO}_2$  > PS+HZSM-5 > PS + 10%  $\text{CeO}_2/\text{HZSM-5}$ . In Table 2, the activation energy values obtained in thermal and catalytic pyrolysis for a conversion  $\alpha = 50\%$  can be seen.

In 2024, the commercial value of a kilogram of ZSM-5 zeolite was US\$300 and of cerium oxide was US\$330 and the average production cost of a kilogram of 10%  $\text{CeO}_2/\text{HZSM-5}$  and 20%  $\text{CeO}_2/\text{HZSM-5}$  catalysts was US\$303 and US\$306, respectively. Recent studies indicate good applicability for cerium oxide in other technologically profitable processes [23] [24]. The cost/efficiency ratio of each catalyst was calculated based on the relationship between efficiency (catalyst conversion at maximum reaction speed) and the total production cost of each catalyst. It can be seen in Figure 5 that the values of the efficiency/cost ratio of the catalysts for HZSM-5 is 0.221, for 10%  $\text{CeO}_2/\text{HZSM-5}$  it is 0.249, for 20%  $\text{CeO}_2/\text{HZSM-5}$  it is 0.240 and for  $\text{CeO}_2$  it is 0.149. Thus demonstrating that the best efficiency/cost ratio observed experimentally is that of the 10%  $\text{CeO}_2/\text{HZSM-5}$  catalyst.

**Table 2.** Temperatures ( $T_a$ ) as a function of the heating rate for the thermal and catalytic conversion of PS and respective activation energy values ( $E_a$ ).

Heating rate ( $^\circ\text{C}/\text{min}$ )	$T_a$ ( $^\circ\text{C}$ ) A	$T_a$ ( $^\circ\text{C}$ ) B	$T_a$ ( $^\circ\text{C}$ ) C	$T_a$ ( $^\circ\text{C}$ ) D	$T_a$ ( $^\circ\text{C}$ ) E
5	411.97	408.99	399.39	428.26	434.92
10	420.24	419.95	411.69	442.03	448.95
20	432.40	430.65	428.68	459.63	454.80
$(E_a)$ (kJ/mol)	274	255	180	173	276

Where: A = PS, B = PS +  $\text{CeO}_2$ , C = PS + HZSM-5, D = PS + 10%  $\text{CeO}_2/\text{HZSM-5}$  and E = PS + 20%  $\text{CeO}_2/\text{HZSM-5}$ .



**Figure 5.** Efficiency to cost ratio of catalysts.

#### 4. Conclusion

In this work, the catalytic material based on HZSM-5 and CeO<sub>2</sub> was successfully developed. The catalysts developed for studies were HZSM-5, 10% CeO<sub>2</sub>/HZSM-5 and 20% CeO<sub>2</sub>/HZSM-5. Through the micrographs presented, it was possible to verify the typical and characteristic morphology of zeolite and cerium oxide. Through XRD, the presence of two crystalline phases relating to zeolite HZSM-5 and cerium oxide was verified in the 10% CeO<sub>2</sub>/HZSM-5 and 20% CeO<sub>2</sub>/HZSM-5 catalysts. The use of cerium oxide as a promoter of the zeolitic catalyst proved to be effective when analyzing the activation energy values achieved in the process. The 10% CeO<sub>2</sub>/HZSM-5 catalyst was the most effective for PS pyrolysis, with an activation energy of 173 kJ/mol, followed by HZSM-5 with an activation energy of 180 kJ/mol. When evaluating the reaction efficiency/cost of the catalysts, it was once again confirmed that the most promising sample was 10% CeO<sub>2</sub>/HZSM-5.

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#### Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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