

Microwave assisted pyrolysis of expanded polystyrene waste using carbon black catalyst

Pirólise assistida por micro-ondas de resíduos de poliestireno expandido utilizando negro de fumo como catalisador

Pirólisis por microondas de residuos de poliestireno expandido utilizando negro de carbón como catalizador

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Abstract

Polymeric materials are increasingly present in everyday life and are discarded on a large scale constituting a challenge for its recycling. This work studied the depolymerization of expanded polystyrene (EPS) waste through microwave using carbon black as catalyst. The catalytic pyrolysis was investigated at five catalysts to polymer ratios (0.0125:1, 0.125:1, 0.25:1, 0.5:1, 1:1). The microwave assisted pyrolysis occurred at 400 W in a fixed time of 12 minutes. Fourier Transform Infrared Spectroscopy (FTIR) and thermogravimetric analysis (TGA) were performed to evaluate the depolymerization. The highest yield of non-condensable gases was obtained at the ratio 0.25:1 with a gaseous yield of 53%. FTIR analysis from liquid fraction of the ratio 0.25:1 showed new absorption bands and new degradation peaks at lower temperatures were observed in thermogravimetry when compared with EPS waste. This study demonstrated the possibility of depolymerization of EPS via microwave using carbonaceous structures, which may contribute to EPS recycling.

Keywords: Polystyrene waste; Catalysis; Waste treatment.

Resumo

Os materiais poliméricos estão cada vez mais presentes no dia a dia e são descartados em larga escala constituindo um desafio para sua reciclagem. Este trabalho estudou a despolimerização de resíduos de poliestireno expandido (EPS) por micro-ondas utilizando negro de fumo como catalisador. A pirólise catalítica foi investigada em cinco razões de catalisadores para polímero (0,0125:1, 0,125:1, 0,25:1, 0,5:1, 1:1). A pirólise assistida por micro-ondas ocorreu a 400 W em um tempo fixo de 12 minutos. Espectroscopia de Infravermelho com Transformada de Fourier (FTIR) e análise termogravimétrica (TGA) foram realizadas para avaliar a despolimerização. O maior rendimento de gases não condensáveis foi obtido na razão 0,25:1 com um rendimento gasoso de 53%. A análise FTIR da fração líquida na proporção 0,25:1 mostrou novas bandas de absorção e novos picos de degradação em temperaturas mais baixas foram observados na termogravimetria quando comparados com resíduos de EPS. Este estudo demonstrou a possibilidade de despolimerização do EPS via micro-ondas utilizando estruturas carbonáceas, o que pode contribuir para a reciclagem do EPS.

Palavras-chave: Resíduos de poliestireno; Catálise; Tratamento de resíduos.

Resumen

Los materiales poliméricos están cada vez más presentes en la vida cotidiana y son desechados a gran escala, constituyendo un reto para su reciclaje. Este trabajo estudió la despolimerización por microondas de residuos de poliestireno expandido (EPS) utilizando negro de carbón como catalizador. La pirólisis catalítica se investigó en cinco proporciones de catalizador a polímero (0,0125:1, 0,125:1, 0,25:1, 0,5:1, 1:1). La pirólisis asistida por microondas tuvo lugar a 400 W en un tiempo fijo de 12 minutos. Se realizaron espectroscopia infrarroja por transformada de Fourier (FTIR) y análisis termogravimétrico (TGA) para evaluar la despolimerización. El mayor rendimiento de gases no condensables se obtuvo en la relación 0,25:1 con un rendimiento de gas del 53%. El análisis FTIR de la fracción líquida en la proporción 0,25:1 mostró nuevas bandas de absorción y nuevos picos de degradación a temperaturas más bajas se observaron en termogravimetría en comparación con los residuos de EPS. Este estudio demostró la

posibilidad de despolimerización de EPS mediante microondas utilizando estructuras carbonosas, lo que puede contribuir al reciclaje de EPS.

Palabras clave: Residuos de poliestireno; Catálisis; Tratamiento de desechos.

1. Introduction

The plastic consumption is increasing around the world with an increasing in population (Silva et al., 2022; Khan et al., 2020; Murad & Alam, 2019). Plastic materials are in high demand for several sectors such as automotive parts, building construction and packaging (Silva et al., 2022; Murad & Alam, 2019; Poletto & Zattera, 2017). The world plastic production crosses 380 million tons/year (Premalatha et al., 2021). Nearly 40% of the plastics are used in the packaging industries (Suriapparao et al., 2021). The usage of a large quantity of different plastic materials results in a large quantity of plastic waste discarded every day (Jiang et al., 2020). The recycling of plastic waste is a preferable option (Morais & Vidigal, 2021); however a limited amount is effectively recycled (Silva et al., 2022; Jiang et al., 2020). On the other hand, disposal of plastic waste in landfill causes loss of valuable material and energy, while improper incineration generates emission of harmful carcinogenic gases which affects both the environmental and human health (Jiang et al., 2020; Prathiba et al., 2018). Mitigation of plastic waste is a challenging for public and private sectors. The recovery of energy and chemicals from plastic waste is a more viable option (Suriapparao et al., 2021) to reduce the quantities of plastic materials in landfill and also reduce the usage of energy and water in its production.

The pyrolysis method has been used to convert plastic waste in valuable products (Bhattacharya & Basak, 2016). The pyrolysis is an efficient method to convert complex polymer molecule into simpler hydrocarbons (Premalatha et al., 2021). However, higher amount of energy consumption and reaction time (Prathiba et al., 2018) are some disadvantages factors when this method is chosen to plastic recycling. Microwave pyrolysis is a potential method to better recycled plastic waste because the processing time and temperature can be greatly reduced (Prathiba et al., 2018). The waste polyolefins can be recycled in microwave assisted pyrolysis to obtain liquid products that may be used as a source of chemicals and fuel without any preliminary treatments (Premalatha et al., 2021; Prathiba et al., 2018).

Microwave absorbers or susceptors materials are added to the feed plastic mixture in the pyrolysis for the efficient conversion of the incident microwave energy into thermal energy (Suriapparao et al., 2021; Menéndez et al., 2010). These materials can help in obtaining the required pyrolysis temperature through higher heating rates in a reduced time when compared with conventional pyrolysis and also maintaining uniform heat distribution (Suriapparao et al., 2021). The susceptors generally are carbon based compounds or metal oxides (Li et al., 2019; Menéndez et al., 2010). Carbonaceous susceptors enhancing the energy conversion efficiency during microwave assisted pyrolysis while processing poor microwave absorbing materials like plastics by providing rapid initial heating (Suriapparao et al., 2021). Microwave penetrate deep inside the susceptor particles and transforms into thermal energy while plastic waste is heated by the contact with the superficial area of the particle from external to its internal part.

Premalatha and coworkers (2021) studied thermal and catalytic pyrolysis of polypropylene waste using batch reactor in the temperature range of 400-460°C. Sulfonated carbon was used as susceptor with four different catalysts to polymer ratios (1:20, 1:10, 1:5, and 1:3). The highest yield of catalytic pyrolytic liquid product was obtained at the ratio of 1:5 with a liquid yield of 86% at 420°C and 50 min of pyrolytic reaction time. Prathiba et al. (2018) evaluated the microwave pyrolysis of polystyrene waste in the presence of activated carbon. At a microwave of 450 W and a polymer to activated carbon ration of 10:1 the highest oil yield of 93 % was obtained with a temperature of 330°C and a reaction time of 5.5 min.

The aim of this study is evaluates the effect of different carbon black polymer ratio to obtain recyclable products from polystyrene waste using microwave assisted pyrolysis. The results indicated the when the reaction conditions were controlled it is possible obtain value-added products and also minimize the plastic pollution.

2. Methodology

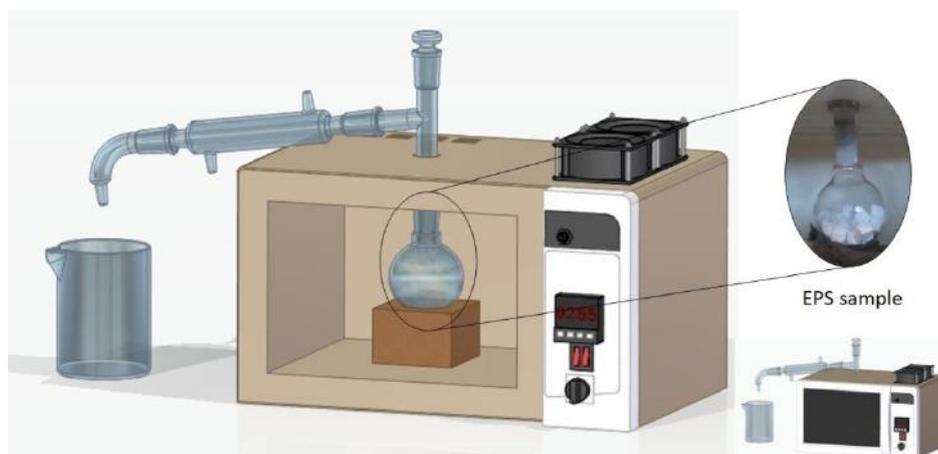
2.1 Materials

Carbon black was supplied from Cabot Carbon Ltd with a medium particle size of 150 μm . Expanded polystyrene wastes were obtained from a local sorting unit located in Caxias do Sul city (Brazil). The EPS wastes from packaging of electronic goods and home appliances were used. Food packaging EPS wastes were not used. The EPS samples were cleaned and cut into small cubes with approximately 8 cm^3 for further utilization.

2.2 Microwave-assisted pyrolysis of waste polystyrene

Figure 1 shown the microwave pyrolysis experiment set up with an output power of 400W. A borosilicate quartz flask of 250 mL holds the EPS samples inside the microwave oven. A borosilicate adaptor was used to connect the quartz flask to the condenser. The quartz flask was insulated with mineral wool to avoid heat loss. The joints are tightly sealed using Teflon tape, to avoid the escape of non-condensable gases. The experimental set up and methodology used in this study were based on the works of Prathiba et al. (2018) and Premalatha et al. (2021).

Figure 1 - Experimental setup used for EPS microwave pyrolysis.



Source: Authors (2022).

The microwave pyrolysis experiments were investigated at five different carbon black catalysts to EPS polymer ratios (0.0125:1, 0.125:1, 0.25:1, 0.5:1, 1:1). The total reaction time was 12 min, divided in three cycles of 4 min each. The reaction temperature was maintained at around 350-400°C. At the end of experiments, liquid, residue and gas yield was obtained by weighing the amount of oil and the residue was weighed after subtracting the mass of carbon black from the residue. The gas yield was calculated by mass balance. The experiments were done in triplicate.

2.3 Characterization of product oil

FTIR analysis was used to determine the functional groups in the product oil obtained after microwave pyrolysis depolymerization. The analyses were performed on Nicolet IS10-Termo Scientific equipment. The result was obtained through the average of 32 scans, in the range of 4000 cm^{-1} to 400 cm^{-1} , with a resolution of 4 cm^{-1} using ATR mode.

Thermogravimetry is an analytical method to measure the mass loss of a substance against temperature. Shimadzu TGA 50 analyser was used to determine the sample decomposition temperature. The temperature was measured from 23°C to 800°C with a heating rate of $10^{\circ}\text{C}\cdot\text{min}^{-1}$ with a nitrogen gas flow of $50\text{ mL}\cdot\text{min}^{-1}$ using a mass sample of approximately 10 mg.

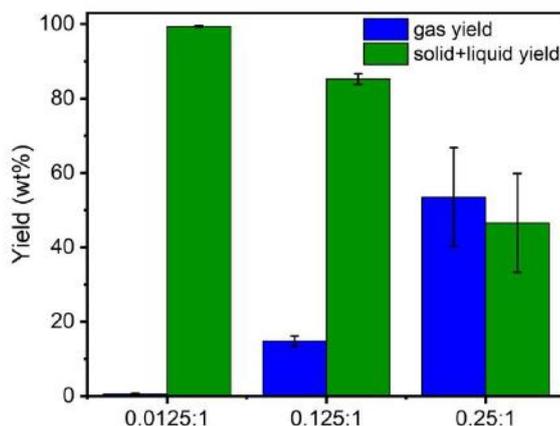
3. Results and Discussion

3.1 Microwave pyrolysis of EPS waste

The microwave pyrolysis tests with catalyst to polymer ratios of 0.5:1 and 1:1 were not satisfactory because both samples are extremely degraded after 30 s exposed to microwave radiation. A possible explanation to this behaviour is based on the high value of dielectric constant of the carbonaceous material which is capable of converting electromagnetic energy to heat (Rex et al., 2020). The dielectric property of the susceptor helps in faster completion of reaction which can generate uncontrolled reactions and faster polymer degradation (Rex et al., 2020; Hussain et al., 2012). On the other hand, as more the amount of black carbon, the polymer receives higher heating rates hence the longer polymer chain gets converted into simpler hydrocarbons which escape as non-condensable gases (Rex et al., 2020, Rosi et al., 2018). Based on these preliminary results, the carbon black to polymer ratios of 0.5:1 and 1:1 were not further tested throughout the presented study.

The catalyst to polymer ratios of 0.0125:1, 0.125:1 and 0.25:1 was then tested. Figure 2 shows the yield of non-condensable gases and solid/liquid residue for different EPS waste to carbon black ratios.

Figure 2 - Effect of EPS waste to carbon black ratio in microwave pyrolysis.



Source: Authors (2022).

It is clearly seen in Figure 2 that different carbon black EPS waste ratios increased the gas yield from $0.62\pm 0.12\%$ to $53.46\pm 13.27\%$. This may be associated with primary vapors undergoing secondary reactions which lead to the formation of more non-condensable gases and coking reaction and generation of high spot temperature for EPS waste decomposition (Rex et al., 2020). Moreover, from the reaction time used in this study, it is observed that lower amounts of carbon black are

inefficient to convert EPS waste in valuable products. The sample containing 0.0125:1 susceptor to polymer ratio is basically formed by a solid sample without generated of a viscous liquid fraction.

The samples with carbon black to polymer ratio of 0.125:1 and 0.25:1 presented and increase in the formation of a viscous fraction in the flask associated with a reduction of the solid residue. The viscous fraction was used for FTIR and TGA analysis.

Table 1 compare the gas yield results obtained in this work with others results from the literature for polystyrene microwave pyrolysis using carbonaceous materials as susceptors.

Table 1 - Comparison of gas yield results during polystyrene microwave pyrolysis.

Carbonaceous material	Carbonaceous to EPS ratio	Reaction time (min)	Gas yield (%)	Reference
Carbon black	1.25:10	12	14.79	this study
Carbon black	2.5:10	12	53.46	this study
Carbon powder	4.73:10	22	3.70	Undri et al. (2014)
Carbon powder	5:10	23	6.40	Undri et al. (2014)
Activated carbon	1:10	5.5	5.74	Prathiba et al (2018)

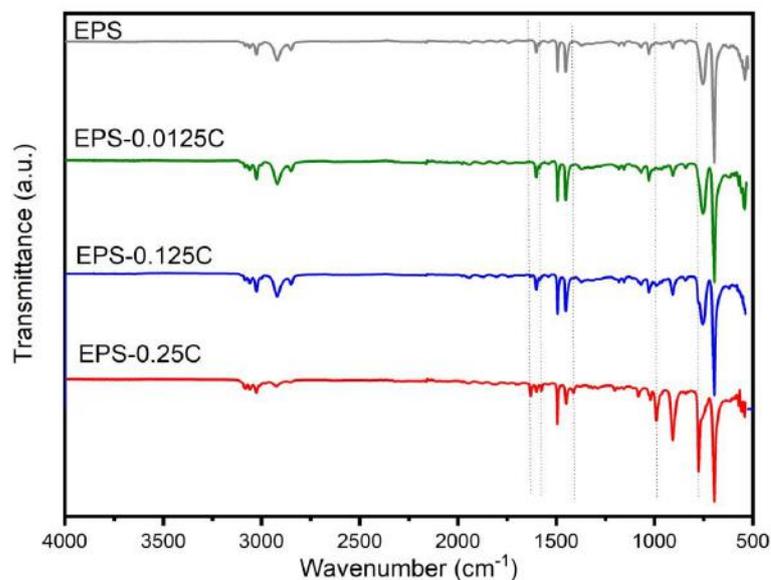
Source: Authors (2022).

The gas yield results in this study are higher than those obtained in previously studies. As discussed before, higher carbon black content originated an increase in the microwave radiation absorption by the susceptor, which leads to an increase in the temperature of the reaction system, providing sufficient energy to convert polystyrene into volatile molecules. Undri et al. (2014) also observed that the high quantities of carbon powder during microwave pyrolysis reaction with EPS resulted in higher gas yield.

3.2 FTIR characterization of liquid fraction

FTIR spectra of EPS and liquid fraction obtained for each sample after microwave pyrolysis is shown in Figure 3.

Figure 3 - FTIR spectra of EPS waste and liquid fraction samples after microwave pyrolysis



Source: Authors (2022).

According León-Bermúdez and Salazar (2008) polystyrene present a band characteristic of the C-H axial stretching associated to the aromatic ring structure at 3081 cm^{-1} . The bands at 3001 cm^{-1} , 2923 cm^{-1} and 2850 cm^{-1} are assigned to C-H bonds from CH_2 . Absorptions that occur between 1943 cm^{-1} and 1728 cm^{-1} are assigned to monosubstitution on aromatic rings (Nasybullin et al., 2015; Undri et al., 2014). The band at 1452 cm^{-1} is associated with C=C-H aryl ring stretching and the band at 1069 cm^{-1} is related to the out-of-plane C-H bond (Undri et al., 2014).

After microwave pyrolysis, bands appeared at 1629 cm^{-1} , 1575 cm^{-1} , 1412 cm^{-1} , 991 cm^{-1} and 775 cm^{-1} for all depolymerised samples. The bands are highlighted in Figure 3. Table 2 presented the assigned of each band observed after microwave pyrolysis.

Table 2 - FTIR absorptions bands of liquid fraction samples after EPS microwave pyrolysis

Wavenumber (cm^{-1})	Attribution*	0.25:1			
		EPS	0.0125:1	0.125:1	
1629	Alkenyl C=C stretching	-	w	w	w
1575	C=C-H aryl ring stretching	w	w	w	m
1412	Vinyl C-H in plane bending	w	w	w	m
991	Vinyl C-H out-of-plane bending	w	w	m	s
775	Aromatic C-H out-of-plane bending	-	w	m	s

* Undri et al (2014) and Mo et al (2013). Where: band intensity report as follow: s, strong; m, medium; w, weak. Source: Authors (2022).

The sample with the lowest carbon black content (0.0125:1) showed only weak bands, similar to the EPS sample. This behaviour may be correlated to the low rate of conversion into gaseous fraction that the 0.0125:1 sample reached in the mass balance. It can be observed in Table 2 that intense bands were only identified in the 0.25:1 sample that have the highest content of carbon black. When the 0.25:1 sample is related to the mass balance, it can be seen that it was the one that showed the highest rate of conversion into a gaseous fraction.

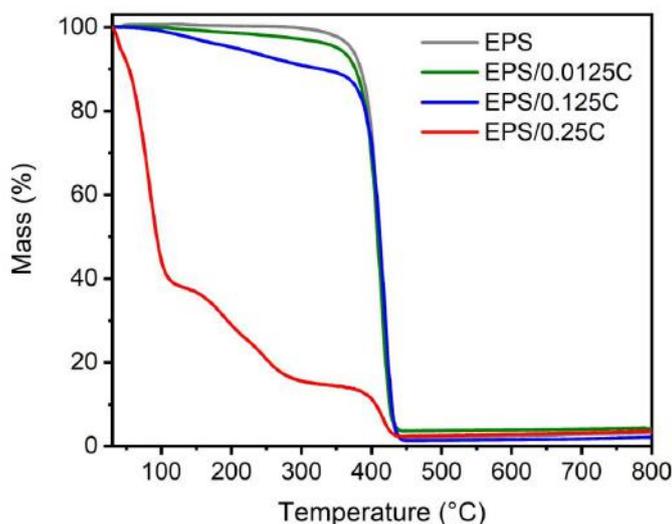
Undri et al. (2014) stated that the benzene, toluene, ethylbenzene, styrene and α -methylstyrene are more than 90% of the compounds present in the liquid fraction at the end of the EPS depolymerization trough microwave pyrolysis. The average

amount of ethylbenzene found by Undri et al. (2014) is 9.38%, while for benzene it was 1.52%. In contrast, Bartoli et al. (2015) did not detect ethylbenzene, but the average of benzene was 0.92%.

3.3 Thermogravimetric characterization of liquid fraction

The thermogravimetric results for EPS and samples submitted to microwave pyrolysis can be seen in Figure 4.

Figure 4 - Thermogravimetric results of EPS and liquid fraction samples after microwave pyrolysis

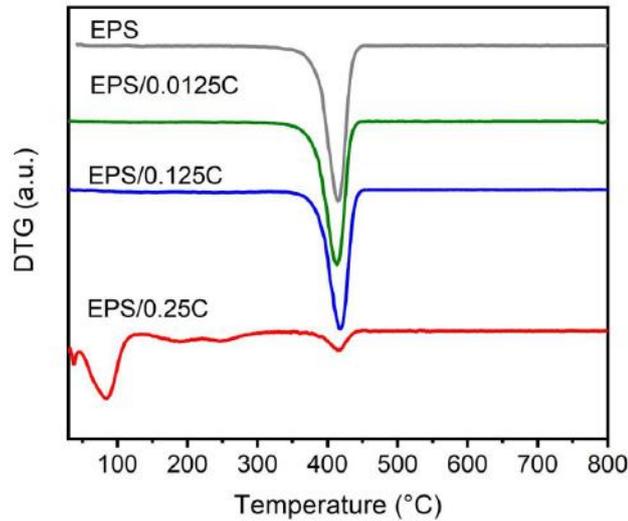


Source: Authors (2022).

The EPS and 0.0125:1 sample showed similar thermogravimetric curves. However, the 0.0125:1 sample showed a reduction in thermal stability when compared to EPS, with a mass loss of approximately 3% up to the temperature of 305°C. This difference may be associated with a weak depolymerization of EPS caused by the small amount of carbon black. Both samples presented a largest mass loss between 325°C and 450°C. The 0.125:1 sample indicated a mass loss of 12% between 100°C and 370°C with a largest mass loss event in the temperature range between 370°C and 450°C. Prathiba et al. (2018) observed through thermogravimetric results that EPS decomposition temperature occurred at 400°C and the maximum decomposition temperature occurred at 425°C.

As can be seen in Figure 4, as the carbon black content increases, a change in the thermogravimetric curve is observed, especially for 0.25:1 sample. The presence of a higher content of carbon black accelerates the depolymerization reaction of EPS and leads to the formation of more volatile compounds. Finally, the 0.25:1 sample has 5 mass loss events, as can be seen in the derivative (DTG) curves, showed in Figure 5.

Figure 5 - DTG curves of EPS and liquid fraction samples after microwave pyrolysis



Source: Authors (2022).

As can be seen in Figure 5, the first mass loss event occurs between 30°C and 45°C, with a peak centred at 38°C and a 3% mass loss. The second occurs between 45°C and 124°C, with a peak centred at 85°C and a mass loss of 59%. The third extends from 140°C to 222°C, with the peak occurring at 189°C and the mass loss of 13%. The fourth event, in turn, takes place between 222°C and 290°C, with a peak centred at 247°C and the mass loss is approximately 9%. The last event is between 355°C and 450°C, with a peak centred at 416°C and a mass loss of 12%.

It can be seen in Table 3 that samples submitted to microwave pyrolysis showed a reduction in temperature of 3% of mass loss, when compared to EPS. This may be associated with the formation of low molar mass compounds. The T3% is the temperature of 3% mass loss for each sample. This parameter was added to verify how the thermal stability of the samples varies after depolymerization. T peak is the peak temperature of degradation.

Table 3 - Thermogravimetric results for EPS and liquid fraction samples after microwave pyrolysis.

Sample	T3% (°C)	T peak (°C)	Residue at 800°C (%)
EPS	358	415	3.3
0.0125:1	305	413	4.2
0.125:1	154	418	2.2
0.25:1	38	416	3.6

Source: Authors (2022).

The results presented in Table 3 reveal that the T3% is drastically reduced with the increase in the carbon black content using in microwave pyrolysis. The carbonaceous susceptor act as a catalyst and promote the EPS waste depolymerization. The method proposed by Broido (1969) was used to determine the activation energy (E_a) of the EPS samples before and after microwave pyrolysis. The Broido's method in an uncomplicated and relatively accurate method (Yassin et al., 2018; Broido, 1969) for determining de E_a values according to Equation 1:

$$\ln [-\ln(1-\alpha)] = -E_a/(RT) + \text{constant} \quad (1)$$

where α is the conversion corresponding to a particular stage of the decomposition reaction, T is the absolute temperature in Kelvin and R is the universal gas constant ($8.314 \text{ J mol}^{-1} \text{ K}^{-1}$). In this method, plotting $\ln [-\ln(1-\alpha)]$ against $(1000/T)$ resulted in straight lines used to determine E_a values from their slopes ($-E_a/R$). The E_a values and their correlation coefficients are presented in Table 4.

Table 4 - Activation energy values of EPS and samples after microwave pyrolysis

Carbonaceous to EPS ratio	E_a (kJ mol^{-1})	R^2
0:1	295.3 ± 0.96	0.995
0.0125:1	248.8 ± 1.46	0.984
0.125:1	46.7 ± 1.44	0.986
0.25:1 (first event)	7.80 ± 0.09	0.991
0.25:1 (second event)	44.4 ± 0.31	0.987

Source: Authors (2022).

As previously observed in the thermogravimetric results, an increase in the carbon black content generates a reduction in the E_a for all samples submitted to microwave pyrolysis when compared to EPS waste. This result confirm the formation of low molecular mass compounds after microwave pyrolysis, since compounds with low molecular mass tend to degrade at low temperatures and require less energy to initiate a degradation process. The 0.25:1 sample presented to main ranges of E_a values. The first one is probably associated to the degradation process that occurs at approximately 100°C and the second may be associated with degradation reaction that initiate at 400°C , as can be seen in Figure 5.

4. Conclusion

The effect of carbon black susceptor on the microwave pyrolysis of EPS wastes was investigated. The results showed that higher quantities of susceptor can lead to a higher amount of non-condensable gases. The better results were obtained with a carbon black to polymer ratio of 0.25:1. Future studies to determine the compounds presented in liquid fraction need to be done. However, based on the results of this study, the valorization of EPS wastes into value-added products may help the environmental through adequate and efficient methods for recycling plastic waste. The challenging of solid waste management could be overcome by converting polymeric wastes into useful products using recycling technologies that consume less water and energy.

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