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Experimental Study of the Influence of Intrinsic Parameters on the Thermal Reactivity of Sawdust, Polyethylene Terephthalate and Composite

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Abstract

Several works have been based on the study of thermal variations in biomass to derive more valuable products such as fuels capable of replacing oil in the event of a crisis or activated carbon used as an adsorbent material, widely used in industry for the elimination of unwanted materials, both in liquid and gaseous environments. A study of thermal parameters such as: heating speed, retention time, drying temperature, carbonization temperature, particle size, was carried out with the aim of determining the characteristic factors of the carbonization of Polyethylene terephthalate (PET), sawdust (SC) and sawdust/polyethylene terephthalate (CPS) mixture. The results of the immediate analysis revealed a very low level of ash in PET (0.013%) compared to the level of ash in sawdust (2.9%), as well as a high level of fixed carbon (82.960%), which suggests the presence of mineral oxides and a significant carbon matrix unlike PET, which indicates a very significant organic matrix (essentially made up of organic matter) with the absence of mineral oxides. The study of thermal parameters showed the water loss from Sawdust (SC) and the Sawdust/Polyethylene terephthalate (CPS) mixture, an increase with temperature, unlike that of PET whose variation is essentially zero. Without heat treatment, sawdust alone contains approximately 7% water. The optimal drying temperature for this study is 110˚C for a stay of 24 hours. It appears that the largest mass losses for the PET samples are between 87.19% and 96.05%, followed by that of the mixture, between 47.33% and 64.37%. And the lowest are observed, those of sawdust (from 24.02% to 62.6%). However, here we can say that the influence of the mass is not great, given the slight difference between the losses by temperature. The results of the study of the influence of

grain size showed that the differences are insignificant, even if we vary the diameter of the grains from simple to triple. To better minimize physical constraints such as the intragranular diffusibility of the volatile matter and the homogeneity of the temperature in the grains, 75 μm particles are found to be optimal for our study. It can be noted when studying the heating rate that the mass loss at the end of the reaction is approximately the same depending on each precursor material. However, it has been demonstrated that the heating rate strongly influences the nature of the reaction products both for volatile materials and for the solid residue as well as on the kinetic parameters of the chemical reaction. Furthermore, the variation in apparent density shows a decrease as a function of the increase in the residence time of the materials in the reactor. As the carbonization time increases, the apparent density decreases. We note, for the lignocellulosic material, that the apparent density stabilizes after 60 minutes.

Keywords

Intrinsic Parameters, Thermal Reactivity, Sawdust, Polyethylene Terephthalate, Composite

1. Introduction

The development of activated carbon from plastic waste is promising and represents an alternative for the recovery of these local materials dumped unnecessarily or simply burned in nature, which further increases the pollution of our environment. In addition, the use of commercial activated carbons is still limited by their high purchase price, their importation, and their use as raw material not available which has a major economic consideration [1]. Carbonization or carbonification (slow pyrolysis) is the more or less slow thermal decomposition of a material into coal and/or gas, in the absence of oxygen or any other oxidant. It is a set of chemical rearrangements according to complex processes and until today not yet precisely defined. Largely these are endothermic reactions, and sometimes exothermic at some stage. However, of all these previous works, carbonization has remained more focused on obtaining results than on the choice of the preponderant preparation factors. Beyond the fact that the quality of the materials obtained depends on the precursor used and mainly on the preparation conditions, factors such as: the heating rate, the retention time, the activation temperature, the carbonization temperature, the activating agents, particle size, etc. interact with each other during preparation. The proportion and nature of the volatile matter and the solid residue change according to the procedure [2]. This is why it is essential to work under strictly constant and known operating conditions, such as the heating rate, the grain size, etc. All these factors modify not only the quantities of gaseous, liquid and solid materials, but also their qualities and the kinetics of their formation. The pyrolysis of

lignocellulosic products could be influenced by several factors such as: the preparation of the sample, the mass of the sample, the residence time within the reactor, the size and the shape of the sample, etc. [3]. Charring is a reaction very sensitive to the operating conditions [4]. Thermal parameters [1]-[7] can strongly orient the reactions that take place within substances subjected to carbonization. But it is clear that, in most of the studies encountered, one factor is fixed at a certain level, while varying the other in order to determine the best optimal preparation condition, which depends on one precursor to another. For this, the main objective is to determine the optimal thermochemical parameters and know their impacts.

2. Experimental

2.1. Plastic Waste: Polyethylene Terephthalate (PET)

Polyethylene terephthalate (PET) plastic waste is collected from urban waste. They are washed, cut into small particles, dried in the sun for 72 hours then kept in an oven at 110˚C for 24 hours. After drying, they are used to study the thermal parameters.

2.2. Sawdust

The biomass used for our study is sawdust. It is obtained from the scientifically named Guarea Cedrata wood from Guarea Laurenti. This bump belonging to the Meliaceae family is widely used by carpenters in the Olézoua district of Yaoundé, Cameroon. It comes in the form of wavy chips and variable lengths. The raw fiber was dried in the open air for 3 days, before being crushed and sieved to extract the fraction of particles of length 75, 300 and 600 μm. The different fractions were retained for the rest of our study.

2.3. Thermal Reactivity Parameters

The monitoring of the influence of humidity, mass, particle size and heating rate, was done in a chamber furnace of the "Carbolite Gero CWF-1100, 30˚C - 1100˚C" type, of the various precursors, SC, PET and CPS, was done according to the evolution of the percentage of material degradation, or loss of mass, which is a dimensionless ratio (Equation (1)).

$$\text{A loss of mass}\left(\%\right)=\frac{m_0-m_{final}}{m_0}\times 100 \quad (1)$$

2.3.1. Influence of the Moisture in the Sample

The precursors selected for our study contain more or less water. The amount of water depends on several factors, among which we cite the very nature of the precursor, its origin, the duration of its exposure in a humid environment and the duration of drying. This study was carried out without prior treatment of 5.0 g of the Sawdust, PET and Sawdust + PET mixture sample. These materials were baked at 80˚C, 90˚C, 100˚C and 110˚C for 24 hours. The final mass made it

possible to eliminate the loss of water from the three samples, according to relationship (2) and we obtained a profile of variation in the humidity rate given in Figure 1.

2.3.2. Influence of the Mass in the Sample

There are several ways to follow the evolution of the mass, among many others, we can mention the differential thermal analysis [5]. However, in the context of our study, we based ourselves on the variations in the mass of the precursors, from 25.0 to 75.0 g depending on the temperature, following a constant heating rate (10˚C/min) and 75 µm in diameter. The results of this study are shown in Figure 2.

$$w(\%) = \frac{m_0 - m_{finale}}{m_0} \times 100 \quad (2)$$

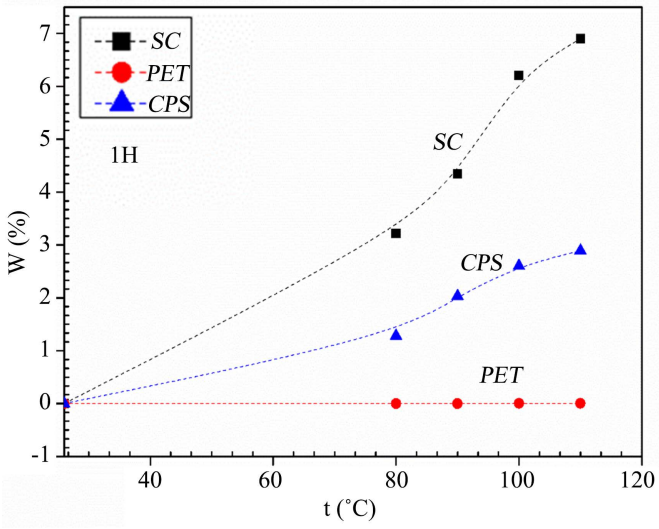

Figure 1. Effects of moisture content of sawdust and PET as a function of temperature.

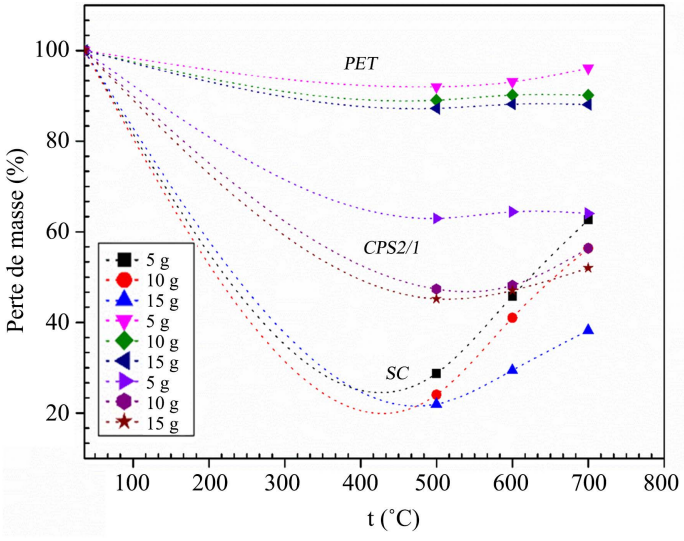

Figure 2. Effect of mass loss, of SC, PET and CPS2/1 in mass function, during pyrolysis.

2.3.3. Influence of Particle Size

In order to study the influence of this physical factor on the loss of mass, this study was carried out for sawdust. This study concerned sizes varying from 75 to 630 µm in diameter at 700˚C and 10˚C/min. The results are reported in Figure 3.

2.3.4. Influence of Heating Rate

Four speeds have been demonstrated, 5˚C/min, 10˚C/min, 15˚C/min and 20˚C/min for the different temperatures, 500˚C, 600˚C and 700˚C. The mass of the precursor retained for this work is 50 g and the results are reported in Figure 4.

2.3.5. Influence of Retention Time in the Reactor

In order to study the influence of time on thermal reactivity, 50 g of the three 75 µm precursors were subjected to 700˚C in the reactor. This study was carried out in a time interval varying from 30 to 180 min. Burn-off and density were determined, the results have been reported in Figures 5-7.

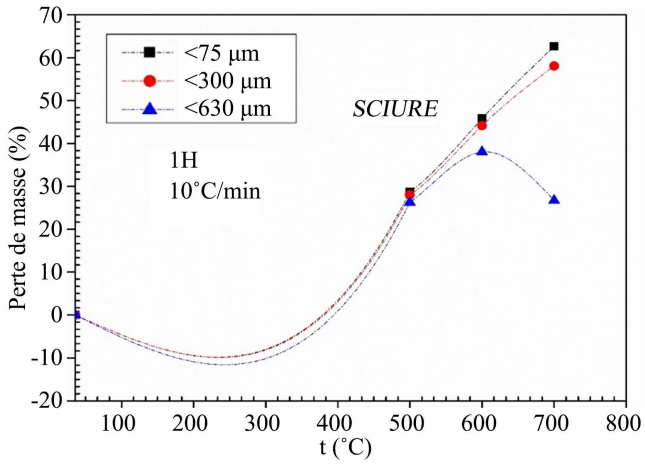

Figure 3. Effect of sawdust particle size on mass loss during pyrolysis.

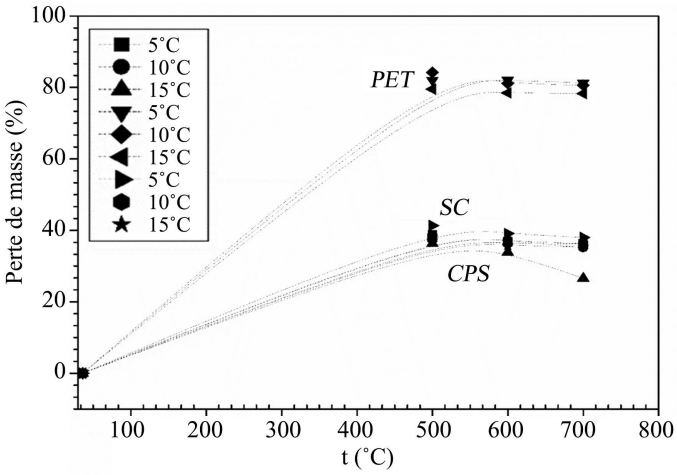

Figure 4. Effect of heating rate on mass loss during pyrolysis.

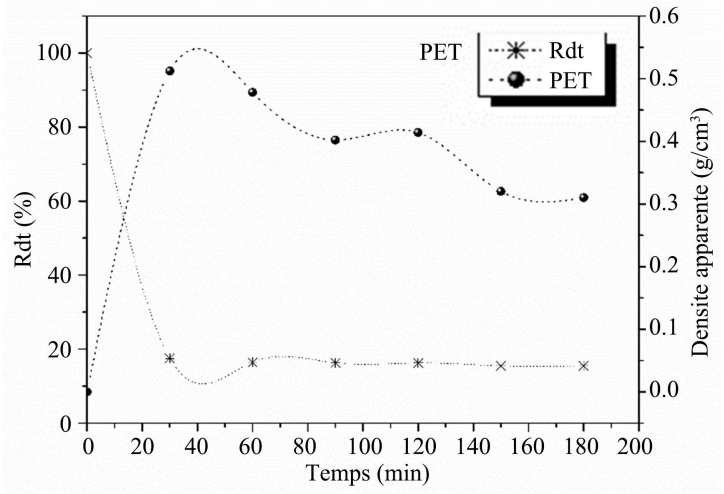

Figure 5. Variation of yield and bulk density of PET as a function of temperature.

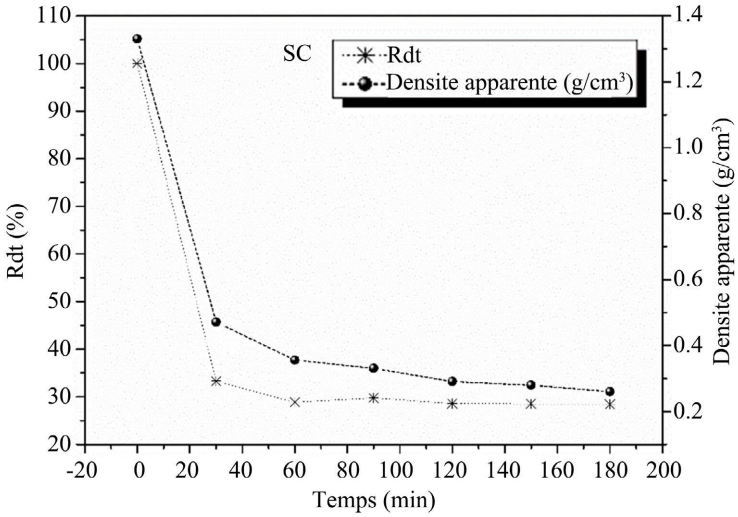

Figure 6. Variation of yield and bulk density of SC as a function of temperature.

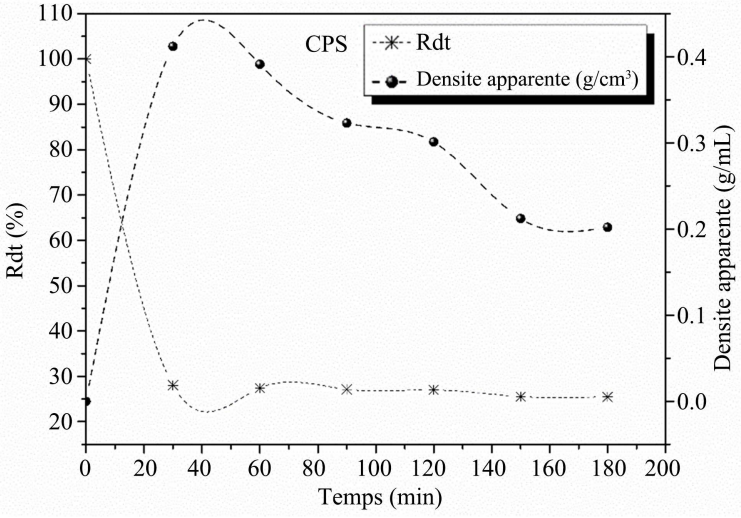

Figure 7. Variation in yield and bulk density of CPS as a function of temperature.

3. Results and Discussion

3.1. Immediate Analysis

The very low ash content of the PET (0.013%) obtained indicates a very large organic matrix (essentially made up of organic matter) with the absence of mineral oxides. the degree of sawdust ash (2.9%) and the high rate of fixed carbon (82.960%), the presence of mineral oxides and a significant carbon matrix. Ashes can be made up of silicates, aluminosilicates, calcium, magnesium, iron, potassium and sodium oxides and are likely to contribute to the catalytic properties of activated carbon [6] [7] [8]. The absence of uncarbonized compounds and the high carbon content in these samples indicate good carbonization of the precursor material [9] [10]. The prepared material therefore consists essentially of carbon. The high volatile matter content (95.7%) of PET has the following advantages for the elaboration of activated carbon: high degree of graphitization, high superior calorific value (PCS) and high number of functional groups [10] [11]. Table 1 also reveals a very low moisture content (0.019%) of PET, for this purpose we would speak of the absence of water, compared to that of sawdust, the activated carbon produced could have a high superior calorific value (PCS) [10]. Immediate tests of the mixture of 1/2 of polyethylene terephthalate with sawdust (CPS), show an increase in the rate of ash (0.960%), humidity (3.150%), carbon (31.70%) and a reduction in volatile matter (64.190%) comparable to PET results. This shows the contribution of sawdust to the characteristic of composite activated carbons. These results suggest that PET and white Bossé sawdust are compatible for the production of composite activated carbon.

3.2. Determination of Pyrolyze Parameters and Study of Thermal Reactivity

3.2.1. Effect of Moisture in the Sample

Figure 1 represents the variation of the humidity rate as a function of the drying temperature, relating to the pyrolysis of sawdust, PET and CPS (mixture PET + Sawdust). We note that the water loss of SC and CPS increases with temperature, unlike that of PET whose variation is substantially zero. Without heat treatment, sawdust alone contains about 7% water. It is therefore sawdust from wood that is poor in water compared to other species such as: green wood and sapwood which can contain up to 25% water. This is a consequence of the fact that the humpback is a tropical tree that has a great ability to adapt to arid climate and drought.

Table 1. Immediate analysis results.

Materials	C (%)	W (%)	MV (%)	CF (%)
SC	2.900 ± 0.030	9.100 ± 0.100	5.040 ± 0.100	82.960 ± 0.027
PET	0.013 ± 0.006	0.019 ± 0.002	95.700 ± 0.020	04.268 ± 0.009
CPS	0.960 ± 0.020	3.150 ± 0.100	64.190 ± 0.3100	31.70 ± 0.443

Furthermore, it can be noted that PET has a substantially zero humidity rate, which can be justified by its crystalline structure, preventing the attachment of the water molecule. SC, PET and CPS baked at 90˚C still contain respectively; 4.34%, 0.0015% and 2.03%, of water. According to Saastamoinen [11], a sample is only completely dry if the boiling point of water is exceeded. Thus, around 110˚C, anhydrous materials are obtained which quickly regain a certain humidity as soon as it is exposed to the open air. In order to avoid problems related to the humidity level in the pyrolysis ovens and for a better interpretation of the results, we processed the data under dry bases and the baking temperature was stopped at 110˚C for a duration of 24 hours, because this temperature does not degrade the nature of the materials even less its structure. In short, it does not engage a complex thermal reactivity, but the only reaction involved is dehydration.

3.2.2. Effect of Sample Mass

One can observe in **Figure 2**, a variation of the loss of mass in relation to the initial mass of the three precursors at 500˚C, 600˚C, 700˚C. This shows the greatest mass losses for the PET samples, between 87.19% and 96.05%, followed by that of the mixture, between 47.33% and 64.37%. And the lowest are observed, with those of sawdust (from 24.02% to 62.6%). However, here we can say that the influence of the mass is not great, given the slight difference between the losses by temperature. Indeed, according to Delmon [12], the path of heat energy inside a pulverulent mass would be represented by a complicated network thus creating a heap effect. On the other hand, among the gaseous molecules resulting from the pyrolysis of the three precursors, there are polymerizable products. These could form large molecules that are likely to be trapped between the grains of the sample when the pile grows in size.

Taking this result into account, all subsequently processed samples will have an initial mass in the order of 5 mg to reduce the relative error due to sensitivity and to control the thermal diffusion phenomenon due to the heap effect.

3.2.3. Effect of Particle Size

Note that during sawdust pyrolysis, the 75 and 300 µm curves have the same appearance and that the differences are insignificant, even if the diameter of the grains is varied by a factor of three. The curve for particles smaller than or equal to 630 µm deviates from the other two, thus reducing the loss of mass. The curve displays a different thermal behavior from those of 75 and 300 µm. We can respectively see the increase in weight loss from 26% to 36% in the interval from 500˚C to 600˚C followed by the decrease from 36% to 27% from 600˚C to 700˚C. These results show that the speed of heat propagation in the lignocellulosic material depends on the particle size. We observe a low thermal diffusivity of 600˚C to 700˚C, reflecting the slowness of the heat front to pass through the thickness of the material. The weight drop between 36% to 27% in the interval of 600˚C to 700˚C can be attributed to the phenomenon of thermal inertia. On the basis

of these results and to better minimize the physical constraints such as the intragranular diffusibility of the volatile matter and the homogeneity of the temperature in the grains, all the experiments, the continuation, will be carried out on samples of the same order of mass, and with a particle size of 75 μm in diameter.

3.2.4. Effect of Heating Rate

From **Figure 4**, we notice that when we vary the heating rate and keeping all the other factors constant, the mass loss at the end of the reaction is approximately the same depending on each precursor material. Nevertheless, it has been demonstrated that the heating rate has a strong influence on the nature of the reaction products, both for the volatile matter and for the solid residue, as well as on the kinetic parameters of the chemical reaction [5] [6].

Indeed, according to the temperature of the reaction medium and according to the time spent in the reactor, the gaseous products which are released can degrade into small molecules of smaller sizes, polymerize or condense, then attach themselves to the solid residue [6].

These fixed products are released when the temperature rises further. On the other hand, we notice a delay in the thermograms towards low temperatures as the heating rate decreases. This phenomenon is very often observed in the literature [3] [7] [8].

This delay is caused by the energy supplied to the system which gains importance when the heating rate decreases, because even if the heat flow is low in the case of low heating rates, the residence time is too high to that the sample stores enough energy to react quickly and break the chemical bonds which break when the energy of their formation is reached.

In addition, one can note the slight shift in the loss on ignition between the speeds. We notice that when we increase the heating rate, the mass loss increases slightly, reflecting the significant thermal change. This leads us to say that the pyrolysis of PET, SC and CPS is a very sensitive reaction. This during the speed of 10˚C/min has been shown to be optimal.

3.2.5. Effect of Retention Time

Retention time is a very important factor in the carbonization process. Indeed, this factor considerably increases the porous volume of materials during carbonization [3]. In this sense, the influence of the heating time on the yield of carbonization and on the apparent density of polyethylene terephthalate (PET), sawdust (SC) of wood and mixture (CPS) was studied.

Figures 5-7 represent the variation of the carbonization yield and of the apparent density as a function of the heating time. We notice that the yield decreases with the residence time in the reactor. This means that the longer the carbonization time, the more the yield of solid residue decreases. Indeed, this phenomenon is accompanied by a degassing which is at the origin of this decrease. It can be noted that the yield of the three precursors stabilizes from 60

minutes and remains almost constant after a reaction time of between 60 and 180 minutes for PET, SC and CPS. This time can be considered as the end time of thermal reactivity of the materials where the heating time seems to have no more effects on the materials. These results corroborate those obtained by Elabed [13], Tchakala [14] and Abo [15] during the activation of bagasse and corn cobs with phosphoric acid.

In addition, the variation of the apparent density shows a decrease as a function of the increase in the residence time of the materials in the reactor. As the carbonization time increases, the bulk density decreases. It is noted, for the lignocellulosic material that the apparent density stabilizes from 60 minutes. It varies very slightly if we further increase the retention time in the reactor. This could be explained by the fact that beyond one hour of treatment, the decrease in the thermal reactivity of the precursor. A thermal stability begins to manifest itself in the structure of the carbonaceous solid and on its surface. This promotes the crystallinity of the material if the residence time is prolonged leading to a decrease in porosity and consequently an increase in the apparent density of the activated carbon [11]. The same phenomenon is observed for the plastic material as well as the mixture from 150 minutes.

These results show a thermal insatiability of polyethylene terephthalate which gives it a good thermal reactivity, unlike sawdust whose stability is faster (1H). A residence time of less than one hour is insufficient for the reaction limit of the three precursors and the temperature. One hour seems to be the ideal treatment time since, beyond the latter, the porosity of our materials decreases.

4. Conclusion

The optimization of the pyrolysis conditions of polyethylene terephthalate (PET), sawdust (SC) and the sawdust/polyethylene terephthalate (CPS) mixture was carried out on the basis of the evolution of the mass loss. It was revealed that the water loss of SC and CPS increases with temperature, unlike that of PET whose variation is essentially zero. Without heat treatment, sawdust alone contains approximately 7% water. This allowed us to say that sawdust from Guarea thompsonisawdust wood is poor in water compared to other species. The dry bases were obtained at 110˚C for 24 hours, without any degradation of the structure of the materials, much less its structure. The study of the variation in mass reveals the greatest mass losses for the PET samples, between 87.19% and 96.05%, followed by that of the mixture, between 47.33% and 64.37%. And the lowest are observed, those of sawdust (from 24.02% to 62.6%). However, where we can observe that the influence of the mass is not great, considering the slight difference between the losses by temperature. The mass which proved to be optimal and whose influence of thermal diffusion due to the heap effect is low is 5 mg. On the basis of these results and to better minimize physical constraints such as the intragranular diffusibility of the volatile matter and the homogeneity of the temperature in the grains, particles of sizes less than 75 μm in diameter, these have

proven favorable to this condition. It was noted the slight shift in the loss on ignition between the speeds and the speed of 10˚C/min proved ideal.

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Conflicts of Interest

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

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