

Characterization of Recycled Polypropylene Composite Material Reinforced with Wood Flour using SEPS-g-MAH as Coupling Agent

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Abstract— A composite material was formed using recycled polypropylene (PPr) as matrix and wood flour of Andean oak (*Quercus Humboldtii*) as reinforcing material. The adhesion between these components of different nature, was improved with an alkali treatment to wood flour and the coupling agent block copolymer of Styrene-Ethylene-Propylene-Styrene grafted with maleic anhydride (SEPS-g-MAH). Four types of composite were formulated, keeping constant the amount of wood flour respect to PPr and varying the percentage of SEPS-g-MAH in 0%, 3%, 5% and 7%. The compatibilizing effect of SEPS-g-MAH was evaluated by moisture absorption test, hardness test, differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) of each composite. The alkali treatment in the wood flour decreased the particle size and increased specific contact area. The water absorption decreased and some thermal properties improved at high content of coupling agent; however a clear trend was no significant between content of SEPS-g-MAH in the composites on the Shore D hardness and degradation temperatures.

Keyword- composite material, recycled polypropylene (PPr), wood flour, coupling agent, thermal stability

I. INTRODUCTION

Over the last years, the marked of innovative materials and technologies for mass consumption have been growing; but with these, pollution linked to waste generated by household and industrial packaging also has increased. In 1992, 2.9 billion urban residents generated 0.64 kg per person at day of solid waste; in 2012, the three billion residents generated 1.2 kg/day, whereas projections for 2025 are 1.42 kg per person at day of solid waste [1]. The interest in the use of composites of recycled polymer matrix with reinforcing natural fiber has increased in the last three decades as alternative in recovery of plastic solid waste (PSW) for applications in the area of the construction and automobile parts industries [2-5].

The success of the compound materials is based in their synergistic characteristics that exceed to those of its two primary constituents, this depend on strict control of certain processing conditions and the essential presence of a coupling agent that allows a proper adhesion between the phases of different chemical nature (between an apolar polymeric structure and a polar structure in the wood) to provide a physicochemical and mechanical features in the material the above with the aim of leading to a material with mechanical and physicochemical features that enable it to be considered a viable option for reusing waste materials through a feasible production process. This research focused on the preparation and characterization of a wood polymer composite (WPC) of recycled polypropylene (PPr) and wood flour Andean oak (*Quercus humboldtii*) that is considered, together with pine, between most popular types of wood used to make furniture [6], but it presents less water absorption at the same processing conditions and relation of wood flour-matrix [7]. Copolymer of Styrene-Ethylene-Propylene-Styrene (SEPS) grafted with Maleic Anhydride (MAH) was used as coupling agent. Processability of composite materials was improved with the addition of a lubricant as Zinc stearate [8].

II. MATERIALS AND METHODS

A. Materials

Andean oak (*Quercus humboldtii* Bonpl., Fagaceae) wood flour was used as reinforcing in the composite. The materials used for treatment of wood flour were distilled water, acetic acid and a solution 1.6 M of sodium hydroxide (NaOH) PPr was used as the matrix of the composites; PPr pellets had dark appearance with mean diameter of the particles of 2.1975mm, density of 0.7866 g/mL and MFI of 14.2060 g/10min. Styrene-ethylene-propylene-styrene copolymer grafted with maleic anhydride (SEPS-g-MAH) was used to improve adhesion between the phases and therefore the properties of composite material and it was synthesized by reactive extrusion [9]. For the extrusion of composite, zinc stearate ($Zn(C_{18}H_{35}O_2)_2$, ZnSt) was used as a lubricant to facilitate processing

B. Treatment of wood flour

The wood flour was separated by size using a sieve shaker EDIBON 0005108 QMS with standard sieves ASTM E 11/95, considering that the properties of the composite improve with the decrease of particle size of the wood flour. The trapped material on 35-mesh sieve was discarded according with recommended size specifications [10].

The wood flour was dipped in 1.6 M NaOH solution for 48 hours, neutralized with acetic acid ($C_2H_4O_2$) and washed with sufficient distilled water to remove any excess reagent. Finally, the wood flour was dried in forced air oven ISORTERM ESCO at 70 °C for 24 hours. [11].

C. Characterization of Wood flour

Corresponding values of mean diameter, number of particles and surface area, were calculated applying the following equations:

$$\text{Mean diameter: } D_M(\text{mm}) = \frac{1}{\sum \frac{\Delta\Phi_i}{D_{Pi}}} \quad (1)$$

$$\text{Number of particles: } N_P = \frac{1}{a \times \rho_p} \times \sum \left(\frac{\Delta\Phi_i}{D_{Pi}^3} \right) \quad (2)$$

$$\text{Surface area: } A_s(\text{mm}^2) = \frac{6}{\rho_p \times \Phi_S} \times \sum \left(\frac{\Delta\Phi_i}{D_{Pi}} \right) \quad (3)$$

Here, $\Delta\Phi_i$ is the mass fraction retained on each sieve, $\overline{D_{Pi}}$ is the arithmetic mean diameter of particles retained in the *i*th sieve, ρ_p is the particle density, Φ_S is the sphericity factor related to particle volume [11]. These values were determined before and after treatment of flour wood treatment with sodium hydroxide and acetic acid to identify the influence of the treatment about the particle size.

D. Obtaining composite material by extrusion

Composite of PPr and wood flour with 0%, 3%, 5% and 7% of SEPS-g-MAH were mixed and processed by extrusion, at screw speed range of 25 to 50 rpm and temperature of 180-200 °C, to determine the effect of the amount of SEPS-g-MAH coupling agent on properties as the hardness, moisture absorption and thermal behavior of material. The composite material obtained was cut into pellets and stored in aluminium bagged to avoid any absorption of humidity.

It was determined that the ignition temperature of the wood flour was 210°C. The amount of wood corresponded to 45% in all samples, as shown in Table I. This percentage was chosen due to this ratio shows good features in the composite, saving costs without compromising the thermal stability [10]. The lubricant amount was 3% to enhance processability of the composite, without implying a significant increase in water absorption. The addition of the lubricant also influences a uniform distribution of wood flour in the matrix [8].

TABLE I
Composition of composite extruded samples

	N° of blends			
	1	2	3	4
Lubricant (%)	3	3	3	3
SEPS-g-MAH (%)	0	3	5	7
Wood flour (%)	43.65	42.3	41.4	40.5
PPr (%)	53.35	51.7	50.6	49.5

E. Characterization of composite material

1) *Water absorption test*: The composites samples for each percentage of SEPS-g-MAH were weighed and immersed in distilled water at room temperature for 24 hours, under controlled temperature and humidity. Then, the samples were removed from the water and superficially dried with filter paper to eliminate the excess water. The samples were weighed to calculate the percentage of water absorbed according to the following equation [12, 13]:

$$\%M = \frac{M_{wet} - M_{dry}}{M_{dry}} \times 100 \quad (4)$$

Where %M is the percentage of water absorbed (%), M_{wet} is the wet mass after immersion in water at 24 hours (g) and M_{dry} is the initial dry mass (g).

2) *Hardness test*: Hardness of the four specimens of composite material was determined according to norm ASTM D2240, which describes methods for testing the hardness properties in plastics. Durometer PCE-D Shore D was used for to measure hardness. Five measurements of hardness were taken in each test specimens of 6mm in thickness, with these data the mean was calculated [14]

3) *Differential scanning calorimetry (DSC)*: Composites with different compositions of SEPS-g-MAH (0 wt%, 3 wt%, 5 wt% and 7 wt%) were characterized in a differential scanning calorimeter (DSC), Q200 V24.4 Build 116 (Universal V4.7A TA Instruments, New Castle, USA), according to norm ASTM D3418. Samples between 5 and 10 mg of sample were heated from 25 °C to 200 °C at a heating rate of 10 °C/min to determine the temperatures of melting and crystallization. The first cycle was used to erase any thermal history of the sample; the cooling curve was used to know the crystallization temperature; and the second heat cycle was used in this paper to provide the melting temperature [15].

The degree of crystallinity (X_c) of the composites was calculated by the following equation [16, 17]:

$$X_c(\%) = \frac{\Delta H_m}{\Delta H_m^0 \times w} \quad (5)$$

Where ΔH_m is the heat of fusion of, $\Delta H_m^0 = 138 \text{ J/g}$ is the heat of fusion of 100% crystalline polypropylene [16, 17] and w is mass fraction for PPr in the composites, this values can be observed in Table I.

4) *Thermogravimetric analysis (TGA)*: TGA analysis was performed on a Perkin Elmer TGA 7 Thermogravimetric Analyzer with Pyrex software. The temperature range chosen was from room temperature (29 °C) to 960 °C with heating rate of 10 °C/min, using pure nitrogen as carrier gas at 40 mL/min. Samples of approximately 10 mg were placed in open alumina pans to analysis the degradation temperature [18].

III. RESULTS AND DISCUSSIONS

A. Characterization of Wood flour

Table II shows that the particle size decreased in the treated wood; this observation is evident by the increase in retained mass fractions ($\Delta\emptyset$) between mesh sizes of 140 and 230. Although the total mass of wood decreased with the treatment, the particle distribution was kept constant between mesh sizes of 45 to 70, where the highest amount of wood was contained.

TABLE II.
Retained fraction ($\Delta\emptyset$) in each mesh of wood flour before and after treatment

N° Mesh	$\Delta\emptyset$ wood flour before treatment	$\Delta\emptyset$ wood flour after treatment
35	0	0
45	0.2150	0.2031
70	0.5872	0.5814
100	0.0811	0.0713
140	0.0751	0.0899
230	0.0416	0.0543
TOTAL	1	1

The volume factor (α) is used to determine the particle number (N_p) and the sphericity coefficient (Φ_s) is used to calculate the ratio of the total surface (SP) of a particle to particle volume. This parameter was approximated as unity, due to the material was

very fine (with form approximated to a sphere). The properties of wood flour were calculated with the equations (1), (2) and (3) and these are show in the Table III.

TABLE III
Properties of the wood flour before and after treatment

Properties	Wood flour before treatment.	Wood flour after treatment.	Difference (%)
Mean diameter (mm)	0.24	0.23	-3.81
N_p/g	253885.91	269738.70	6.24
Surface area (mm^2)	22264.72	20894.34	-6.15
Specific Surface area (mm^2/g)	225.81	323.94	43.46

From the above data it concluded that the average particle size was decreased in the treated wood with $C_2H_4O_2$ and NaOH; this treatment allowed the elimination of components such as hemicellulose, fats and waxes, causing mass losses in the particles of wood flour. The number of particles and the specific surface area were increased with treatment, due to the diameter reduction that allowed a better packing or distribution of the material per unit mass. The microfibrils in the wood structure, suffered a longitudinal contraction in presence of NaOH [19]. The use of greater wood quantity was necessary due to mass loss caused by the treatment. The mass loss was 34.58% after treatment.

The NaOH treatment is one of the most used chemical mechanisms of modification in which the hemicellulose and other impurities present in the wood flour are removed. This alkali treatment produces a less dense interfibrillar region while providing more homogeneous aspect [20] with more exposed cellulosic surface [21]. The treatment also contributes to the elimination of volatile compounds whose degradation generates micro-voids in the composite [20]. The wood flour treatment was realized to ensure best conditions of processing, and the granulometry allowed establish the effect of this procedure on the characteristics of the wood.

B. Characterization of composite material

1) *Water absorption test:* The water permeability degree of the composites decreased with increasing coupling agent content, as it is shown in the Fig. 1, that occurred due to an increase in molecular interactions between the chain of PPr with the surface hydroxyl (-OH) groups of wood in presence of coupling agent, hence the interfacial adhesion and the stress distribution were increased. The affinity to water was reduced due to the low availability of hydroxyl groups in the composite. The coupling agent improved the adhesion at hydrophobic matrix with the wood filler. The Fig. 1 shows as the moisture absorption was lower with the increase of coupling agent at 0 and 3% of SEPS-g-MAH, however this effect was less significant with higher concentrations of SEPS-g-MAH, the difference between 5% and 7% of SEPS-g-MAH was only 1%.

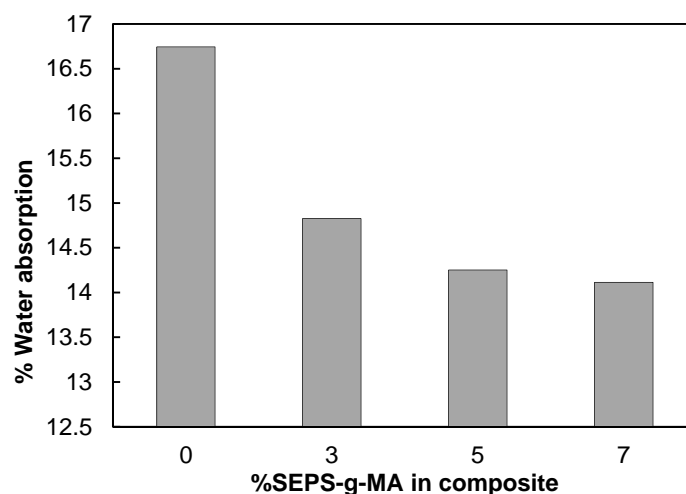


Fig. 1. Effect of SEPS-g-MAH content on the water absorption of PPr and wood flour composite.

The absorption of water takes place mainly in the wood surface through its gaps and flaws [22], this generates instability, poor mechanical properties [23] and favorable conditions for fungal growth [24]. the lowest percentage of water absorption was at 7% of SEPS-g-MAH (14.11%); however, this value is above the typical range of moisture absorption between 0.7 and 2%, after 24 hour of immersion, for WPC [24-27], due to the high content of wood flour (around 45% wood flour and 50% PPr) of composites analyzed; the absorption percentage increase with high amounts of filled [28]; Other data have also shown the same trend, wood flour-PP composites at filler loading of 50 wt% exhibited higher water absorption after 24 hours immersed [29]. According to Klyosov, the porosity of the materials is another factor that increases the moisture content due to the presence of chains of pores in the exposed wood flour. The composites are porous materials due to the initial amount of moisture in the phases and the volatilization of some components during processing, characteristic that make them susceptible to absorption and desorption of water and to the deformation the material under changing environmental conditions which contributes to poor interfacial adhesion and lower thermal and mechanical properties [30].

The wood particle size also influences moisture absorption. Although, the reduced size of the wood flour improves its distribution on the matrix and the uniformity, the small particles tend to agglomerate in the composite, generating greater water absorption. Moreover the wood treatments can increase the surface area of WPCs, resulting a higher contact of the free surface hydroxyl groups with water [31].

2) *Hardness test*: Table IV shows that the composites with 3% and 7% of SEPS-g-MAH exhibited the highest and lowest hardness value, respectively. An increase in amount of coupling agent does not guarantee a better performance in the material hardness. The highest data deviation, of the composite without SEPS - g - MAH, may be due to the increase of heterogeneity by the absence of coupling agent.

TABLE IV
Shore D hardness for composites of wood flour with PPr

% SEPS-g-MAH	Hardness	Standard deviation
0	69.28	1.74
3	69.82	0.98
5	69.60	0.49
7	69.20	0.80

Previous studies demonstrating that the hardness increases with increasing the wood flour content [32] and it is affected by the type of used coupling agent [33]; however, if these factors are kept constant, the hardness of WPCs does not show a marked dependence with coupling agent amount [33, 34]; similar results were found in the present research, the values of hardness were quite close at different coupling agent content.

Shore D Hardness values of WPCs are within the range of 60-72 according with other articles [32-34], even with higher degree of grafting, between 0.94 and 1.4 wt% [33].

3) *Differential scanning calorimetry (DSC)*: Fig. 2 shows the DSC thermograms of PPr and wood flour composites with different contents of coupling agent. Two melting peaks were presented in the heating second scan for all the composites, this behavior was also observed by Pracella et al. [35]. The first peak is observed near 145°C for the composites and decreases its intensity by increasing the amount of coupling agent, this may be due to fusion-recrystallization-fusion process, by the imperfections formed during the crystallization [36], this behavior is less visible to other compositions having coupling agent, which confirms his role in thermal stability and homogeneity of the composite.

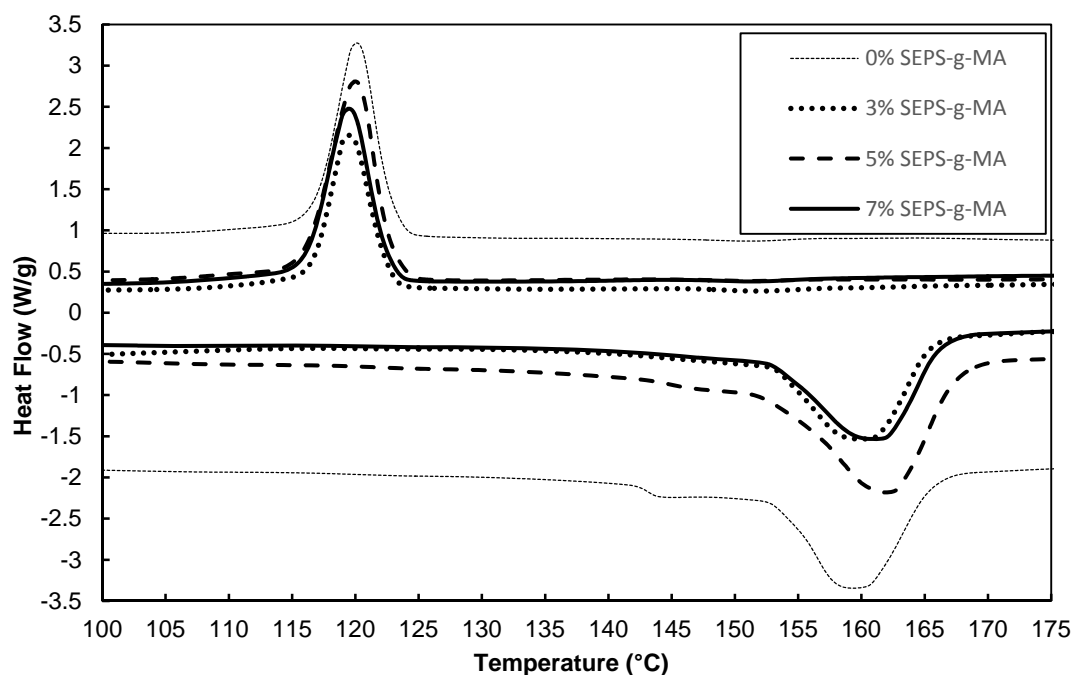


Fig. 2. DSC Thermograms of PPr and wood flour composites at different SEPS-g-MAH contents.

The values for melting and crystallization temperature, fusion and crystallization point, and degree of crystallinity, are summarized in Table V.

TABLE V
Information provided by the thermogram of composite material

%SEPS-g-MAH	Melting temperature (°C)	Enthalpy of fusion (J/g)	Crystallization temperature (°C)	Enthalpy of Crystallization (J/g)	Crystallinity (%)
0%	160.01	27.89	120.09	56.26	46.19
3%	160.69	25.18	119.46	45.93	43.14
5%	162.34	37.62	119.98	62.94	65.85
7%	161.65	27.09	119.47	55.73	48.47

As shown in Table V, the melting temperatures of composites did not substantial change with increased of coupling agent, similar results were obtained by others authors [16, 37]. The melting temperature of PPr (165.47°C) is higher than that of other composites [16]. The same trend was also found for the enthalpy of fusion the data suggest the absence of a significant dependence with the amount of coupling agent [37]. The enthalpy of fusion of PPr (51.60 J/g) is higher than of the composites due to the wood flour in the composite, absorbs more heat energy than the thermoplastic polymer [38].

The crystallization temperature variations were lower than 0.3%, furthermore the PPr crystallization temperature was greater (121.68°C) than for composites [38, 39]; Bouza et al. suggested that the decrease in the crystallization temperature can be attributed to the reduction of nucleating activity by the wood flour in the crystallization of polypropylene due to presence of the coupling agent [39]. This is indicative of a better compatibility between wood flour and the PPr [36]. Enthalpies of fusion and crystallization were not clearly affected by the content of SEPS - g -MAH.

All composites showed higher crystallinity compared with PPr, corresponding to 37.39%. The composite, with 5% of SEPS-g-MAH, exhibited the higher values in melting point and the crystallinity due to the increase of the amount of coupling agent [16]. However, composition of 7% of SEPS-g-MAH in composite was less favorable on properties studied because of an increase in branches that limited the contact surfaces between the phases [40] and poor adhesion between the polymer matrix and wood flour, which generates poor thermal performance.

4) *Thermogravimetric analysis (TGA)*: Fig. 3 shows the first mass loss of composite, close to 100°C, corresponding to loss of moisture. Composites degradation occurs in two stages [16], the degradation of some wood components was evidenced between 170°C and 280°C [12, 36], and degradation of the polymeric matrix

occurs from 320-480 °C (corresponding to the greatest loss of mass), this indicates degradation of the saturated and unsaturated carbons of polypropylene. In another study, similar results were obtained with a composite of PP with banana fiber using PP-g-AMH as coupling agent, the greatest loss of mass occurred in the range of 288 to 442 °C, which is similar to the material obtained in this study [41].

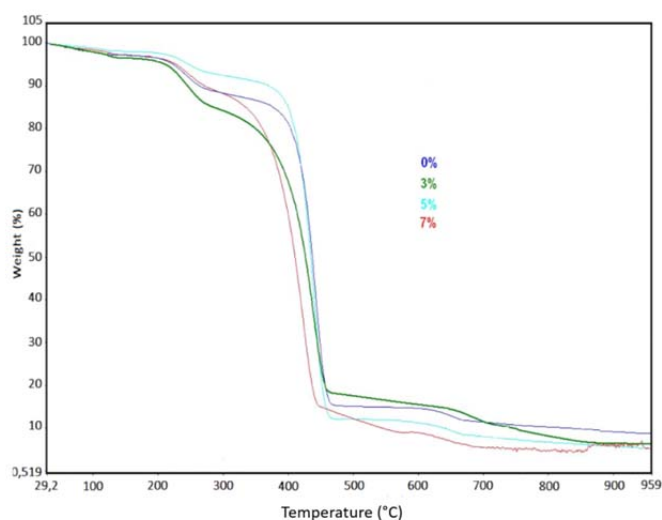


Fig. 3. TGA for each of the compositions of SEPS-MAH in the composite.

An interesting behavior was observed after 700 °C for the composite with 7% of coupling agent, it was evidenced other composition, the degradation of material and an increase of mass after 870 °C, which could be an indication that a chemical reaction was carried out. Botros reported that in the presence of moisture, the MAH reacts with ZnSt to form the ionomer of Zn; steric acid may be also formed, which stimulates the degradation of the composite [42]. The composite without compatibilizer generated higher waste content.

The trend was not clear between the compatibilizer content and degradation temperature of the composites, as shows Table VI; similar results was found by Bouza et al., for composites with high content of wood in WPC with silane as coupling agent [39].

TABLE VI
Degradation temperature of composites

%SEPS-g-MAH	Degradation temperature (°C)
0%	417.13
3%	400
5%	409.23
7%	378

IV. CONCLUSION

Alkaline treatment allowed the reduction of wood flour particle size and increased specific surface area, this improved the dispersal in the matrix of the composite. Mass loss of wood due to treatment was 34.58 %.

The water absorption decreased in the composites with the increase of the content of SEPS-g-MAH, however the influence on this property was stronger at lower percentages of compatibilizer. The percentages of water absorption obtained by immersion for 24 hours, were higher compared with other composite materials, this can be explained by the high content of wood, the porosity of the material, wood particle agglomeration and/or increase in wood surface area due to treatment with NaOH.

Although it cannot establish a clear relationship between the amount of coupling agent and Shore D hardness in the proposed composite materials, the values obtained for this property have a value according to studies with other types of compatibilizer in WPC.

Two melting peaks were observed at the second heating scan; the intensity of the first peak decreased with increasing the amount of coupling agent, which it allowed to suggest the influence of SEPS - g -MAH in thermal stability and homogeneity of the composite. The more marked peak, second peak corresponding to the melting temperature of the composite, did not have significant change with the addition of coupling agent, these melting temperatures did not exceed the melting temperature of PPr used as matrix, and similar behavior was observed with heat of fusion. The slight decrease in the crystallization temperature, suggest a compatibilizing effect of SEPS - g -MAH in the composite, but it was not possible to establish a clear influence on the amount of

coupling agent on enthalpy of crystallization. The addition of SEPS-g-MAH resulted in a clear increase in crystallinity of the composites, his presence as compatibilizer improved the interfacial adhesion and therefore some thermal properties; however a high amount of this SEPS - g -MAH did not ensure a proportional increase in its thermal performance.

The relationship between the amount of coupling agent and degradation temperature of the composite, was not unclear. The composite material with the greatest content of SEPS-g-MAH was degraded at lesser temperature, and it generated higher amount of decomposition waste.

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