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Comparison between two different pretreatment technologies of rice straw fibers prior to fiberboard manufacturing: Twin-screw extrusion and digestion plus defibration

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ABSTRACT

The present work compares two different pretreatment technologies, i.e. twin-screw extrusion, and steaming digestion plus defibration, for producing a thermo-mechanical pulp from rice straw for fiberboard manufacturing. Five liquid/solid ratios from 0.43 to 1.02 were tested for twin-screw extrusion pretreatment, while liquid/solid ratios from 4 to 6 were used for digestion pretreatment. Energy consumption, and characteristics of the extrudates (twin-screw extrusion) and pulps (digestion) (including fiber morphology, chemical composition, thermal properties, apparent and tapped densities, as well as color) were the analyzed parameters for the resulting lignocellulosic fibers. The results showed that liquid/solid ratio had influence on energy consumption of the equipment for both defibrating methods For the twin-screw extrusion method, a lower liquid/solid ratio required more energy while for the digestion plus defibration the effect was the opposite. The corresponding total specific energy consumption ranged from 0.668 kW h/kg to 0.946 kW h/kg dry matter for twin-screw extrusion, and from 6.176 kW h/kg to 8.52 kW h/kg dry matter for digestion plus defibration. Thus, the pulping method consumed about nine times more energy than that of the twin-screw extrusion. In addition, for twin-screw extrusion, the liquid/solid ratio did not have a substantial effect on fiber characteristics with similar chemical compositions and thermal properties. For twin-screw extrusion, the energy consumption was 37% reduced when the liquid/solid ratio was increased from 0.43 to 1.02. Instead, for digestion plus defibration, the energy increase was 38% when the liquid/solid ratio increased from 4 to 6.

1. Introduction

Rice (*Oriza Sativa* L.) is cultivated to feed more people and animals over a longer period than any other crop. As far back as 2500 B.C., rice has been documented in the history books as a source of food and for tradition as well (Thomas, 1997). Rice straw is a by-product of rice crop with a straw to grain ratio of 1.4 (Kim and Dale, 2004). In terms of total production, rice is the second most important grain crop in the world after maize. The world annual rice production in 2014 was about 741 million tons (FAOSTAT, 2016). It gives an estimation of about 1139 million tons of rice straw per year worldwide (FAOSTAT, 2016), and a large part of this is used for cattle feed, for bioethanol production, or incorporated into the soil as an organic amendment. Possible uses for rice straw are limited by its low bulk density, a slow degradation in the soil, the harboring of rice stem diseases (the possible transmission of diseases to the future crop), and a high ash content which can be a problem for subsequent ethanol or energy production (Binod et al., 2010). Currently, field burning is still the major practice for removing rice straw, particularly in less developed countries, causing air pollution, thus affecting public health (Mussatto and Roberto, 2004) and contributing to the global warming (Kanokkanjana and Garivait, 2013; Sarnklong et al., 2010). According to Kanokkanjana and Garivait (2013), about 56% of the total rice straw production was burned in Thailand in 2010. As climate change is extensively recognized as a threat to development, there is a growing interest to find alternative uses for rice straw.

All plants including rice straw have the form of a heterogeneous complex of carbohydrate polymers. Cellulose and hemicelluloses are

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densely packed by layers of lignin, which protect them against enzymatic hydrolysis. Thus, a pretreatment step is necessary to break lignin seal, until exposing cellulose and hemicelluloses for a subsequent enzymatic action or contributing to the biomass defibration (Vandenbossche et al., 2016, 2015, 2014).

Several researchers have investigated the use of rice straw and other agricultural wastes as fiber source in the composite industry (El-Kassas and Mourad, 2013; Evon et al., 2012; Halvarsson et al., 2008; Li et al., 2010; Pan et al., 2010; Theng et al., 2015a; Wu et al., 2011; Zhang and Hu, 2014; Zhao et al., 2011), in particular to produce fiberboards by thermopressing, the latter being usable for furniture or in the building industry. Different methods for fiber pretreatment were tested, i.e. chemical, mechanical, and thermo-mechanical pretreatment to obtain resources for their purposes. Recently, Vandenbossche et al. (2016, 2015, 2014) used the twin-screw extrusion technology for conducting the thermo-mechanical and thermo-chemo-mechanical pretreatment of different lignocellulosic biomass sources, in the case not for the subsequent manufacture of composite materials but for the production of second-generation bioethanol using a biocatalytic action. Evon et al. (2015, 2014, 2012, 2010a, 2010b) also produced self-bonded fiberboards from the cake generated during the biorefinery of sunflower whole plant using a twin-screw extruder. In addition, Theng et al. (2015a) prepared a thermo-mechanical pulp from corn biomass by digestion plus defibration to produce binder-free fiberboards. Migneault et al. (2010) produced medium-density fiberboards using thermo-mechanical pulps from different pulping processes. Lastly, Mancera et al. (2012, 2011) developed fiberboards using thermo-mechanical pulps from different agricultural wastes, all produced using steam-explosion. However, there is no scientific literature dealing with the energy consumption of equipment for fiber preparation using different defibrating technologies prior to board manufacturing.

The present paper aimed to explore the appropriate and beneficial technology for fiber preparation as a raw material for fiberboard manufacturing using the same batch of rice straw, comparing two different techniques: twin-screw extrusion and digestion plus defibration, without any chemical agent addition. A Clextral (France) Evolum HT 53 twin-screw extruder model and a digester reactor (designed by LEPAMAP, University of Girona, Spain) with Sprout-Waldron defibrator (model 105-A) were used in this study. The overall energy consumption of the equipment and the properties of the pretreated rice straw fibers (i.e. fiber morphology, apparent and tapped densities, chemical composition and thermal stability) from both technologies were compared to provide more options to industrial sectors.

2. Experimental

2.1. Material

Thermo-mechanical fractionation was conducted using a single batch of rice straw (*Oriza Sativa* L.), i.e. the whole plant except the panicle and the grain. The rice straw was of French origin and it was supplied by the JCL AGRI company (Bouge-Chambalud, France). It was harvested in October when the plant maturity was reached. The rice straw was previously crushed using a hammer mill (Elecra BC P, France) fitted with a 6 mm screen. The moisture content of the rice straw was 7.4 ± 0.2% (French standard NF V 03-903).

2.2. Twin-screw extruder

The thermo-mechanical fractionation of the grinded rice straw was conducted using a pilot-scale Clextral Evolum HT 53 (France) co-penetrating and co-rotating twin-screw extruder. The twin-screw extruder had eight modular barrels, each 4D in length (with D corresponding to the screw diameter, i.e. 53 mm), except module 1 having an 8D length, and different twin-screws which had segmental screw elements. Module 1 was cooled by water circulation. Modules 2–8 were heated by electric resistance and cooled by water circulation. The material temperature was measured at the end of modules 2, 5 and 7, and at the beginning of module 8. The material pressure was measured at the end of modules 2, 5 and 7. The screw rotation speed (Ss), the inlet flow rates of grinded rice straw and water (Qs and Q_L, respectively), and the barrel temperature (θc) were monitored from a control panel.

2.3. Thermo-mechanical fractionation of grinded rice straw in the twin-screw extruder

Grinded rice straw was fed into the extruder inlet port using a constant weight feeder (Coperion K-Tron SWB-300-N, Switzerland) in the first module, at a 15 kg/h wet matter inlet flow rate. Water was injected using a piston pump (Clextral DKM Super K Camp 112/12, France) at the end of module 3. After water injection, two series of BL22-90° bilobe paddle-screws (2D in total length) were located in modules 5 to disperse intimately water inside the grinded rice straw. The CF1C reversed simple-thread screws with grooves (1.5D in total length) were positioned at the beginning of module 8 to give an intense shearing/mixing action to the liquid/solid mixture. The screw speed (S_S) was fixed at 150 rpm and the set values for the barrel temperature were 25, 80, 110, 110, 110, 110, 110 and 100 °C at the level of modules 1–8, respectively. The experimental variable of this part of the study was the liquid/solid (L/S) ratio (i.e. Q_L/Q_S), the latter varying in five levels from 1.02 (E_1 extrudate) to 0.43 (E_5 extrudate), as it can be seen in Table 1. This operation condition is in agreement with previous work (Uitterhaegen et al., under review) in order to obtain a good thermo-mechanical extrudate for fiberboard production.

Twin-screw extrusion was performed for 10 min before any sampling to ensure the stabilization of the operating conditions. The operating conditions, including in particular the feed rates of grinded rice straw and water, the temperature along the screw profile and the current feeding the motor, were recorded during sampling and then used for the production cost calculation. Upon achieving steady operation, the extrudate was immediately collected over a period of 10 min to avoid any variability of the outlet flow rate. Sample collection time was determined with a stopwatch. For each liquid/solid ratio tested, sample collection was carried out once and the extrudate was then weighed. Its moisture content was also measured immediately after its collection according to the NF V 03-903 French standard.

The total specific energy (TSE) consumption (in W h/kg dry matter) of the extrusion process is defined as the sum of three specific terms, i.e. (i) the specific mechanical energy (SME), (ii) the specific cooling energy (SCE) and (iii) the specific thermal energy (STE). All these three specific energies are calculated from the recorded data of the operating conditions using Eqs. (1), (2), and (3), respectively.

$$SME = (454 \times I \times \cos\varphi \times S_s/S_{max})/Q_s \quad (1)$$

Where: *I* is the current feeding the motor (A), *cos φ* the theoretical yield of the twin-screw extruder motor (cos φ = 0.95), S_s the screw rotation speed (rpm), S_{max} the maximal screw rotation speed (S_{max} = 800 rpm), and Q_s is the inlet flow rate of dried grinded rice straw (kg dry matter/h).

$$SCE = m \times C_p \times |\Delta T|/Q_s \times 3600 \quad (2)$$

Where: *m* is the inlet flow rate of cooling water (kg/h), C_p the calorific capacity of water (C_p = 4180 J/kg K), and $|\Delta T|$ is the difference in temperature between the inlet and the outlet of the cooling water circuit (K).

$$STE = P \times 1000/Q_s \quad (3)$$

Where: *P* is the heating power. The heating power used in this calculation was the sum of the heating powers of all the heated modular zones along the twin-screw extruder barrel (i.e. modules 2–8). The control panel of the extruder was set to record the heating power as a

Table 1
Operating conditions used for extrudate production and results of the thermo-mechanical fractionation of rice straw biomass in the Clextral Evolum HT 53 twin-screw extruder.

Trials	E_1	E2	E_3	E_4	E_5
Operating conditions					
S_S (rpm)	150.4 ± 1.5	150.4 ± 1.5	150.4 ± 1.5	150.6 ± 1.5	150.6 ± 1.4
Q_S (kg/h)	15.8 ± 0.0	15.8 ± 0.4	15.5 ± 0.0	15.7 ± 0.3	15.2 ± 0.0
H_S (%)	7.4 ± 0.2	7.4 ± 0.2	7.4 ± 0.2	7.4 ± 0.2	7.4 ± 0.2
Q_S (kg/h dry matter)	14.6 ± 0.0	14.6 ± 0.4	14.3 ± 0.0	14.6 ± 0.3	14.0 ± 0.0
Q_L (kg/h)	15.0 ± 0.0	12.7 ± 0.0	10.5 ± 0.0	8.2 ± 0.0	6.0 ± 0.0
Q_L/Q_S (i.e. L/S ratio)	1.02	0.87	0.73	0.57	0.43
W_E (kg/h)	16.0 ± 0.0	13.8 ± 0.0	11.5 ± 0.0	9.3 ± 0.0	7.0 ± 0.0
θ_{C7} (°C)	107 (107.6 ± 0.7)	107 (109.5 ± 0.7)	107 (111.3 ± 0.6)	107 (111.5 ± 0.4)	107 (111.4 ± 0.9)
θ_{C8} (°C)	100 (102.0 ± 0.6)	100 (100.6 ± 0.4)	100 (101.2 ± 0.4)	100 (101.2 ± 0.4)	100 (101.1 ± 1.7)
Extrudate					
Q_R (kg/h)	26.8 ± 0.0	25.1 ± 0.0	22.9 ± 0.0	21.5 ± 0.0	19.0 ± 0.0
H_R (%)	45.3 ± 0.9	41.7 ± 0.1	37.4 ± 0.4	32.2 ± 2.5	26.0 ± 1.9
Q_R (kg/h dry matter)	14.6 ± 0.0	14.6 ± 0.0	14.3 ± 0.0	14.6 ± 0.0	14.0 ± 0.0
W_R (kg/h)	12.1 ± 0.0	10.5 ± 0.0	8.6 ± 0.0	6.9 ± 0.0	4.9 ± 0.0
E_W (kg/h)	3.9 ± 0.0	3.3 ± 0.0	2.9 ± 0.0	2.3 ± 0.0	2.1 ± 0.0
E_W (%)	24.2 ± 0.0	23.9 ± 0.0	25.6 ± 0.0	25.3 ± 0.0	29.9 ± 0.0
Energy consumed					
I (A)	74.6 ± 4.7	81.7 ± 3.4	81.8 ± 4.1	91.0 ± 4.1	113.4 ± 6.7
SME (kW h/kg dry matter)	0.391 ± 0.024	0.428 ± 0.017	0.438 ± 0.022	0.479 ± 0.021	0.620 ± 0.036
m (kg/h)	2370.6 ± 27.7	2432.0 ± 56. 6	2397.3 ± 47.4	2432.4 ± 51.5	2535.6 ± 106.2
|ΔT| (K)	0.73 ± 0.36	0.77 ± 0.34	0.78 ± 0.35	0.79 ± 0.35	1.01 ± 0.42
SCE (kW h/kg dry matter)	0.137 ± 0.066	0.147 ± 0.065	0.152 ± 0.067	0.153 ± 0.068	0.211 ± 0.088
P (kW)	2.04 ± 0.22	1.99 ± 0.25	1.69 ± 0.17	1.66 ± 0.13	1.59 ± 0.24
STE (kW h/kg dry matter)	0.139 ± 0.015	0.135 ± 0.017	0.118 ± 0.011	0.114 ± 0.009	0.112 ± 0.016
TSE (kW h/kg dry matter)	0.668 ± 0.106	0.712 ± 0.1	0.709 ± 0.101	0.747 ± 0.99	0.945 ± 0.141
Production cost (€/kg dry matter)					
Total production cost	0.053 ± 0.008	0.057 ± 0.008	0.057 ± 0.008	0.060 ± 0.008	0.076 ± 0.011

S_S is the screw rotation speed (rpm); Q_S is the solid inlet flow rate (kg/h); H_S is the solid moisture content (%); Q_L is the set value for the liquid inlet flow rate (kg/h); L/S ratio is defined as the ratio of the inlet flow rate of liquid (Q_L) to the inlet flow rate of solid (Q_S); W_E is the real liquid inlet flow rate (kg/h); θ_{C7} (°C) is the barrel temperature of module 7 (set value first mentioned, plus temperature measured during sampling in parentheses); θ_{C8} (°C) is the barrel temperature of module 8 (set value first mentioned, plus temperature measured during sampling in parentheses); Q_R is outlet flow rate of the extrudate (kg/h); H_R is the moisture content of the extrudate, measured immediately after sampling; W_R is the calculated water outlet flow rate in the extrudate (kg/h); E_W is the estimated water vapor outlet flow rate (kg/h and% of the inlet flow rate of liquid water); I is the current consumed by the motor of the twin-screw extruder (A); SME is the specific mechanical energy (kW h/kg dry matter); m is the cooling water flow rate (kg/h); |ΔT| is the difference in temperature between the inlet and the outlet of the cooling water circuit (K); SCE is the specific cooling energy (kW h/kg dry matter); P is the heating power supplied by the twin-screw extruder; STE is the specific thermal energy (kW h/kg dry matter); TSE is the total specific energy (kW h/kg dry matter). The total production cost is defined as the TSE × 0.08 €/kW h (electricity price in France in 2016).

percentage of the maximal value of the heating power available for all the heated modules every 5 s. In this study, the twin-screw extruder had seven heated modules, situated from zones 2–8, with a maximal value for the heating power of 5.0 kW, except in zone 5 where it was 3.4 kW. The heating power of each module was calculated using Eq. (4).

$$P_{module} = M \times P_{max}/100 \quad (4)$$

Where: M is the average percentage of the maximal value of the heating power during sampling (%), and P_{max} is the maximal value of the heating power available for the corresponding heated module (kW).

2.4. Rotary digester reactor and Sprout-Waldron defibrator

The thermo-mechanical pulp of rice straw was conducted using a laboratory scale rotary digester, designed by University of Girona, Spain and a Sprout-Waldron defibrator (model 105-A). The digester had two heating resistances with a heating speed of 1 °C/min and a motor making the digester rotating vertically. On the other hand, the Sprout-Waldron defibrator was used to defibrate the grinded and digested rice straw. The Sprout-Waldron defibrator was used with tap water and equipped with a filtrate bath. Both digester and defibrator had an electric monitor to measure the energy consumption for further production cost calculation.

2.5. Thermo-mechanical pulp (TMP) preparation

Grinded rice straw was fed into the digester with distilled water at liquid/solid ratios of 4, 5, and 6 (P_1, P_2 and P_3, respectively), at the maximal biomass plus water mass (8 kg) per batch, previously heated to 80 °C. The temperature (160 °C), duration (30 min), and liquid/solid ratio (6) profile chosen for cooking was previously optimized for the thermo-mechanical pulp (TMP) preparation from rice straw (Theng et al., 2015b). The digested pulp was then washed with tap water, filtered and moisture content of the material was measured according to the French standard NF V 03-903. Operating conditions such as mass of grinded rice straw and water, production yield of TMP and current feeding the motor were recorded. The masses of solid inlet and liquid inlet were calculated using Eqs. (5) and (6), respectively.

$$m_w = m_d/(100 - \%MC_i) \times 100 \quad (5)$$

Where: m_w is the mass of humid inlet (kg); m_d is the mass of dry inlet (kg dry matter); and $\%MC_i$ is the moisture content of the inlet (%).

$$m_L = L/S \times m_d - (m_w - m_d) \quad (6)$$

Where: m_L is the mass of water inlet (kg); and L/S is the liquid/solid ratio, defined as the ratio of the water mass (including both liquid water and moisture inside rice straw) to the dry solid mass.

The production yield was calculated following Eq. (7).

$$\%Yield = (m_p \times (100 - \%MC_p)/100)/m_d \times 100 \quad (7)$$

Where: m_P is the mass of digested pulp (kg) and $\%MC_p$ is the moisture content of pulp (%).

The digested pulp was then passed one time through the Sprout-Waldron defibrator with addition of spraying tap water, and filtered again to eliminate the excess of water.

2.6. Chemical composition characterization

Before each analysis, the materials (i.e. rice straw, extrudates (twin-screw extrusion) and TMP (digestion plus defibration)) were milled using a Foss (Denmark) Cyclotec 1093 cutting mill without any sieve. The moisture contents were determined according to the French standard NF V 03-903. The mineral contents were determined according to the French standard NF V 03-322. An estimation of the three parietal constituents (cellulose, hemicelluloses, and lignin) was made using the ADF-NDF method of Van Soest and Wine (1967, 1968). An assessment of the water-soluble components was made by measuring the mass loss of the test sample after 1 h in boiling water. All analyses were carried out in duplicate.

2.7. Morphological characterization

The morphological analysis was carried out using a MorFi Compact analyzer (TechPap, France), which is, among other parameters, able to calculate average length, average diameter, average aspect ratio and fines percentage. All characterizations were performed in duplicate.

2.8. Tapped density analysis

Tapped density was measured using a Granuloshop Densitap ETD-20 (France) volumenometer fitted with a 250 mL graduated cylinder. Before compaction, apparent density was also measured. All measurements were conducted in duplicate.

2.9. Color measurement

Color of rice straw, extrudates and TMP was measured using a spectrophotometer (Konica Minota CR-410, Japan). The color measurements were made using the CIE L*a*b* referential, which is widely employed for non-luminous objects. The illuminant was D65, and the observer angle was 2°. In the L*a*b* color space, L* is the lightness and it varies from 0 (black) to 100 (white), and a* and b* are the chromaticity coordinates: +a* is the red direction, −a* is the green direction, +b* is the yellow direction, and −b* is the blue direction. The center is achromatic (Konica Minolta Sensing, 2007). All determinations were carried out six times on each powder of rice straw, extrudate, and TMP. These powders were obtained using a Foss Cyclotec 1093 (Denmark) cutting mill fitted with a 1 mm screen.

The measured L* color values were used to estimate the darkening of the extrudates and TMP compared to the rice straw. In addition, the color difference (ΔE*) between the rice straw and each analyzed extrudate and pulp was calculated using the following formula:

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \tag{8}$$

2.10. TGA measurements

Thermogravimetric analysis (TGA) of rice straw, extrudates and TMP was carried out using a Shimadzu TGA-50 (Japan) analyzer. Dynamic analysis was conducted under air at a heating rate of 5 °C/min, from 25 to 800 °C. The materials were previously crushed using a Foss Cyclotec 1093 (Denmark) cutting mill fitted with a 1 mm screen. For all measurements, the mass of the test sample was around 8 mg. The weights of samples were measured as a function of temperature and stored. These data were later used to plot the percentage of undegraded sample (1 − D) (%) as a function of temperature, where

$$D = (W_0 - W)/W_0 \tag{9}$$

and W_0 and W are the weights at the starting point and during scanning (mg). All measurements were carried out in duplicate.

3. Results and discussion

3.1. Extrudate production by twin-screw extrusion

The extrudate production using the twin-screw extrusion technology was conducted continuously. The mean inlet flow rate of rice straw was 15.6 kg/h using a 150 rpm screw speed (Table 1). And, because the maximal screw speed of the Clextral Evolum HT 53 twin-screw extruder is 800 rpm, it is reasonable to assume that a 80 kg/h maximal inlet flow rate could be accessible from such a twin-screw machine. When the liquid/solid ratio in the twin-screw extruder reduced from 1.02 to 0.43, the extrudate flow rate (Q_R) decreased progressively from 26.8 to 19.0 kg/h. A decrease in its moisture content (H_R) from 45.3 to 26.0% was logically observed at the same time. Thus, the extrudate flow rate and its moisture content dropped about 29% and 43%, respectively. As the L/S ratio decreased, the mixture became more and more viscous, and its transportation through the CF1C reversed screws was more and more difficult, leading to a progressive increase in both filling of CF1C screws and material residence time in these restrictive elements. Thus, the shearing action given to the rice straw became much higher as the L/S ratio decreased. This was illustrated by the increased material pressure in module 7, from 10.1 to 19.5 bar (Fig. 1a), simultaneously with the increase in the current feeding the extruder motor, from 75 to 113 A (Table 1). In particular, the increase in the material pressure in module 7 was much significant (about one half of all trials) between E_4 (0.57 L/S ratio) and E_5 (0.43 L/S ratio) extrudates, i.e. from 14.9 to 19.5 bar. The same tendency was also observed for the current, the latter increasing from 91 to 113 A in the same L/S ratio range.

In addition, the increase in the viscosity of the liquid/solid mixture resulted in higher proportions of water evaporation at the outlet of the twin-screw extruder (until 30% of the injected water for the lowest L/S ratio, i.e. 0.43). The estimated outlet flow rate of water vapor varied

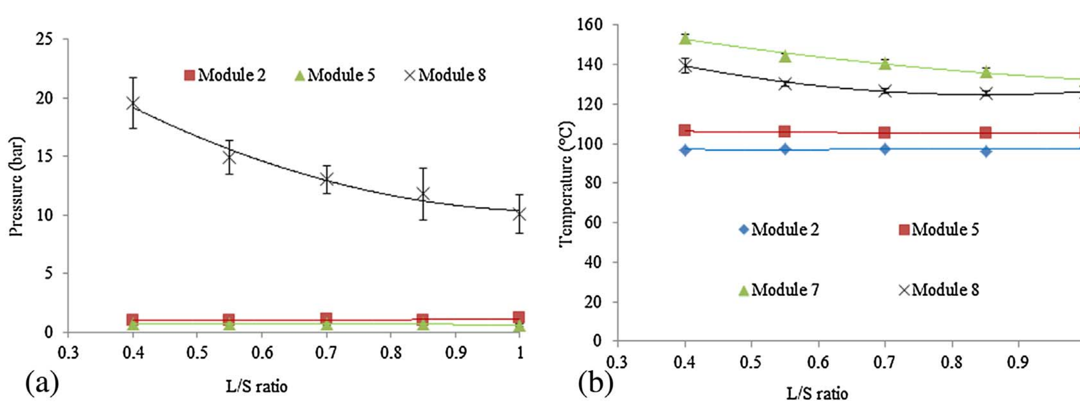

Fig. 1. Temperature (a) and pressure (b) of the material in different places along the Clextral Evolum HT 53 twin-screw extruder barrel.

from 3.9 to 2.1 kg/h as the L/S ratio decreased from 1.02 to 0.43. In proportion to the amount of liquid water injected at the end of module 3, the evaporated water increased just a little (from 24 to 26%) for L/S ratios between 1.02 and 0.57. On the contrary, it became much higher (i.e. 30%) for the E_5 extrudate. This illustrated that the 0.43 L/S ratio was probably too low, possibly causing a degradation of the rice straw fibers. Thus, no lower L/S ratio was tested in this study for the twin-screw extrusion process.

As the current feeding the motor of the twin screw extruder increased when the inlet flow rate of injected water was reduced, the specific mechanical energy (SME) increased at the same time: from 391 to 620 W h/kg dry matter (Table 1). The SME increase was limited until a 0.73 L/S ratio (+10%). Then, it was +23% at 0.57 L/S ratio and it reached +52% for the minimal L/S ratio. Table 1 also revealed an increase in the specific cooling energy (SCE), from 137 to 211 W h/kg dry matter, as the L/S ratio decreased. However, this increase was much linear. The latter might be the result of a higher tendency of the mixture to self-heating, in particular at the level of the reversed screws where the machine is completely filled, with lower liquid/solid ratios, the mixing of the mixture plus its transportation along the screw profile thus being more difficult (Gautam and Choudhury, 1999a, 1999b; Kartika et al., 2010, 2006, 2005).

In contrast, an opposite phenomenon occurred for the specific thermal energy (STE). Indeed, the machine required less heating power when the L/S ratio decreased, the STE value varying from 139 to 112 W h/kg dry matter. Firstly, the amount of liquid water, which was injected at ambient temperature, was then reduced and this contributed to a reduction of the heating power required for having the mixture temperature increasing until the set value in modules 4–6. In addition, because the mixture was more viscous with lower L/S ratios, the filling of both reversed screws and conveying screws positioned immediately upstream became more and more important, thus leading to a progressive self-heating of the material in this zone of the screw profile (Fig. 1b): from 132 to 153 °C at the end of module 7, and from 126 to 140 °C at the beginning of module 8. And, because the material always suffered self-heating at this location, the machine did not need to provide any heating power at the end of the screw profile. On the contrary, the material temperature remained quite constant in modules 2 and 5, where the filling of the bilobe paddle-screws and especially of the conveying screws was much lower (i.e. no material self-heating in these two zones of the screw profile).

In accordance with the above remarks and comparing the three specific energy requirements, SME had systematically the most important contribution. As an example, with the highest L/S ratio (i.e. 1.02), the mechanical part contributed about 59% of the total energy consumed by the twin-screw extruder, followed by the heating and cooling energy (both 21%) (Fig. 2). In the case of the lowest liquid/solid ratio (i.e. 0.43), the mechanical part still remained the majority proportion (66%). However, the cooling part was then much more significant than the heating one (i.e. 22% and 12%, respectively).

Seven modules along the twin-screw extruder barrel were heated by electric resistance, i.e. modules 2–8. But, the heating power occurred only in three modules, i.e. modules 3, 4 and 6, all situated at the level or after the injection point of water and consisting only in conveying screws, i.e. non filled elements. The heating power supplied in zones 3 and 6 remained quite independent on the L/S ratio used (Fig. 3). On the contrary, it increased progressively (from 0.7 to 1.4 kW) in zone 4 as the L/S ratio increased. As a reminder, module 4 was situated immediately after the injection point of water. And, because water was at room temperature when injected, the more the inlet flow rate of water, the more the tendency of the mixture temperature to decrease and the more the heating power required. As a consequence, the total heating power supplied by the twin-screw extruder tended to drop linearly when the liquid/solid ratio was reduced.

As the total specific energy consumption was the combination of the three specific energies, it was in the range of 0.668–0.945 kW h/kg dry matter. In addition, both SME and SCE tended to increase as the L/S ratio decreased. In parallel to the energy consumption, the total production cost increased from 0.056 to 0.076 €/kg dry matter when the L/S ratio decreased from 1.02 to 0.43 (Table 1).

3.2. Chemical composition of rice straw and extrudates

Rice straw consists predominantly of cell walls (cellulose, hemicelluloses, and lignin), i.e. the same organic compounds as those of wood sources. The chemical composition of rice straw varies between varieties and growing seasons (Shen et al., 1998). In this study, the chemical compositions of rice straw from three different locations in France and Spain, all harvested in October, were determined (Table 2). They showed little differences in the chemical composition of these three rice straw batches However, they were in the range of other chemical compositions mentioned in the literature (Garay et al., 2009; Garrote et al., 2002; Maiorella, 1983; Rahnama et al., 2013; Shen et al., 1998). It is also conceivable to think that the chemical composition of these three batches could differ due to their respective storage conditions. The rice straw used in this work for thermo-mechanical fractionation was purchased from JCL AGRI (Bouge-Chambalud, France), a forage merchant. It was stored almost one year after harvesting, while the rice straws from Spain were provided directly by farmers. Rice straw from Girona (Spain) was collected when the rice was harvested, i.e. at maturity, and then stored during approximately two years in clean and dry conditions at LEPAMAP research group, University of Girona. The third batch originated from Valencia (Spain). It was provided by a farmer from Valencia province, after almost one year storage in its yard after harvesting.

The results in Table 2 reveal that rice straw from France had less minerals and less water soluble components (14.7% and 16.0% of dry matter, respectively), compared to the two batches from Spain (15–20% and 17–19%, respectively). Rice straw from Valencia had the highest mineral content (i.e. 19.5%), and this might be due to its storage conditions. Indeed, because this batch was wet in some parts, it is reasonable to assume that part of the organic compounds were degraded over time due to the proliferation of fungi or leached, in particular the water-soluble ones, thus leading to a concurrent increase in the mineral content.

For the three main organic compounds (i.e. cellulose, hemicelluloses and lignin), rice straw from France contained more lignocellulosic fibers than those from Spain, cellulose and hemicelluloses representing 37.7% and 28.0% of its dry matter, respectively (Table 2). On the contrary, the lignin content was medium for the French batch. This is the rice straw batch originating from France which was chosen for this study, not because of its higher fiber proportion but because it was available in much greater quantity, thus allowing a sufficient feeding of the pilot-scale twin-screw extruder used in this study (much higher inlet flow rate required compared to the digester reactor plus the Sprout-Waldron defibrator). In addition, because minerals, especially silica, can generate phenomena of premature wear on the screw elements and also on the extruder barrels, it also appeared that the most reasonable choice for twin-screw fractionation was the rice straw batch with the least mineral content (i.e. the one from France), in order to reduce wear.

Table 3 shows the chemical composition of rice straw before and after twin-screw extrusion. The extrusion thermo-mechanical treatment did not change it a lot, whether the L/S ratio was high (1.0, i.e. E_1 extrudate) or median (0.7, i.e. E_3 extrudate). These results were confirmed by the thermogravimetric analysis of rice straw and extrudates E_1 to E_5 (Fig. 4).

Indeed, thermogravimetric analysis of rice straw and extrudates showed that all TGA degradation curves (Fig. 4a) under air had quite the same appearance, meaning once again that chemical compositions were all comparable and that the different operating conditions used in the twin-screw extruder had no significant influence on the thermal

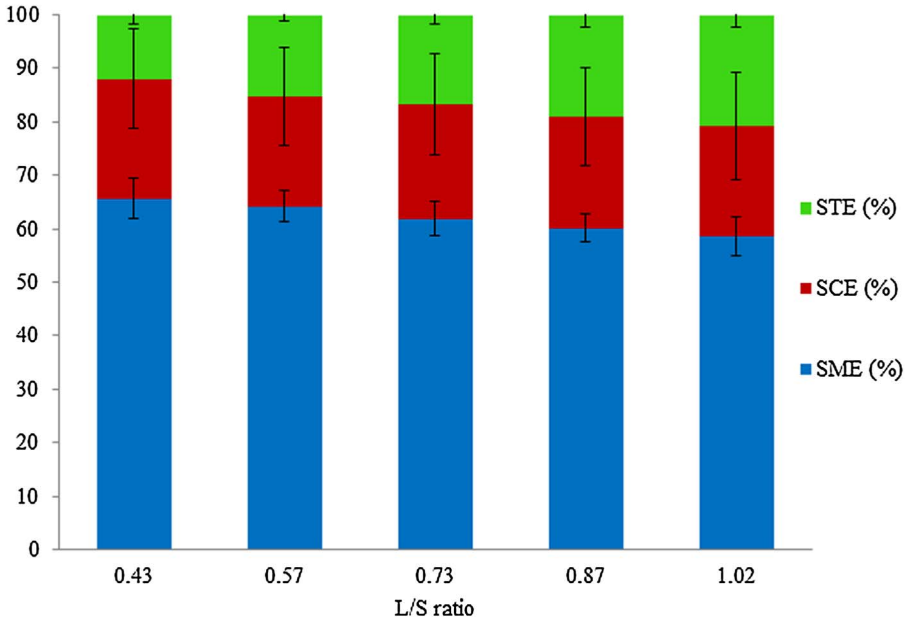

Fig. 2. Contribution of the three specific energy consumptions (%) in the Clextral Evolum HT 53 twin-screw extruder as a function of L/S ratio.

degradation of organic compounds inside the different extrudates. A first mass loss was observed at 100 °C, corresponding to the water evaporation. Moisture content of the starting material was 7.4% (Table 1), and the mass loss observed in the corresponding TGA curve was associated approximately with the same percentage. Then, the thermal degradation of organic compounds took place essentially in one stage (between 220 and 340 °C), leading to a mass loss of approximately 50% of the sample dry mass. Finally, another degradation phenomenon was also observed at higher temperature, i.e. between 400 and 476 °C. However, the latter was associated with a much lower mass loss (about 20% of the sample dry mass).

Using data dealing with the thermal degradation of fibers, cited by some researchers in previous studies (Beaumont, 1981; Evon et al., 2015; Hatakeyama and Hatakeyama, 2006; Schaffer, 1973), it is reasonable to assume that the main thermal degradation stage (220–340 °C) could be associated with the simultaneous breakdown of water-soluble compounds, hemicelluloses, and cellulose. The subsequent stage, beginning at about 400 °C, would in this case correspond essentially to the thermal degradation of lignin. However, because TGA analysis of all materials was conducted under air atmosphere, part of this second thermal degradation stage could also correspond to the oxidation of the degradation products from the previous stage (Uitterhaegen et al., 2016). At the end of all measurements, the undegraded samples accounted for less than 15% of the test sample mass, corresponding to the minerals contained in rice straw and extrudates (Table 3).

3.3. Physical properties of extrudates

Table 4 shows the main morphological characteristics of fibers inside the extrudates (E_1 to E_5) produced using different operating conditions. Such characteristics were determined using a MorFi Compact analyzer, and they include length, diameter, and aspect ratio (defined as the ratio of the length to the diameter) of fibers, and fine percentage inside the extrudates. From the mean length (L_W) and diameter (D) of the extruded fibers, the corresponding L_W/D aspect ratios were between

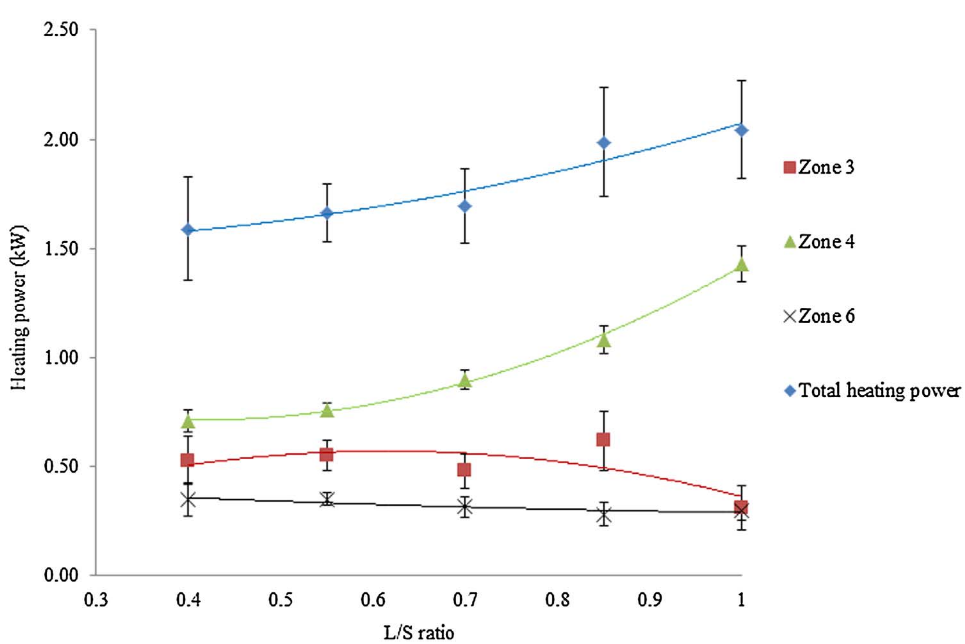

Fig. 3. Heating power along the barrel of the Clextral Evolum HT 53 twin-screw extruder.

Table 2
Chemical composition of rice straw from three different origins (% of dry matter).

Origin	Minerals	Cellulose	Hemicelluloses	Lignin	Water-soluble components
Bouge-Chambalud, France	14.7 ± 0.1	37.7 ± 0.3	27.9 ± 0.4	7.2 ± 0.1	16.0 ± 0.1
Girona, Spain	15.5 ± 0.1	34.8 ± 0.1	27.9 ± 0.0	6.5 ± 0.4	18.9 ± 0.1
Valencia, Spain	19.5 ± 0.1	33.8 ± 0.1	25.0 ± 0.5	8.1 ± 0.2	16.6 ± 0.2

20.9 and 22.6. In addition, a slight decrease in the aspect ratio was observed with a decreasing liquid/solid ratio in the twin-screw extruder, especially for its two lowest values (i.e. 0.55 and 0.4), and such phenomenon was previously observed for thermo-mechanical pulps produced using steam plus a mechanical defibration treatment (Alila et al., 2013; Flandez et al., 2012; Theng et al., 2015a). Such aspect ratio decrease resulted mainly in the decrease of the mean length of fibers, from 571 to 494 μm (i.e. −14%), as the L/S ratio decreased from 1.02 to 0.43. The fiber size distribution inside the extrudates (Fig. 5) and their microscopic images (Fig. 6) confirmed the results of the mean length and width of fibers using the different L/S ratios during extrusion. In this study, the use of a 1.02 L/S ratio showed the best fiber properties (i.e. higher length and higher aspect ratio) and also the lowest energy consumption. Thus, a 1.25 L/S ratio was also tested to produce even longer and cheaper fibers. However, the extrudate sampling was not possible from such condition due to the return of water at the level of the rice straw feeding zone (i.e. module 1). And, this illustrated the fact that higher mechanical shear and higher self-heating of the material at the level of the reversed screws caused by low L/S ratios contributed in more cutting of the fibers.

The particle size distribution inside the extrudates (Table 4) revealed also the presence of small particles (approximately 25 μm × 500 μm). This population contained not only the smallest fibers but also spherical particles, i.e. fines, originating from the thermo-mechanical breakdown process of rice straw, and corresponding to a 63% mean content. Fig. 5 illustrated the distribution of the fibers' dimension by weighted length and width in five categories, each from 200 to more than 1500 μm and from 5 to more than 67 μm, respectively. As it can be seen in Fig. 5, the proportion of shorter fibers increased when the L/S ratio was reduced, particularly for E_4 (L/S ratio 0.57) and E_5 (L/S ratio 0.43) extrudates. Lastly, apparent and tapped densities of the extrudates were quite low with maximal values of 162 and 216 kg/m^3, respectively (Table 4).

Looking at the influence of the operating conditions tested on the apparent and tapped densities of extrudates, both densities are decreasing when the L/S ratio increased: from 162 to 56 kg/m^3, and from 216 to 81 kg/m^3, respectively. Referring to the results of densities, a lower L/S ratio during twin-screw extrusion made not only shorter fibers (Table 4) but also a denser and heavier extrudate. On the contrary, because fibers originating from the highest L/S ratios were longer, their entanglement between them was favored, leading to a bulkier and therefore to a less dense extrudate.

The effect of the liquid/solid ratio during twin-screw extrusion on the extrudate color, in comparison to the one of rice straw, is provided in Table 5. A decrease in the L/S ratio contributed in the decreases in both L* and b* values, simultaneously with a slight increase in the a* one. The significant L* decrease observed as the decreasing L/S ratio, revealed a progressive darkening of the extrudates in comparison to the initial rice straw color, with a color difference (ΔE*) varying from 6.2 to 8.2. According to Ilo and Berghofer (1999), color is an important quality assurance parameter of feed and biological products, which can also be indirectly related to the nutritional value of the products. In this case (i.e. the thermo-mechanical defibration of rice straw using the twin-screw extrusion), the alteration degree of the structure of rice straw fibers inside the twin-screw extruder barrel is a key factor in the quality of the extrudates. It is largely dependent on the L/S ratio used, the latter largely influencing the mechanical shear transmitted to the material and its self-heating. Therefore, the increase in the darkening of the extrudate observed with the decrease in the L/S ratio could be directly correlated to the progressive self-heating undergone by the material in the restrictive part of the screw profile, i.e. where mechanical shear is applied (from 132 to 153 °C for the material temperature at the end of module 7, and from 126 to 140 °C at the beginning of module 8), contributing to the partial degradation of the organic compounds inside rice straw, especially the smaller and the most thermal sensitive molecules.

3.4. TMP production by digester reactor plus Sprout-Waldron defibrator

Thermo-mechanical pulp from rice straw was produced by digestion plus defibration using a cooking condition in terms of temperature and duration previously optimized, i.e. 160 °C and 30 min, respectively (Theng et al., 2015b). Operating conditions for the TMP preparation and its results for energy consumption and production cost are detailed in Table 6.

Water was added at the maximum mass in terms of the digester reactor capacity, i.e. 8 kg for biomass plus distilled water mass. In parallel, the mass of the starting solid was 1.61, 1.33, and 1.15 kg dry matter for P_1, P_2, and P_3 pulps, respectively, corresponding to liquid/solid ratios of 4, 5 and 6, respectively. All trials were digested using the same conditions and then cooled to 105 °C before opening and washing using tap water. Depending on the initial liquid/solid ratio used (i.e. 4, 5, and 6), the mass of the recovered digested pulp was 1.38, 1.17, and 1.01 kg dry matter, respectively. During digestion, some organic compounds, especially the water-soluble ones, were removed from the biomass. This was illustrated by the results of production yield obtained: 85.7%, 88.0%, and 87.8% following the different liquid/solid ratios used (i.e. 4, 5, and 6, respectively), corresponding to a dry mass

Table 3
Chemical composition of rice straw (French origin), extrudates (E), and pulps (P).

Materials	Moisture (%)	Minerals (% of DM)	Cellulose (% of DM)	Hemicelluloses (% of DM)	Lignin (% of DM)	Water-soluble components (% of DM)
Rice straw	7.4 ± 0.1	14.7 ± 0.1	37.7 ± 0.3	27.9 ± 0.4	7.2 ± 0.1	16.0 ± 0.1
E_1 (L/S ratio 1.02)	7.5 ± 0.1	14.3 ± 0.2	36.2 ± 0.5	33.0 ± 0.6	5.5 ± 0.5	15.9 ± 0.1
E_3 (L/S ratio 0.73)	7.1 ± 0.0	14.4 ± 0.1	37.0 ± 0.9	28.4 ± 0.2	6.8 ± 0.3	17.3 ± 0.3
P_1 (L/S ratio 4.0)	4.8 ± 0.2	10.7 ± 0.0	47.6 ± 0.1	28.6 ± 0.1	8.7 ± 0.1	5.1 ± 0.1
P_2 (L/S ratio 5.0)	4.8 ± 0.3	11.0 ± 0.1	47.5 ± 0.0	29.5 ± 0.9	8.1 ± 0.1	4.8 ± 0.6
P_3 (L/S ratio 6.0)	4.6 ± 0.3	10.5 ± 0.1	48.2 ± 0.3	28.3 ± 0.3	9.7 ± 0.2	5.1 ± 0.0

Moisture contents were measured after conditioning in climatic chamber (25 °C and 60% relative humidity), and DM is dry matter.

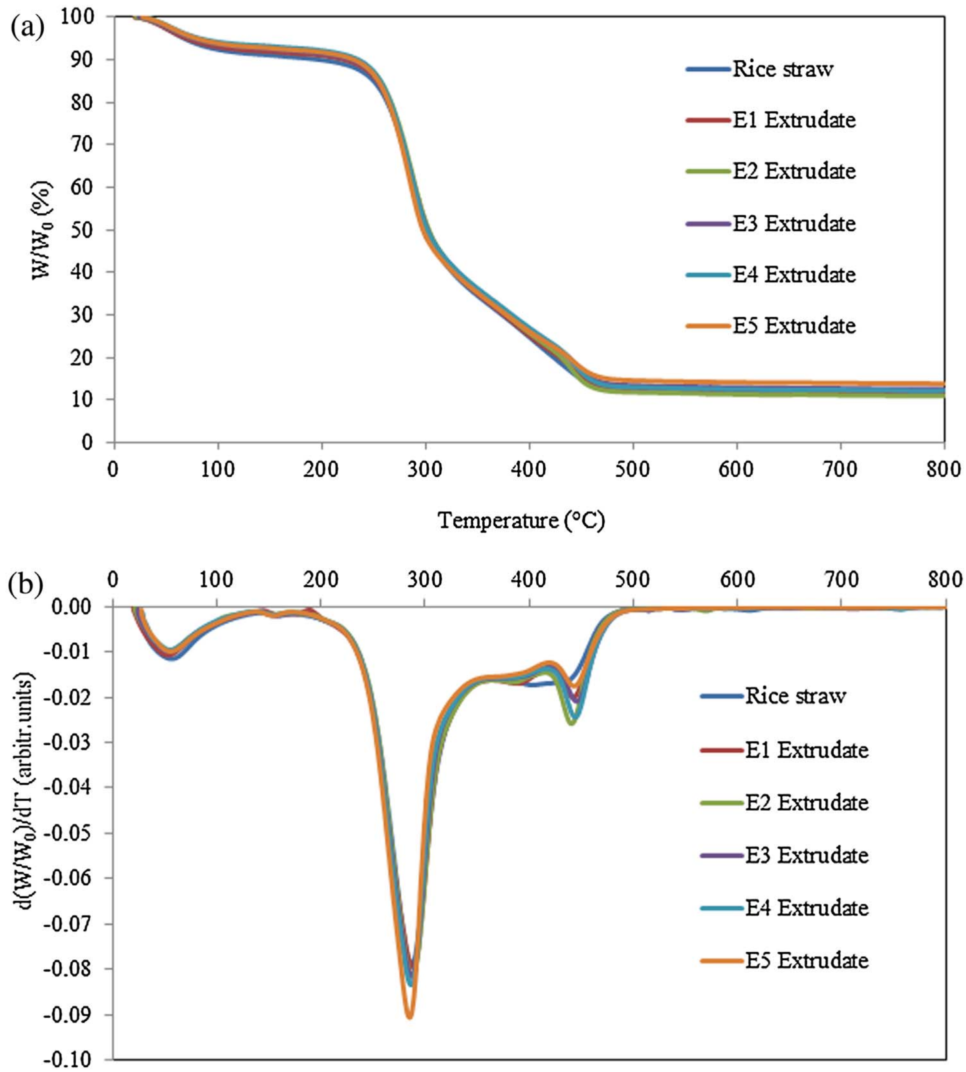

Fig. 4. TGA (a) and dTGA (b) curves of rice straw and extrudates.

loss of 14.3%, 12.0%, and 12.2%, respectively. This was also confirmed by the decrease in the contents of water-soluble compounds inside digested pulps compared to the rice straw (Table 3). The TMP production yield (87.8%) for rice straw originating from France was a little higher compared to rice straw grew in Girona (Spain) when cooked using the same operating conditions and inside the same digester reactor, i.e. 84.7% (Theng et al., 2015b) and 82.0% (So, 2016), corresponding to a reduction in the dry mass removing during digestion: 12.2% instead of 15.3% and 18.0%. This finding is likely to be related with the fact that the chemical composition of French rice straw revealed less water-solubles than the one collected in Girona: only 16.0% instead of 18.9% (Table 2).

The energy consumption for the whole process of TMP preparation, i.e. pre-heating, heating, and digestion in the digester reactor plus defibration in the Sprout-Waldron defibrator, was 8.5–8.6 kW h for P_1, P_2, and P_3 pulps (Table 6), indicating that it was quite independent on the liquid/solid ratio used. From these values, it can be seen that the digester reactor consumed almost all the energy while the defibration step contribution using the Sprout-Waldron defibrator was only 0.4%. To be more precise, there were three successive steps during the discontinuous digestion process, including (i) the pre-heating of the digester reactor from 16 °C (storage temperature of the machine) to 80 °C (temperature at which the raw material was added), (ii) the heating of the digester reactor plus the liquid/solid mixture from 80 °C to 160 °C (target cooking temperature), and (iii) the digestion itself at the target temperature and during a certain duration (i.e. 30 min). Among these,

Table 4
Morphology, and apparent and tapped densities of fibers in the extrudates (E) and pulps (P) made from rice straw.

Materials	L_W (µm)	D (µm)	L_W/D (aspect ratio)	Fines (%)	Kink angle (°)	Apparent density (kg/m^3)	Tapped density (kg/m^3)
E_1 (L/S ratio 1.02)	571.5 ± 7.8	25.5 ± 0.0	22.4 ± 0.3	63.8 ± 0.4	126.5 ± 0.7	56.3 ± 6.5	80.7 ± 5.4
E_2 (L/S ratio 0.87)	544.0 ± 4.2	24.5 ± 0.0	22.2 ± 0.2	62.8 ± 1.5	125.0 ± 0.0	63.9 ± 1.3	88.2 ± 0.2
E_3 (L/S ratio 0.73)	571.0 ± 7.1	25.3 ± 0.0	22.6 ± 0.3	54.2 ± 4.9	123.5 ± 0.7	69.5 ± 8.6	100.3 ± 9.9
E_4 (L/S ratio 0.57)	505.0 ± 5.7	23.8 ± 0.1	21.2 ± 0.2	74.7 ± 3.4	125.0 ± 0.0	118.3 ± 0.7	151.6 ± 0.2
E_5 (L/S ratio 0.43)	494.0 ± 1.4	23.6 ± 0.1	20.9 ± 0.1	61.8 ± 0.5	124.5 ± 0.7	161.9 ± 1.6	216.4 ± 0.5
P_1 (L/S ratio 4.0)	371.7 ± 9.0	22.8 ± 0.0	16.3 ± 0.4	69.6 ± 2.3	133.0 ± 1.0	53.5 ± 1.1	69.3 ± 0.9
P_2 (L/S ratio 5.0)	377.0 ± 5.7	22.0 ± 0.1	17.0 ± 0.2	68.2 ± 1.5	133.3 ± 1.5	47.4 ± 1.1	62.7 ± 1.0
P_3 (L/S ratio 6.0)	386.0 ± 2.8	21.9 ± 0.1	17.9 ± 0.4	65.3 ± 3.6	134.0 ± 1.0	48.0 ± 1.8	62.9 ± 1.8

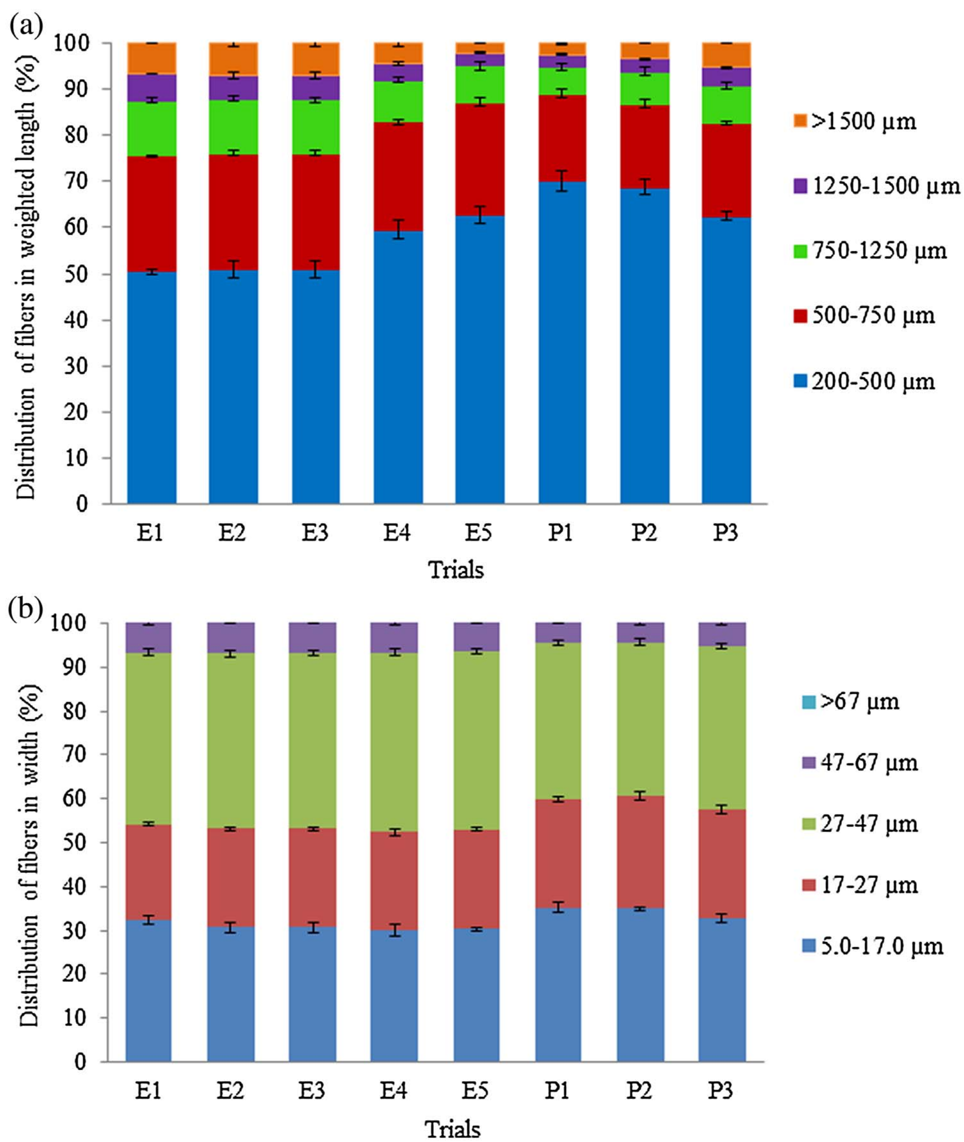

Fig. 5. Distribution of fibers in weighted length (a) and in width (b).

the heating stage was the one consuming the most energy (approximately 60%), followed by the pre-heating step (around 33%), for all operating conditions tested (Fig. 7). This revealed that the majority of energy consumption was spent on warming the digester reactor and then on heating the raw material after being placed in the digester since the digestion operation was a discontinuous process, meaning that the reactor temperature decreased between two successive digestion batches when it was opened and then cleaned using tap water. Conversely, the electric resistance of the digester reactor transmitted just a little more energy in order to maintain the 160 °C temperature set value during the entire cooking duration.

During the digestion plus defibration process, the specific energy consumption increased when increasing the liquid/solid ratio. As revealed in Table 6, the energy consumptions per unit weight of dry matter for pre-heating, heating, digestion and defibration were 2.0–2.8, 3.7–5.1, 0.4–0.6, and 0.024–0.035 kW h/kg dry matter, respectively, as the L/S ratio increased from 4 to 6, corresponding to a total specific energy consumption increasing from 6.2 to 8.5 kW h/kg dry matter of pulp. Thus, the total production cost of pulp logically increased as the liquid/solid ratio used increased: from 0.49 €/kg dry matter at 4 liquid/solid ratio to 0.68 €/kg dry matter at 6 liquid/solid ratio (Table 6).

3.5. Chemical composition of rice straw TMP

The chemical compositions of the three rice straw TMP produced using digester reactor plus Sprout-Waldron defibrator are indicated in Table 3. The thermo-mechanical treatment using digestion plus defibration contributed in changes in the biomass chemical composition, with an increase in contents of both cellulose and lignin (from 38% to 48%, and from 7% to 10%, respectively), simultaneously with a large decrease in the content of water-soluble compounds (from 16% for initial rice straw to 5% for TMP). Because large amounts of water were used during the digestion step, some of the water-soluble organic compounds, especially free sugars, were removed from rice straw during digestion, thus leading to a 86–88% production yield. These results were confirmed by the thermogravimetric analysis of rice straw and pulps P_1 to P_3 (Fig. 8).

Indeed, the TGA degradation curves (Fig. 8a) under air had quite difference appearance between pulps and rice straw, meaning once again that chemical compositions were changed. As a result, because water-soluble compounds were highly thermal sensitive, the beginning of the thermal degradation inside pulps occurred at higher temperature: around 230 °C instead of around 205 °C for rice straw. A first mass loss was observed at 100 °C, corresponding to the water evaporation. Moisture content was 7.4% and 4.6–4.8% for rice straw and pulps,

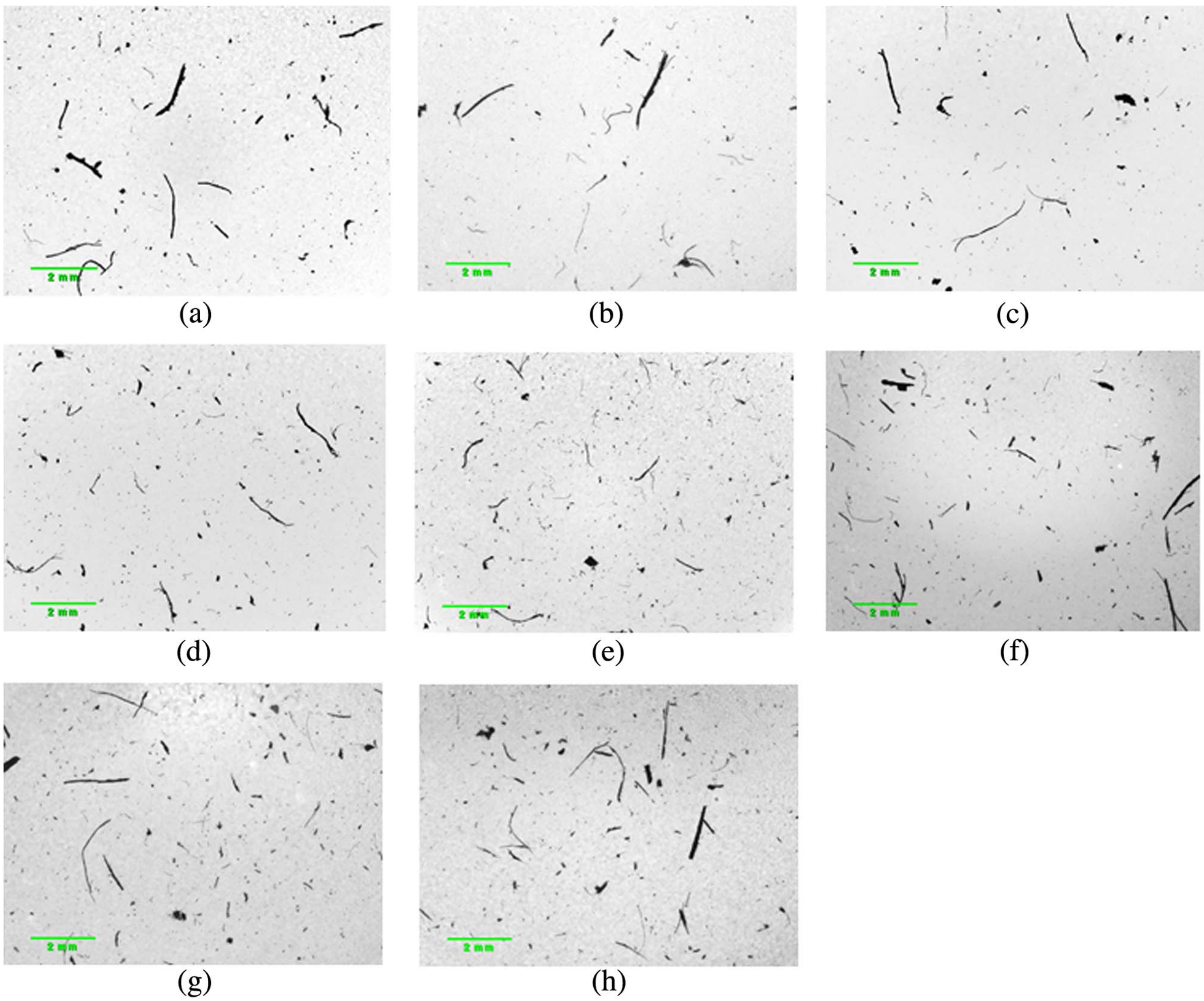

Fig. 6. Microscopic images of fibers taken during their morphological analyses using the MorFi Compact analyzer: for extrudates E_1–E_5 (a–e) and for pulps P_1–P_3 (f–h).

Table 5
Influence of the operating conditions on color in the CIE L*a*b* referential of the extrudate (E) and pulp (P) powders, and comparison with color of the starting material (i.e. rice straw powder).

Materials	L*	a*	b*	ΔE*
Rice straw	82.6 ± 0.3	3.7 ± 0.1	16.5 ± 0.2	–
E_1 (L/S ratio 1.02)	77.4 ± 0.3	4.1 ± 0.1	13.2 ± 0.5	6.2
E_2 (L/S ratio 0.87)	77.1 ± 0.4	4.3 ± 0.1	13.3 ± 0.2	6.4
E_3 (L/S ratio 0.73)	75.9 ± 0.1	4.2 ± 0.1	12.9 ± 0.2	7.6
E_4 (L/S ratio 0.57)	76.3 ± 0.3	4.1 ± 0.1	12.9 ± 0.3	7.2
E_5 (L/S ratio 0.43)	74.9 ± 0.7	4.2 ± 0.2	13.9 ± 0.5	8.2
P_1 (L/S ratio 4.0)	75.7 ± 0.4	4.2 ± 0.1	12.7 ± 0.5	7.8
P_2 (L/S ratio 5.0)	75.9 ± 0.3	4.1 ± 0.1	13.5 ± 0.3	7.3
P_3 (L/S ratio 6.0)	75.8 ± 0.3	4.1 ± 0.1	13.2 ± 0.3	7.5

respectively (Table 3), and the mass loss observed in the corresponding TGA curves was associated approximately with the same percentage. Then, the thermal degradation of organic compounds inside pulps took place mainly in one stage (in the range 230–350 °C), leading to a mass loss of approximately 55% of the sample dry mass. In agreement to some researchers in previous studies (Beaumont, 1981; Evon et al., 2015; Hatakeyama and Hatakeyama, 2006; Schaffer, 1973), the main thermal degradation stage is associated with the simultaneous breakdown of organic compounds (i.e. residual water-soluble compounds, hemicelluloses, and cellulose). Finally, another degradation phenomenon was also observed between 365 and 470 °C, however this was associated with a lower mass loss (about 20% of the sample dry mass), corresponding to the degradation of lignin and also to the oxidation of the degradation products from the previous stage, as the TGA analysis was conducted under air atmosphere (Uitterhaegen et al., 2016). At the end of all measurements, the undegraded samples represented around 9% of the test sample mass, corresponding to the minerals contained in pulps (Table 3).

3.6. Physical properties of TMP

The main morphological characteristics of TMP fibers (P_1 to P_3) produced using different liquid/solid ratios are shown in Table 4. From the mean length (L_W) and diameter (D) of the thermo-mechanical fibers, the corresponding L_W/D aspect ratios were between 16.3 and 17.9. In addition, a slight increase in the aspect ratio was observed with an increasing liquid/solid ratio (i.e. from 4 to 6) in the digester reactor and Waldron-Sprout defibrator, and such phenomenon was previously observed for twin-screw extrusion (Table 4) but also in the case of a steaming plus a mechanical defibration treatment (Alila et al., 2013; Flandez et al., 2012; Theng et al., 2015a). Such aspect ratio increase resulted mainly in the slight increase of the mean length of fibers, from 371 μm to 386 μm (i.e. +4%) as the liquid/solid ratio increased from 4 to 6. The fiber size distribution inside pulps (Fig. 5) and their microscopic images (Fig. 6) confirmed this result. This illustrated the fact that

Table 6
Operating conditions and results of the thermo-mechanical fractionation of rice straw by steaming in the digester reactor plus defibration using the Sprout-Waldron 105-A defibrator.

Trials	P1	P_2	P_3
Operating conditions			
Solid mass (kg dry matter)	1.61	1.33	1.15
Water mass (kg)	6.39	6.67	6.85
Solid plus water mass (kg)	8.00	8.00	8.00
L/S ratio	4	5	6
Temperature (°C)	160	160	160
Duration (min)	30	30	30
Digested pulp			
Pulp mass (kg dry matter)	1.38	1.17	1.01
Production yield (%)	85.7	88.0	87.8
Energy consumption (kW h)			
Pre-heating digester (16–80 °C)	*2.79*	*2.73*	*2.81*
Heating digester (80–160 °C)	*5.09*	*5.13*	*5.14*
Digestion (160 °C, 30 min)	*0.61*	*0.61*	*0.62*
Defibration in Sprout-Waldron defibrator	*0.033*	*0.032*	*0.035*
Total energy consumption	8.523	8.502	8.605
Specific energy consumption (kW h/kg dry matter of pulp)			
Pre-heating digester (16–80 °C)	2.022	2.333	2.782
Heating digester (80–160 °C)	3.688	4.385	5.089
Digestion (160 °C, 30 min)	0.442	0.521	0.614
Defibration in Sprout-Waldron defibrillator	0.024	0.027	0.035
Total specific energy consumption	6.176	7.266	8.520
Total production cost (€/kg dry matter)	0.494	0.581	0.682

L/S ratio is defined as the ratio of the water mass (including both liquid water and moisture inside rice straw) to the dry solid mass. The total production cost is defined as the total specific energy consumption × 0.08 €/kW h (electricity price in France in 2016).

lower L/S ratio at high temperature contributed in more degradation and cutting of the fibers.

The particle size distribution inside pulps (Table 4) revealed also the presence of small particles (approximately 25 µm × 500 µm). This population contained not only the shortest fibers but also a large amount of fines, originating from the thermo-mechanical breakdown of rice straw and corresponding to a 65–70% range content. In addition, as it was already the case inside the extrudates, the proportion of fines and shorter fibers inside pulps tended to decrease as the mean length of fibers increased (Table 4 and Fig. 5). Lastly, apparent and tapped densities of the TMP were low with maximal values of 53 and 69 kg/m^3, respectively (Table 4).

Looking at the influence of the liquid/solid ratio used on the apparent and tapped densities of pulps, both densities are slightly decreasing when the L/S ratio increased: from 53 to 48 kg/m^3 and from 69 to 63 kg/m^3, respectively. And, such tendency was already observed in the case of extrudates (Table 4). According to the density results, a lower L/S ratio during steaming digestion and mechanical defibration made not only shorter fibers (Table 4) but also a denser and heavier TMP. On the contrary, because fibers originating from the highest L/S ratios were longer, their entanglement between them was favored, leading to a bulkier and therefore to a less dense pulp. Lastly, due to the high liquid/solid ratios used during digestion, it should also be noted that TMP revealed much lower densities than the extrudates. Firstly, the low values of pulp density could be the consequence of a higher kink angle. Indeed, the kink angle values presented in Table 4 were 133–134° for pulps and 123–126° for extrudates. Thus, because fibers inside pulps were more curved, their stacking at compaction was disadvantaged. Secondly, pulps were made of agglomerates, and this resulted in empty zones between them (before and after compaction), thus contributing to an artificial decrease in both apparent and tapped densities.

The effect of the liquid/solid ratio during digestion plus defibration on the color of pulps, in comparison to the one of rice straw, is provided in Table 5. An increase in the L/S ratio from 4 to 6 had no influence on the change in color. However, the significant decrease in L* and b* values, simultaneously with the slight increase in the a* one, revealed a darkening of the three pulps produced in comparison to the initial rice straw color, with a color difference (ΔE*) varying from 7.3 to 7.8. Such darkening was also observed in the case of extrudates (Table 5), and this was the consequence of the alteration of the structure of rice straw fibers inside TMP. Indeed, from the operating conditions used during digestion plus defibration, i.e. high temperature (160 °C) during 30 min, a partial degradation of the organic compounds inside rice straw occurred, especially the smaller and the most thermal sensitive molecules, thus contributing to the darkening of pulps.

3.7. Extrudate (twin-screw extrusion) versus pulp (digestion plus defibration), a comparison

When comparing the two methods for rice straw fiber pretreatment prior to fiberboard manufacturing, the L/S ratios used were extremely different. On the one hand, L/S ratios varied from 1.02 to 0.43 inside the horizontal twin-screw extruder. On the other hand, L/S ratios were much more important (i.e. 4–6) inside the vertical digester plus defibrator. Thus, the use of the twin-screw extrusion technology allowed a subsequent reduction in the L/S ratio used. This was due to the fact that the capability of the water to be diffused inside the solid plant matrix is clearly favored inside the twin-screw reactors thanks to the quality of the contacting between the liquid and solid phases (Evon, 2008; Bouvier and Campanella, 2014). From this, it is reasonable to assume

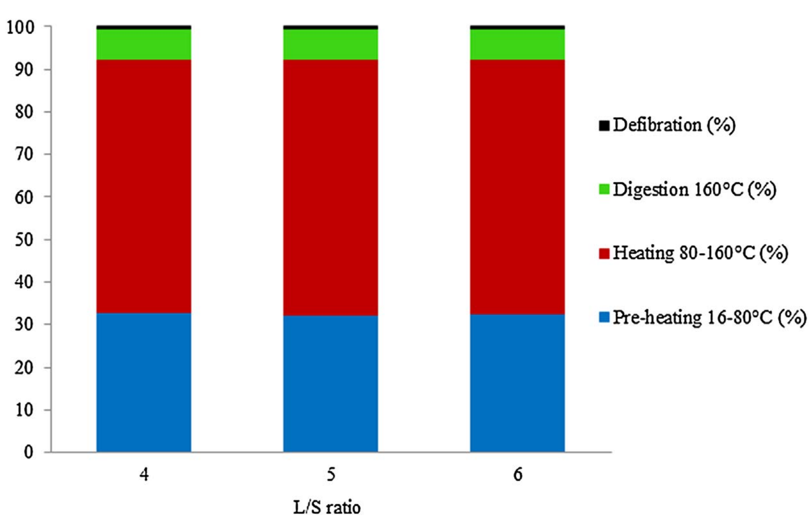

Fig. 7. Contribution of the different steps for TMP preparation using a rotary digester plus a Sprout-Waldron 105-A defibrator on the energy consumption (%).

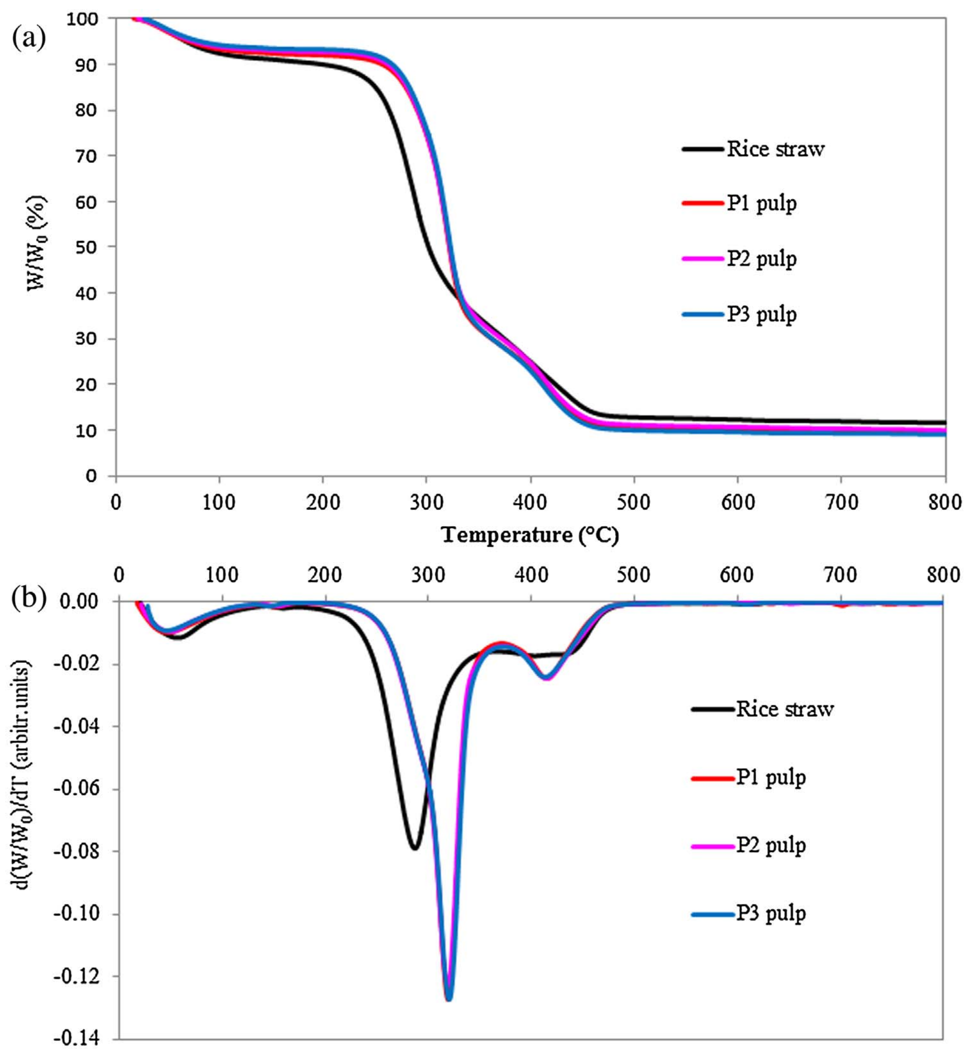

Fig. 8. TGA (a) and dTGA (b) curves of rice straw and TMP.

that this large difference in the L/S ratios used will affect the properties of the obtained fibers (in particular their length, aspect ratio, and apparent and tapped densities) and also their production cost in terms of electricity consumption.

Comparing twin-screw extrusion and digestion plus defibration, the influence of the liquid/solid ratio used on the specific energy consumption and overall production cost of rice straw fiber pretreatment was opposed, i.e. a decrease in extrusion (Table 1) as opposed to its increase for digestion plus defibration (Table 6) with increasing liquid/solid ratio. In addition, the fiber pretreatment using twin-screw extrusion had some advantages compared to the digestion plus defibration method, listed as following:

- higher inlet flow rate and, as a continuous process, better productivity;
- lower amounts of water and lower temperature;
- lower energy requirement and production cost, approximately nine times less important: the twin-screw extrusion technology appeared as a much more economical pretreatment for rice straw fibers, without taking into account the investment cost of the equipment. Looking at the selling price of some commercial fibers, hardwood and softwood fibers having a 300–500 µm mean length (i.e. perfectly comparable to that of fibers inside both extrudates and pulps) and used for the mechanical reinforcement of thermoplastic matrices molded using injection cost around 0.50 €/kg. In the same way, the selling price of a bleached kraft pulp is about 0.55 €/kg. By comparison, depending on the L/S ratio used, the production cost of the extrudates was only 0.05–0.08 €/kg dry matter, and it was 0.49 €/kg for P_1 pulp. This confirms the low cost for the production of thermo-mechanical fibers using the twin-screw extrusion technology, even if this price does not include neither the personal expenses nor the amortization cost of the equipment;
- higher aspect ratio, which should lead to an improvement of the entanglement of fibers inside fiberboards, thus possibly contributing to better mechanical reinforcement;
- higher amount of water-soluble components, in particular free sugars: because such components can contribute to the self-bonding of fiberboards using hot pressing (Hashim et al., 2012, 2011a, 2011b; Tajuddin et al., 2016), this could be an advantage for their cohesion and mechanical strength.

On the other hand, two disadvantages can be listed for the extrusion method compared with the digestion plus defibration one. Firstly, taking into account the TGA results for both treated materials, extrudates were more thermal-sensitive, and it will be probably necessary to use lower values for molding temperature during thermopressing (i.e. no more than 200 °C) in such a way as to avoid any degradation of organic compounds. In addition, establishing a parallel with papermaking, it is reasonable to assume that the ultimate strength of fiberboards made from TMP should be higher than that of fiberboards made from extrudates. Indeed, in the paper industry, a higher specific surface and a decrease in the mineral content (cases of TMP compared to extrudates) should promote the compatibility of lignocellulosic fibers, leading to an increasing amount of bonds between fibers (i.e.

higher relative bonded area) and thus to a higher fiberboard compaction (Page, 1969; Vilaseca et al., 2008).

The next step will consist in using both pretreated rice straw materials, i.e. extrudates and pulps, to compare their respective performances for fiberboard making, with or without addition of a natural binder. By doing so, in terms of board mechanical strength, a comparison between the contributions of (i) water-solubles inside extrudates to self-bonding and (ii) the higher relative bonded area for TMP will become possible (Theng et al., under review).

4. Conclusions

The thermo-mechanical pretreatment of rice straw fibers was investigated in this study using two different technologies, i.e. twin-screw extrusion, and digestion plus defibration. For both technologies, the specific energy consumption of the equipment led to the production cost calculation, the latter depending on the liquid/solid ratio used. For the twin-screw extrusion process, the total specific energy ranged from 0.945 to 0.668 kW h/kg dry matter as the liquid/solid ratio increased from 0.43 to 1.02, meaning that a bigger expense occurred at lower liquid/solid ratio. On the contrary, for the digestion plus defibration method, the more the liquid/solid ratio the more the energy consumed (until 8.52 kW h/kg dry matter for a 6 liquid/solid ratio). Comparing both technologies, twin-screw extrusion was about nine times less energy consuming and cheaper than the digestion plus defibration process. In addition, extrudate fibers were much more economical than commercial fibers in terms of production cost. However, in terms of the equipment cost, twin-screw extrusion is a quite expensive technology for investment. The pilot-scale twin-screw equipment used in this study costed approximately thirteen times more than the laboratory equipment consisting of the digester plus defibrator. Thus, because the twin-screw extruder costs more than the other machines, the depreciation accounting of the extruder will require for the company using it a longer period than for the digestion plus defibration technique. The pretreated rice straw fibers obtained using these two thermo-mechanical processes will then be usable for the manufacture of fiberboards using hot pressing (Marechal, 2001; Theng et al., 2017; Theng et al., 2015b).

For twin-screw extrusion, the total specific energy was a combination of three specific energy types, i.e. SME, SCE, and STE. Among these, the specific mechanical energy was the most important contribution. It was between 59 and 66% of the total, depending on the liquid/solid ratio used. For digestion plus defibration, the total specific energy was the combination of a digestion cost through steaming in a rotary digester (for thermo-mechanical fractionation) and a defibration using a Sprout-Waldron defibrator (for refining the particle size). The digestion step represented almost 100% of the total production cost, while defibration contributed to less than 0.4%.

The different liquid/solid ratios used for extrusion had no effect on the main characteristics of treated lignocellulosic fibers, including their chemical compositions and their thermal properties. However, for the morphology of these treated fibers, a slight decrease in their mean length, their mean diameter and their mean aspect ratio was observed when the quantity of water was reduced. The same effect was observed for fibers treated using digestion plus defibration. In addition, the shortest treated fibers revealed higher apparent and tapped densities, in particular inside the extrudates produced using the two lowest liquid/solid ratios.

Furthermore, in comparison to the initial rice straw biomass, the chemical compositions and thermal properties of the extrudates remained the same, although the extrudates were a little browner than the starting material. On the contrary, the chemical compositions and thermal properties of the TMP were changed, compared to the rice straw raw material. The most important change was their contents in water-soluble components, which were partly removed during the digestion step, conducted using high amounts of water, at a high temperature (160 °C), and for a long duration (30 min). And, pulps were also a little browner than the initial rice straw biomass.

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