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The effect of wood extractives on the thermal stability of different wood species

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ABSTRACT

This study compares the thermal stability of different wood species, which is an important factor for the production of wood–polymer composites (WPCs), and investigates the effect of extraction on thermal properties. The chemical composition of four wood species – *Quercus alba*, *Pinus radiata*, *Eucalyptus grandis* and *Acacia cyclops* – has been determined, as the species is expected to affect the thermal stability of wood. Subsequently, the hot-water (HW) extractives, ethanol/cyclohexane (E/C) extractives and both extractives were eliminated from the wood via Soxhlet extraction and the thermal stability of the wood determined with thermogravimetric analysis (TGA) under identical conditions. The results suggest that a higher cellulose and lignin content leads to better thermal stability of wood in different temperature regimes. In all cases, the removal of extractives improved the thermal stability of the wood. The effect of combined extractions was more pronounced than of an individual extraction and E/C-extraction caused less improvement in the thermal stability of wood than HW extraction. The degradation of the investigated wood extractives occurred at low rates over a broad temperature range. Pure cellulose exhibited superior thermal stability compared to wood, but differences were observed between the investigated wood species.

1. Introduction

Although the use of wood–polymer composites (WPCs) increased significantly in recent years, they have several limitations, such as the low thermal stability and the moisture absorption of wood and the poor adhesion between nonpolar, hydrophobic polymers and the polar, hydrophilic wood particles [1]. WPCs can replace pure plastics in applications where the increased stiffness originating from the wood fibers is an advantage. They can also replace impregnated wood for various outdoor applications [2]. The main application area of WPCs is in the building-, automotive- and packaging-industry [3].

The thermal stability of wood is a very important parameter in the production of WPCs. They are usually manufactured by mixing wood and polymer at a temperature above the melting temperature of the polymer. Wood is used as a filler material and to improve mechanical strength in plastics that are processed at temperatures below 200 °C [4]. This limits the type of plastics that can be processed into WPCs and the applications in which they can be used. The degradation of wood due to high temperatures can lead to undesirable properties, such as odor and discoloration and the loss of mechanical strength [5].

The major chemical components of wood are cellulose, hemicelluloses, lignin and extractives and they degrade at different temperatures. Wood materials are known to present different degradation profiles depending on the wood composition. Cellulose is highly crystalline, which makes it thermally stable. Hemicelluloses and lignin, on the other hand, are amorphous and start to degrade before cellulose [6]. Hemicelluloses are the least thermally stable wood components, due to the presence of acetyl groups [7]. Lignin degrades partly over a wide temperature range, starting at relatively low temperatures [8]. Wood also contains a small amount of extractives, which include, amongst others, lipids, phenolic compounds, terpenoids, fatty acids, resin acids, steryl esters, sterol and waxes [9–12]. Generally the extractive content varies between 2% and 5% but can be as high as 15% [13]. Although extractives contribute merely a few percent to the entire wood composition, they have significant influence on its properties, such as mechanical strength or color [14] and the quality of wood can be affected by the amount and type of these extractives [15]. Extractives can even be toxic and harmful to the environment [11]. The oxidation of extractives tends to increase the acidity of wood and promote degradation [16]. Some extractives with an unpleasant smell can be emitted during the production of WPCs [17] or may create an unfavorable effect on the thermal stability of the final product [18]. Since different wood species have a different composition of the major components, a difference in thermal stability can be expected. The amount of extractives differs between wood species, but is also influenced

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by factors, such as growing conditions and the amount of heartwood [15].

The influence of wood extractives on the thermal stability of wood and WPCs has not been extensively studied. Mészáros et al. [19] found that extracted wood samples showed slight differences in the thermal behavior compared to the un-extracted samples. They found that the thermal degradation of the wood extractives from *Robinia pseudoacacia* occurs in two stages: the first one occurs between 130 and 250 °C, while the second decomposition stage takes place between 250 and 550 °C. The second stage occurs at similar temperatures as the other wood components, which explains the slight difference between the extracted and un-extracted wood samples. According to Várhegyi et al. [20], the removal of some extractives causes a decrease in the fixed carbon content, a decrease in char yield and a displacement of the entire thermogravimetric (TG) curve towards higher temperatures. It is therefore crucial to investigate the exact role of extractives on the thermal degradation of wood.

Extractives can be removed with a single solvent or a combination of solvents, such as ethanol, water, benzene, dichloromethane, chloroform or a mixture (1:2) of ethanol/cyclohexane [21,22]. Conventional solvent extraction and fractionation followed by high performance liquid chromatography, or gas chromatography combined with off-line derivatization has been employed to characterize wood extractives [23,24].

The aim of the work presented here was to investigate the effect of extractives on the thermal stability of four different wood species. Although extraction of wood is time consuming and expensive it might affect the thermal properties of wood and the WPCs positively. TGA was used to quantify the weight loss and to compare the thermal degradation of wood before and after different extraction steps.

2. Materials and methods

2.1. Wood samples

Wood samples from four different species, namely *Quercus alba*, *Pinus radiata*, *Eucalyptus grandis* and *Acacia cyclops* were investigated. The samples were converted into chips with a Wigger pilot size chipper, containing four blades adjusted to produce approximately 20 mm long woodchips. These chips were further comminuted with a Retsch mill to a particle diameter of about 180 μm.

2.2. Chemical analysis

Cyclohexane purchased from Sigma–Aldrich and absolute ethanol from Merck Chemicals (PTY) Ltd. (Saarchem), were used to determine the amount of organic extractives in the wood samples. Sulphuric acid purchased from Merck Chemicals (PTY) Ltd. (Saarchem) was used for the lignin determination. Acetyl acetone from Sigma–Aldrich and dioxane, hydrochloric acid, methanol and diethyl ether from Merck Chemicals (PTY) Ltd. (Saarchem) were used for the cellulose determination.

Solvent ethanol/cyclohexane (E/C) and water extraction were performed according to Tappi standard T 264 om-88 and the Klason lignin content was determined according to Tappi 222 om-88 [22]. The cellulose content was determined with the Seifert method [25].

2.3. Characterization

Thermogravimetric analyses (TGA) were conducted with a PerkinElmer TGA 7 Thermogravimetric Analyzer. This method comprises the measure of changes in weight as a function of temperature changes with a resolution of 0.1 mg in a nitrogen atmosphere. 4 mg wood were analyzed, and heated from 20 to 900 °C at a rate of 20 °C/min in a platinum sample pan.

Table 1
Chemical composition of investigated wood species

	Q. alba	*P. radiata*	*A. cyclops*	*E. grandis*
Cellulose content (%)	38.11	31.84	47.43	37.71
Lignin content (%)	25.01	22.13	22.72	20.61
E/C extractives (%)	02.03	03.45	02.41	01.23
Hot-water extractives (%)	03.16	02.35	03.63	01.78
Others (%)	22.37	40.23	23.81	38.67

3. Results and discussion

3.1. Chemical composition of wood

In order to determine the effect of the chemical composition of different wood types on the thermal stability, the amount of cellulose, Klason lignin, hot-water (HW) extractives and E/C extractives was determined for all samples. The degradation temperature of wood is expected to be related to the heat stability of the individual wood components and differences in the thermal stability between wood species can be attributed to the variation in chemical composition [26].

All samples were measured in triplicate and the results are presented in Table 1, which shows the content of cellulose, lignin, HW and E/C extractives of the investigated wood species. The hemicelluloses form part of the remaining percentage.

The cellulose content was determined to be 47% for *A. cyclops*, 38% for *Q. alba* and *E. grandis* and 32% for *P. radiata*. *Q. alba* had the highest lignin content with 25%, followed by *A. cyclops* and *P. radiata* with 23% and 22% and *E. grandis* with 21%.

The amount of the extractives soluble in organic solvent (E/C) and HW of the different wood sample are somewhat different. *Q. alba*, *A. cyclops* and *E. grandis* contain more polar extractives, which is indicated by the higher amount of HW than E/C extractives. The E/C-soluble extractive content, comprising waxes, fats, resins and some gums [22], was the highest in *P. radiata*, followed by *A. cyclops*, *Q. alba* and *E. grandis*. The water-soluble extractive content, comprising of tannins, gums, sugars, starches and coloring matters [22] is the highest in *A. cyclops* followed by *Q. alba*, *P. radiata* and *E. grandis*.

The four investigated wood species are clearly distinguishable by differences in their composition and a different thermal behavior can therefore be expected.

3.2. Thermal stability of wood

The thermal stability of the four wood species was determined with TGA, which measures the weight loss of the sample with increasing temperature, as shown in Fig. 1a. All curves show a small weight loss before 100 °C, which can be attributed to the evaporation of water. The weight loss rate gradually increased above 200 °C and a distinct weight loss appeared between 200 and 400 °C. All four wood samples exhibit a loss of about 75 wt.% at 400 °C. *Q. alba* has the highest weight loss and *P. radiata* the lowest.

DTG curves displayed in Fig. 1b allow a comparison of the thermal stability between the wood species. With exception to *P. radiata*, the degradation profiles show two peaks at and a shoulder at T_1 and it becomes apparent that the degradation between 200 and 400 °C is actually a two-step process. The first degradation event was observed between 218 and 260 °C, and can be attributed to decomposition of hemicelluloses and the slower decomposition

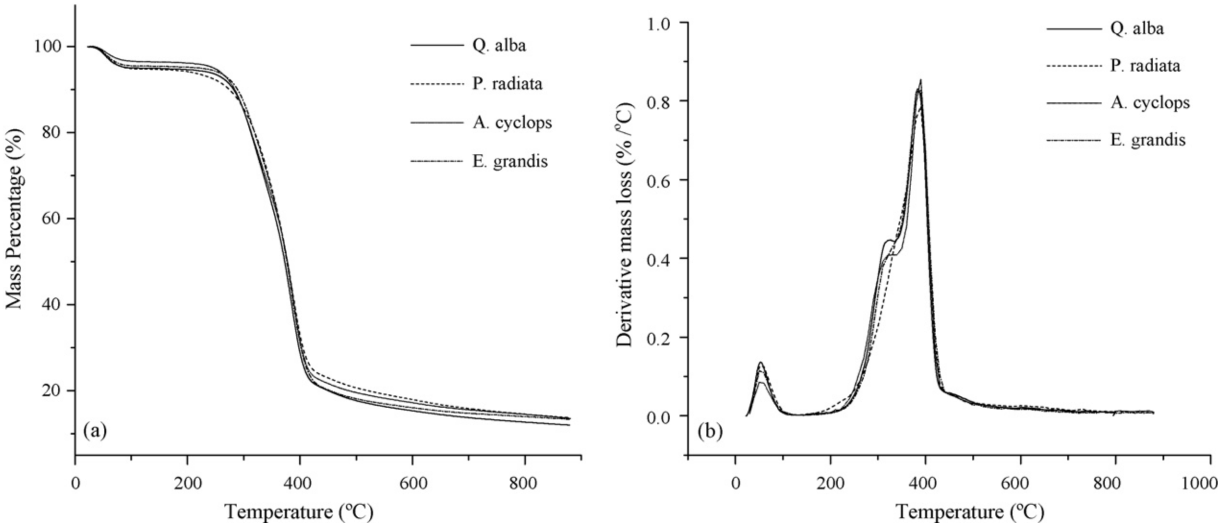

Fig. 1. (a) TGA and (b) DTG curves of the four wood species.

of lignin. The second degradation above 350 °C can be attributed to the degradation of cellulose. These results are consistent with previously published results [27–30]. It is known that the hemicelluloses decompose before lignin and cellulose [27–28]. Marcovich and Villar [29] claimed that around 175 °C, some wood components start to decompose: low temperature degradation at a low rate occurs in lignin and hemicelluloses. Nunez et al. [5] cited that the degradation of cellulose takes place at high temperature associated with pyrolitic degradation of lignin. Kim et al. [30] confirmed that the hemicelluloses degrade between 180 and 350 °C, the lignin degrades between 250 and 500 °C, and the degradation of cellulose take place between 275 and 350 °C.

Table 2 shows the various transitions of all four wood species before and after extractions. T_{o} is the onset temperature, at which degradation starts, T_1 and T_2 are decomposition temperatures that appear as shoulders in the TGA and as peaks in the DTG graph. T_{f} is the final decomposition temperature above which the sample does not degrade any further and a mass residue remains.

The decomposition curve of *P. radiata* did not show a shoulder at T_1. The first peak appeared below 100 °C and can be assigned to the evaporation of water. The second peak at 388 °C was broad, which means there was overlapping between the decomposition of hemicelluloses, cellulose, lignin and wood extractives. According to Mészáros et al. [19], in some wood species, the decomposition of the wood extractives can continue up to 550 °C, overlapping with the decomposition of polysaccharides and lignin.

Table 2

Thermal degradation temperatures and residue weight of the investigated wood species

Wood species	T_{o} (°C)	T_1 (°C)	T_2 (°C)	T_{f} (°C)	Residue at 600 °C (%)
Q. alba	258.40	321.71	385.69	538.03	12.01
P. radiata	218.93	–	388.41	513.82	13.67
A. cyclops	245.18	321.23	387.50	524.82	13.53
E. grandis	234.58	324.74	385.39	507.71	13.34
After HW extraction					
Q. alba	268.51	328.01	389.38	542.64	09.16
P. radiata	235.40	–	399.07	521.89	09.26
A. cyclops	254.72	316.71	390.99	530.37	11.96
E. grandis	251.80	316.71	392.61	515.32	08.64
After E/C extraction					
Q. alba	259.54	322.05	385.28	547.05	08.80
P. radiata	235.54	–	399.07	530.09	09.54
A. cyclops	256.03	318.67	392.61	538.47	11.36
E. grandis	238.30	315.64	391.49	526.86	11.17
After HW and E/C extraction					
Q. alba	267.97	324.74	391.49	542.63	07.40
P. radiata	240.87	–	399.07	515.28	08.17
A. cyclops	266.58	321.71	396.04	526.51	09.57
E. grandis	250.63	317.16	394.49	512.86	09.36

The onset temperatures T_{o} (the beginning of hemicelluloses decomposition), the first decomposition temperatures T_1 (the shoulders in Fig. 1b), the second decomposition temperatures T_2 (the peaks in Fig. 1b), the final decomposition temperatures T_{f} and the residual weight at 600 °C are summarized in Table 2. Comparison of T_{o} indicates that *P. radiata* and *E. grandis* started to degrade at lower temperatures than *A. cyclops* and *Q. alba*, which means that at low temperatures wood from *P. radiata* and *E. grandis* is less stable than wood from *Q. alba* and *A. cyclops*. This could be explained by the higher cellulose content determined in *A. cyclops* and *Q. alba*. With exception of *P. radiata*, T_1 did not differ significantly for the four wood species and appeared around 322 °C. T_2 is in a similarly close temperature range around 386 °C for all four species. The final decomposition temperature T_{f} of *Q. alba* and *A. cyclops* occurred at higher temperatures than for *P. radiata* and *E. grandis*. This indicates that wood from *P. radiata* and *E. grandis* is also less stable at higher temperatures than the other two species, which could be attributed to the lower lignin content. Tserki et al. [31] illustrated that the lower stability of wood at low temperatures can be attributed to a high lignin and hemicelluloses content, while the high stability at low temperatures is due to a higher cellulose content. They also demonstrated that lignin appears to be more heat resistant than hemicelluloses and cellulose at high temperatures due to its low degradation rate. This confirms that the decomposition of lignin occurs in a wider temperature range than that of hemicelluloses and cellulose [19]. At 600 °C, no significant quantitative difference in wood residue between the four wood species could be determined. The highest wood residue was determined for *P. radiate* and the lowest for *Q. alba*.

It can be concluded that a higher cellulose and lignin content of wood leads to a better thermal stability over the entire temperature range. *Q. alba* and *A. cyclops* showed better thermal stability than *P. radiata* and *E. grandis*.

3.3. *Thermal stability after hot-water extraction*

There have been several reports about the effect of the water-soluble extractives on the mechanical and physical properties of

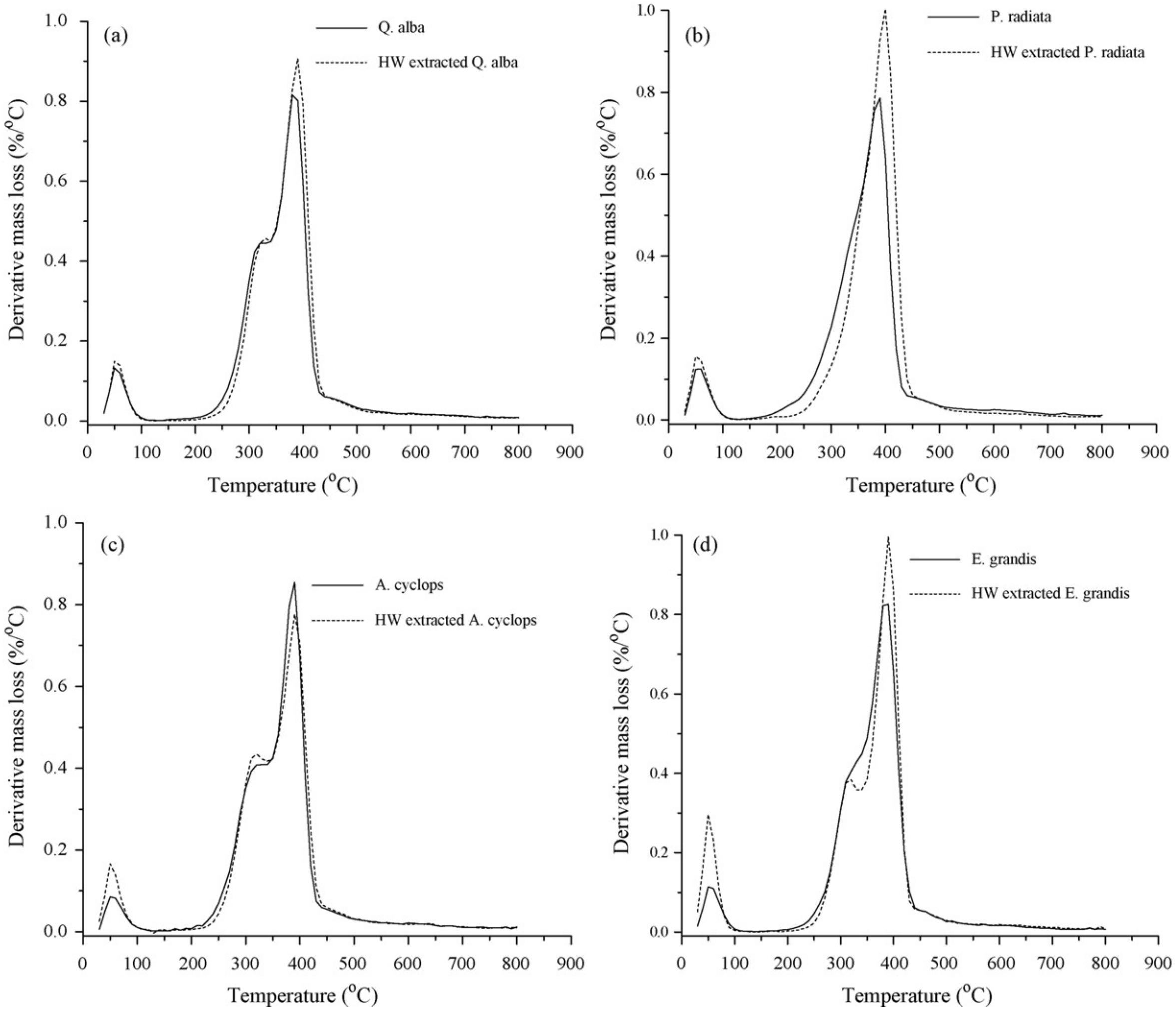

Fig. 2. DTG curves before and after hot-water extraction. (a) *Q. alba*, (b) *P. radiata*, (c) *A. cyclops* and (d) *E. grandis*.

wood materials [21,32,33]. Although these extractives impart the favorable properties of nature color, odor, taste, density and decay, they are most troublesome because they mainly cannot keep water out of the wood that is in service. Water-soluble extractives can have a significant effect on the wettability, transverse crushing strength, young's modulus, density, durability, shrinkage, hygroscopicity and permeability, etc. In contrast, the effects of these extractives on the thermal stability of wood materials have been very little documented [20,26].

The degradation profiles of all species changed after HW extraction, as displayed in Fig. 2. Differences occur in the width and height of the shoulders and the peaks. In the case of *Q. alba* and *E. grandis*, the areas under the shoulders and peaks become smaller after extraction and the peaks are narrower. For *A. cyclops* the shoulder is higher and the peak is smaller and narrower after extraction, whereas for *P. radiata* no shoulder can be detected and the peak becomes more pronounced after extraction. The values before and after HW extraction of the four wood species are summarized in Table 2.

The most significant changes observed, are the changes in the position of T_o, which are increased for all four wood species after HW extraction. This indicates that the thermal decomposition of some HW extractives takes place in this temperature range and that these compounds have been partly eliminated from the wood samples by the HW extraction. Shi-fa et al. [17] subjected wood sawdust and polypropylene powder to heat treatment of about 290 °C for 8 min, similar to that employed on an industrial scale. They found that an unpleasant smell was emitted when the inner temperature of the wood sawdust reached 130 °C and they concluded that phenols, acids, aldehydes, ketones, furan derivatives and nitrogen containing compounds were most likely responsible for this and most of these compounds are hot-water extractives.

These HW extractives decompose at lower temperatures than hemicelluloses and appear to protect the hemicelluloses. Ohtani et al. [34] assumed that hot-water extractives suppress the decomposition and loss of hemicelluloses by acting as a protector for hemicelluloses during the alkaline cooking.

T_1 (the shoulder) was shifted to a higher temperature for *Q. alba* and to lower temperatures for *A. cyclops* and *E. grandis*. This is probably due to the extraction of low molecular weight polysaccharides and some hemicelluloses (e.g. arabinogalactant) from the wood samples, especially in the case *A. cyclops* and *E. grandis*, as HW extraction is known to extract these materials [35]. As before the extraction, no shoulder was observed for *P. radiata*. T_2 and T_f were shifted to higher temperatures for all four wood species. The deviations in the T_o, T_1, T_2, T_f and the shape and the position of the peaks in DTG curves indicate, that the degradation of HW extracted wood occurs at higher temperatures and over a slightly narrower temperature range. This is in agreement with results reported by Várhegyi et al. [20], who found that HW extraction causes a dis-

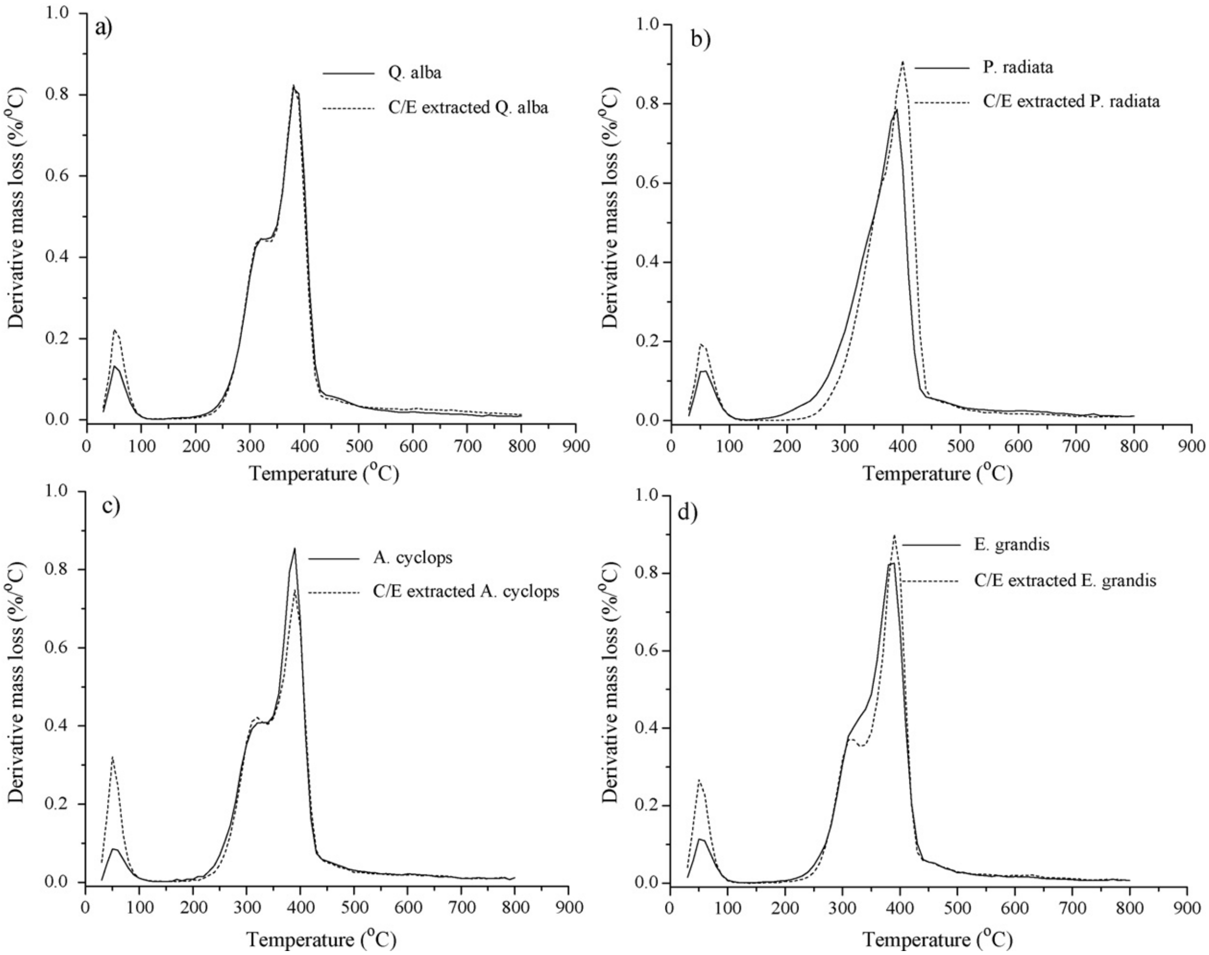

Fig. 3. DTG curves before and after E/C extraction. (a) *Q. alba*, (b) *P. radiata*, (c) *A. cyclops* and (d) *E. grandis*.

placement of TGA curves to higher temperatures. Finally, there is a significant decrease in the in the quantity of wood residue for the four wood species. In agreement with previously published results, hot-water washing and extraction causes a significant decrease in the char yield of some wood species [20,36], removes a portion of the cell structure (low molecular weight polysaccharides) [26] and eliminates some inorganic matter or extractives [20], which may catalyze the decomposition of natural polymers resulting in a lower decomposition temperature and a higher amount of char [19,20,37].

The differences in the thermal stability of wood before and after HW extraction proves that some extractives were removed, that have an influence on the thermal stability of wood and also that the thermal decomposition of these extractives occurs in a wide temperature range between ±200 and 500 °C.

3.4. Thermal stability after E/C extraction

The effects of ethanol/cyclohexane extraction on the thermal stability of wood appear to be less pronounced compared to HW extraction, as illustrated in Fig. 3. The DTG curves of *Q. alba* before and after E/C extraction are nearly identical and the changes are negligible. In the cases of *P. radiata*, *A. cyclops* and *E. grandis* T_{o}, T_2 and T_{f} were shifted towards higher temperatures, while T_1 showed a small shift towards lower temperatures.

The effect of E/C extraction on the thermal stability of *P. radiata* and *E. grandis* was more distinct than for *Q. alba* and *A. cyclops*. For *P. radiata* the area under the peak became smaller and the peak narrower and the effect of E/C extraction was most significant. T_{o}, T_2 and T_{f} shifted to significantly higher temperatures. For *E. grandis* the height of the shoulder was decreased and the peak increased, which results in a net decrease of the area under the shoulder and the peak. T_{o}, T_2 and T_{f} of *E. grandis* showed a shift towards higher temperatures, while T_1 was decreased.

The quantity of wood residue decreases for all four wood species after E/C extraction and is more significant for *P. radiata* and *Q. alba*, than for *A. cyclops* and *E. grandis*. The wood residue after E/C extraction is comparable to the residue after HW extraction.

As for the HW extraction, it can be noted that the changes of the TGA curves after E/C extraction prove the elimination of some organic extractives, which influence the thermal stability of wood. The thermal decomposition of the E/C extractives occurs in a narrower temperature range, than that of HW extractives.

It can be concluded here that E/C extraction caused less improvement in the thermal stability of wood than hot-water extraction. This is because hot-water extraction eliminates a greater quantity of materials, removes a portion of the cell structure and extracts some inorganic extractives or matters.

3.5. Thermal stability after HW and E/C extraction

In order to achieve complete extraction, solvent and water extraction was performed. The results are displayed in Table 2 and Fig. 4. The area under the shoulder and the peaks decreased and the peaks became narrower. T_{o}, T_2 and T_{f} of all wood sam-

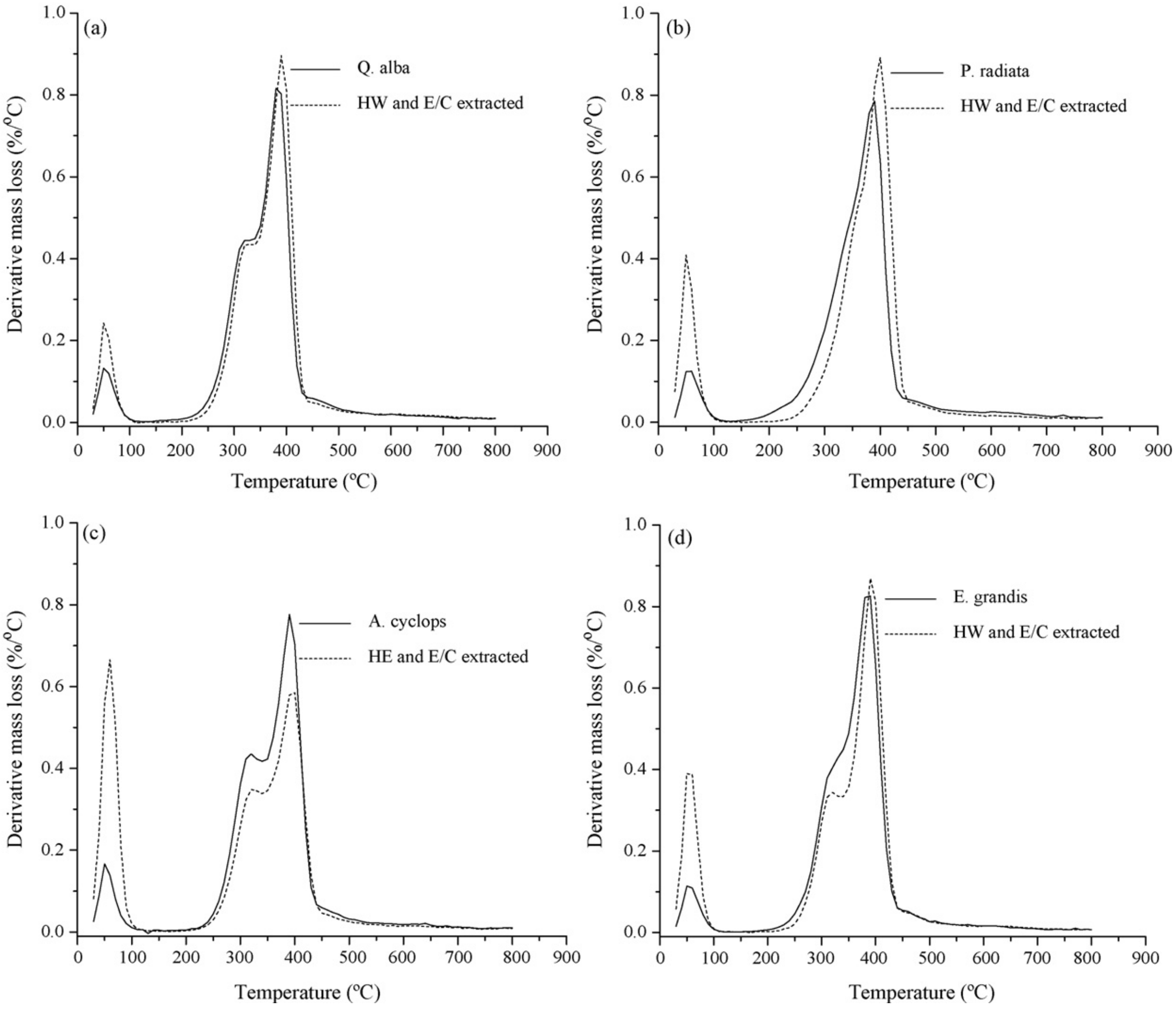

Fig. 4. DTG curves before and after HW and E/C extraction. (a) *Q. alba*, (b) *P. radiata*, (c) *A. cyclops* and (d) *E. grandis*.

ples showed a clear shift to higher temperatures. T_1 of *Q. alba* was shifted to a higher temperature, while T_1 of *A. cyclops* and *E. grandis* shifted to lower temperatures. A significant decrease in the quantity of wood residue could be observed in all four wood species.

These changes prove again, that the elimination of extractives increases the thermal stability of wood. The changes after HW and E/C extraction were more pronounced than for HW or E/C extraction alone, which could be expected, as each individual process affects different extractives, which all have an impact on the thermal stability of wood.

When comparing the thermal stability of the wood species, the order remains the same as for the un-extracted wood: oak and acacia wood shows a better thermal stability than pine and eucalyptus wood.

3.6. *Thermal stability of extracted cellulose*

The thermal stability of pure cellulose from all four wood species was determined and slight differences were found, which shows that differences in cellulose composition exist between the wood species and cellulose is a unique material [38]. A comparison of the thermal degradation between the fours cellulose types is shown in Fig. 5.

The degradation in all cellulosic materials occurred around 360 °C. Zickler et al. [39] claimed that at temperatures below 310 °C wood cellulose was hardly degraded, whereas above 360 °C the kinetics was too fast to allow in situ measurements. Also, within this temperature range, the thermal degradation mechanisms of cellulose were found to differ along and across cellulose fibrils. The extracted cellulose showed better thermal stability than the wood

Table 3
Thermal degradation temperatures and residue weight of extracted cellulose

Extracted cellulose from	T_o (°C)	T_1 (°C)	T_2 (°C)	T_f (°C)	Residue at 600 °C (%)
Q. alba	320.66	–	370.25	550.15	0.006
P. radiata	320.87	–	378.07	550.32	02.63
A. cyclops	329.99	–	373.23	552.11	01.91
E. grandis	342.18	–	387.76	556.93	01.91

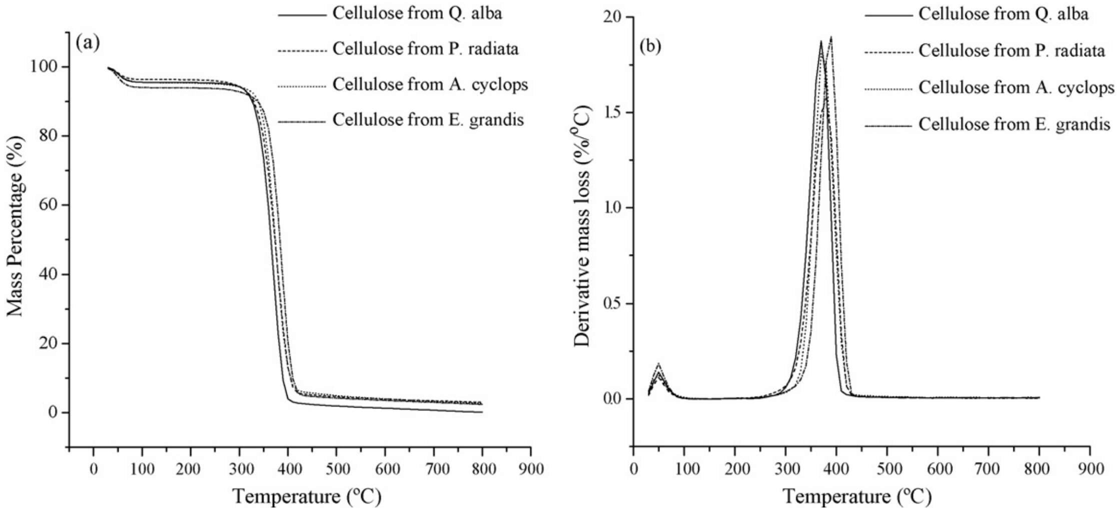

Fig. 5. TGA and DTG curves of cellulose from the four wood species.

itself. Cellulose from *E. grandis* and *A. cyclops* had a higher thermal stability than that from *Q. alba* and *P. radiata*, as shown in Table 3. Cellulose from *Q. alba* and *P. radiata* showed almost the same T_o and T_f, while T_2 and the residue were significantly higher for *P. radiata*. The residue from cellulose of *A. cyclops* and *E. grandis* was the same, but T_o, T_2 and T_f were higher for cellulose from *E. grandis*. It has been previously stated that [40,41] the stability of cellulose depends on the crystallinity and the intermolecular hydrogen bonded domains and difference in thermal stability can be ascribed to the crystallinity of these materials [38,39,42]. Wang et al. [43] mentioned that a higher content of acid sulfate groups in cellulose leads to lower degradation temperatures. Hu et al. [44] stated that the degradation efficiency of cellulose increases with pH. In fact, the degradation of cellulose appears to be not fully understood and is still a field of ongoing research.

As illustrated above all extraction processes resulted in an improvement of the thermal stability of wood, independent of the species. This means that it would be preferable to use extracted wood as a filler material for the production of WPCs, because the processing temperature could be raised to above 200 °C. This would reduce the limitation in available plastics that can be processed together with wood and also the applications in which WPCs can be used. The effects of HW extraction appeared to more profound than that the effects of E/C extraction. The more complete the removal of wood extractives is, the closer the thermal stability of wood will be to that of cellulose.

4. Conclusion

The effects of wood extractives on the thermal stability of different wood species were investigated. The chemical composition of the four wood species – *Q. alba*, *P. radiata*, *A. cyclops* and *E. grandis* – and the thermal stability were determined. The results showed significant differences in the chemical composition and degradation characteristics. Wood with a high cellulose and lignin content showed a better thermal stability.

The effects of HW, E/C and combined HW and E/C extraction on the thermal stability of the four wood species were studied. In all cases, TGA and DTG curves shifted to higher temperatures, which means an improvement in thermal stability. The effect of combined extractions was more pronounced than that of any one individual extraction, which indicates that the thermal stability was better the more complete the removal of extractives was. E/C-extraction alone was less effective for the improvement in thermal stability than HW extraction, since hot-water eliminates a greater quantity of extractives, some inorganic materials and a portion of the cell structure (low molecular weight polysaccharides).

The thermal stability of the extracted cellulose from the four wood species was also determined and exhibited a higher thermal stability than the wood itself, which would allow the processing with a polymer above 200 °C. Further work is, however, required to fully understand the effects of wood extractives on the thermal stability of WPCs, such as a comparison of the thermal stability of WPCs filled with un-extracted wood species and extracted wood.

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