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COMPARISON OF ACTIVATION ENERGY OF THERMAL DEGRADATION OF HEAT STERILISED SILVER FIR WOOD TO LARVAL FRASS REGARDING FIRE SAFETY

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ABSTRACT

Heat sterilization is a practical and environmentally friendly method to kill all forms of pests (eggs, larvae, adults) in solid wood materials, but it can influence wood properties. Thermogravimetry is a useful tool for the analysis of thermal degradation of wood and wood-based materials. Methods of chemical analysis, thermal analysis and statistical analysis were used on the samples of silver fir wood and larval frass treated at 60 °C and 120 °C for 10 hours. The activation energies of thermal degradation of wood and frass samples changed negligibly. Correlations between wood components and activation energies were found to be high for wood samples but not for larval frass samples. Therefore, fire safety of wooden parts of buildings is not deteriorated by heat sterilization and can be used without significant alteration of the wood composition and properties.

Keywords: silver fir wood, *Hylotrupes bajulus,* heat sterilization, thermogravimetry, activation energy.

INTRODUCTION

Wood as a natural material can be infested with various kinds of wood-destroying insects. New methods to protect wooden materials from this infestation are constantly looked for, and the known methods are being improved. One of these methods is heat sterilization. Heat sterilization is a practical and environmentally friendly treatment to kill all forms of pests (eggs, larvae, adults) in solid wood materials and prevent the transfer of pests between regions and states (WANG *et al.* 2010). The current international standard for heat sterilization of solid wood packaging materials is the International Standard for Phytosanitary Measures (ISPM) Pub. No.15, "Guidelines for Regulating Wood Packaging Material in International Trade," which requires heating wood to a minimum core temperature of 56 °C for a minimum of 30 min (WANG 2010). According to the same standard for heat sterilization of firewood, there are required more severe conditions, the core temperature must be held at 71 °C for 75 min (WANG *et al.* 2010). During the heat sterilization of the wood in building structures, wood is treated with air heated to the temperature of 120 °C, where it is necessary to achieve at least the temperature of 55 °C – 60 °C in the wood cross-section for the period of 1 h. It is sufficient for the coagulation of proteins and killing all forms (eggs, larvae, adults) of wood-destroying insects and at the same time, it reduces the amount of terpenes present in wood which is then less attractive to

insects. On the other hand, the increased temperature can deteriorate mechanical and fire-technical characteristics of wood and change its physical properties (KACIK *et al.* 2012; KUBS *et al.* 2016; MARTINKA *et al.* 2018; MARTINKA *et al.* 2017; OSVALD and GAFF 2017). One of the wood-destroying insects is *Hylotrupes bajulus*. It is structural insect pest of world-wide importance, as this species has been introduced from Europe to all major continents (BECKER 1979). The larvae infest and damage most of the common seasoned coniferous timbers used in buildings, and their tunnelling often results in loss of structural integrity of infested wood and financial losses caused by the treatment and replacement of damaged wood (FETTKOTHER *et al.* 2000).

Thermogravimetry (TG) is often used to analyze complex mixtures, because of the characteristic thermal decomposition temperature of each component (CARRIER *et al.* 2011). It was found that the pyrolytic decomposition of wood in the inert atmosphere occurs at 250 °C – 300 °C for hemicelluloses, 300 °C – 350 °C for cellulose, and 300 °C – 500 °C for lignin (SONG *et al.* 2004). There are various methods to calculate the activation energy of thermal degradation from the data obtained by TG, for example, Ozawa-Flynn-Wall method, Kissinger method, Freeman and Carroll method, Coats and Redfern method, and others (HATAKEYAMA and LIU 1998).

The kinetic equation of common type can be written as follows:

$$\frac{d\alpha}{dt} = k(T)f(\alpha) \tag{1}$$

where $f(\alpha)$ is a function, the type of which depends on the reaction mechanism, $k(T)$ the temperature dependent rate constant, T the absolute temperature, t the time, and α the degree of transformation, which can be calculated by the formula:

$$\alpha = \frac{w_0 - w}{w_0 - w_f} \tag{2}$$

where w is the mass fraction present at any time, w_0 is the initial mass fraction, and w_f is the final mass of the sample. The temperature dependence of the rate constant is usually described by the Arrhenius equation:

$$k = Aexp\left(-\frac{E}{RT}\right) \tag{3}$$

where A is a pre-exponential factor, E the activation energy, R the gas constant (8.314 $Jmol^{-1}K^{-1}$). Under constant heating rate:

$$\frac{dT}{dt} = q = constant \tag{4}$$

and after substitution in (1) and some transformations:

$$\int_0^\alpha \frac{d\alpha}{f(\alpha)} = \frac{A}{q}\int_0^T exp\left(-\frac{E}{RT}\right)dT \tag{5}$$

Then denote Eq. (5) with:

$$g(\alpha) = \int_0^\alpha \frac{d\alpha}{f(\alpha)} = \frac{A}{q}\int_0^T exp\left(-\frac{E}{RT}\right)dT \tag{6}$$

where $g(\alpha)$ is the integral function of conversion.

Coats-Redfern used an asymptotic approximation for the resolution of Eq. (6), obtaining:

$$\ln\left(\frac{g(\alpha)}{T^2}\right) = \ln\left(\frac{AR}{qE}\right) - \frac{E}{RT} \tag{7}$$

Plotting in $\ln[g(\alpha)/T^2]$ versus $1/T$ should result in a straight line with a slope $-E/R$ and an intercept providing the values E and A. There are different expressions of $g(\alpha)$ for the

different solid-state mechanisms. Assuming a reaction order of 1 (YANG *et al.* 2010), the reaction mechanism function is $f(\alpha)=(1-\alpha)$. The integral equation can be written as:

$$\ln\left(\frac{-ln(1-\alpha)}{T^2}\right) = \ln\left(\frac{AR}{qE}\right) - \frac{E}{RT} \quad (8)$$

Coats and Redfern method (Eq. (8)) (COATS and REDFERN 1964) was used in this article to calculate activation energies of thermal degradation of wood and larval frass samples. This method is simple and suitable for calculations of activation energy of isothermal thermal analysis, as long as the plot of $\ln[g(\alpha)/T^2]$ versus $1/T$ is linear (EBRAHIMI-KAHRIZSANGI and ABBASI 2008).

To our knowledge, the influence of the heat treatment on the chemical composition and activation energy of thermal degradation of *Hylotrupes bajulus* frass has not been reported in any literature yet.

The aim of this work is therefore to compare activation energy of thermal degradation of heat sterilised silver fir wood to larval frass in regard to fire safety, using methods of thermal analysis.

MATERIALS AND METHODS

Materials

Silver fir wood (*Abies alba*) from two beams infected by *Hylotrupes bajulus* was mechanically disintegrated to sawdust using a laboratory mill POLYMIX PX-MFC 90 D (KINEMATICA AG, Luzern, Switzerland) (KAČÍK *et al.* 2017). Particles with a size below 0.01 mm were separated (Fritsch, Germany) and divided into six parts (three samples from each beam). Two samples were saved as a reference, and the others were thermally treated. Samples of larval frass were scratched from the same infected beams used for wood samples. These samples were also divided into six parts, two of them were used as a reference, and the remaining four were thermally treated.

Methods

Heat treatment

Heat treatment was conducted using a laboratory type heating oven Memmert UNB 200 (Fisher Scientific, Loughborough, UK), which was controlled to an accuracy of ±1 °C under atmospheric pressure at the temperatures of 60 °C and 120 °C. After the temperature reached the target value, the temperature was kept constant for 10 h, because at the thermal protection of wood in building structures against pests, wood is treated for 10 h with air heated to the temperature of 120 °C where the temperature of 55 °C – 60 °C must be achieved in the cross-section of the wood beam.

Chemical analysis

Samples were extracted from the Soxhlet apparatus (Sigma Aldrich, Munich, Germany) with a mixture of ethanol and toluene according to the ASTM D1107-96 (2013b). The lignin content was determined according to ASTM D1106-96 (2013a). Holocellulose was determined using the method by WISE *et al.* (1946), and cellulose by the Seifert method (1956). Hemicelluloses were calculated as a difference between holocellulose and cellulose. All measurements were taken on four replicates at each temperature. The data were calculated as a percentage of oven-dry weight per unextracted wood.

Thermal analysis

Thermal analyses were carried out by using the simultaneous thermal analyser NETZSCH STA 449 F3 (NETZSCH-Gerätebau GmbH, Selb, Germany). Experiments were performed

at the temperature range of 25 °C – 800 °C with the heating rate of 10 °C min^{-1} in air atmosphere. The data were acquired and analysed using the NETZSCH Proteus software.

Statistical analysis

For all parameters, multiple comparisons were first subjected to an analysis of variance (ANOVA) and significant differences between mean values of control and treated samples were determined using Duncan's multiple range test at a *p*-value of 0.05.

RESULTS AND DISCUSSION

Chemical analysis

The amounts of the extractives and the lignin in wood samples slightly decreased during the heat treatment (Fig. 1). The decrease in the lignin content was observed also for pinewood treated at this temperature range (KACIK *et al.* 2016). The polysaccharide yields decreased with the increasing treatment temperature; the amounts of the cellulose slightly increased, whilst the amounts of hemicelluloses decreased (Fig. 1). Similarly, in pine wood treated by the same method, the holocellulose content decreased (KACIK *et al.* 2015).

The amount of extractives in larval frass samples decreased during the heat sterilisation as well (Fig. 1). On the other hand, the amount of the lignin in these samples increased. Cellulose content decreased, after initial increase; on the contrary, hemicellulose content increased, after an initial decrease. The holocellulose decreased during the heat treatment (Fig. 1).

Untreated as well as heat-treated samples of larval frass contained more extractives than wood samples. The same was observed for the lignin content. The amounts of the cellulose were similar in both, wood samples and larval frass samples. On the other hand, the hemicellulose yields in the larval frass samples were about 12% lower than in the wood samples. The holocellulose yields were about 6% lower in the larval frass samples than in the wood samples (Fig. 1).

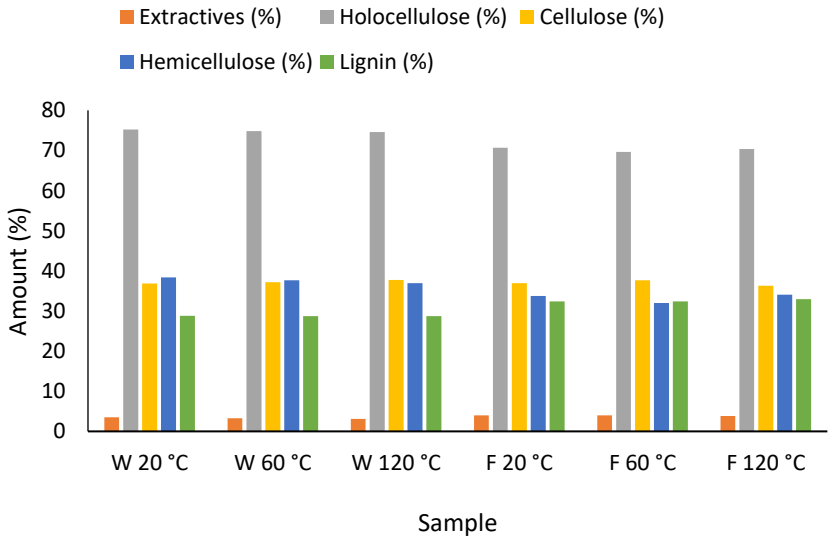

Fig. 1 The amounts of main wood components in samples (W = wood, F = frass).

Statistical analysis

Significant ($p < 0.05$) change in chemical composition of wood samples was only in the hemicelluloses between the untreated sample and the sample treated at 120 °C. Other chemical changes in wood samples were insignificant (Fig. 2–6).

In larval frass samples, the significant changes were observed for holocellulose and hemicellulose between samples treated at 20 °C and 60 °C, and for hemicellulose, it was also between samples treated at 60 °C and 120 °C and for the cellulose between samples treated at 60 °C and 120 °C as well. Chemical changes of extractives and the lignin in larval frass samples were insignificant (Fig. 2–6).

Changes between the amounts of extractives from untreated wood and extractives from untreated larval frass samples were not significant as well as changes in cellulose. Also, the changes in the cellulose amounts observed at 60 °C between the wood and larval frass samples were insignificant. Other changes between the wood and larval frass samples were significant (Fig. 2–6).

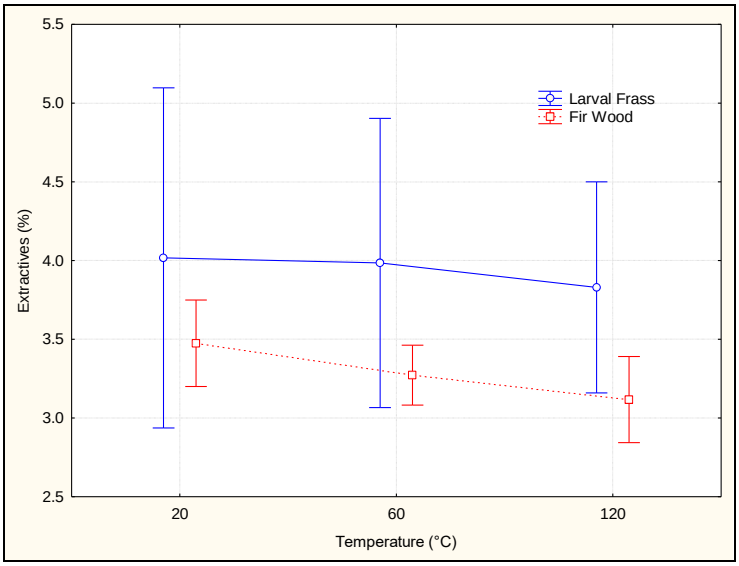

Fig. 2 Influence of temperature on content of extractives in wood and larval frass samples with 95% confidence intervals.

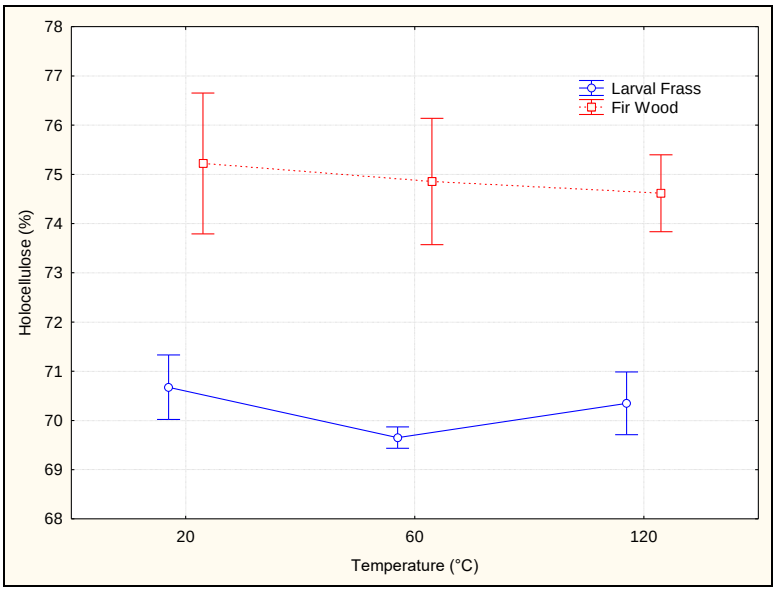

Fig. 3 Influence of temperature on content of holocellulose in wood and larval frass samples with 95% confidence intervals.

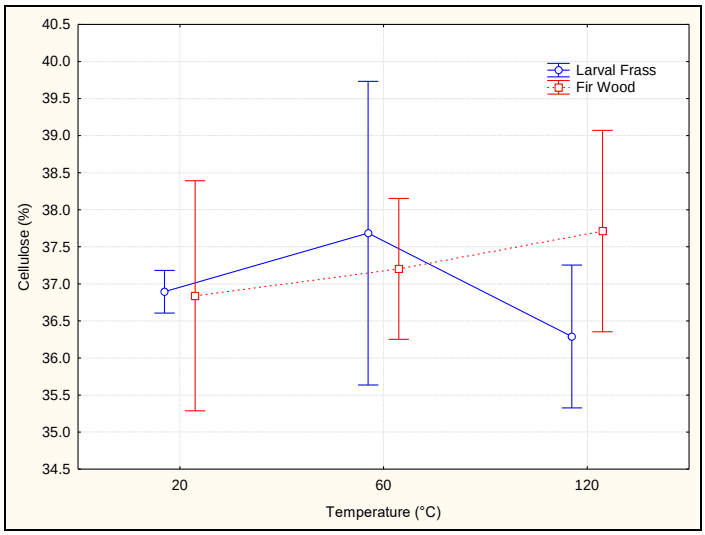

Fig. 4 Influence of temperature on content of cellulose in wood and larval frass samples with 95% confidence intervals.

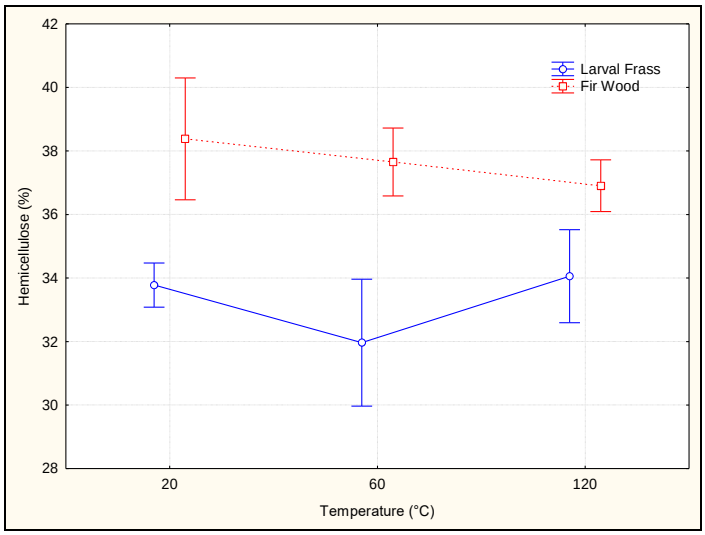

Fig. 5 Influence of temperature on content of hemicellulose in wood and larval frass samples with 95% confidence intervals.

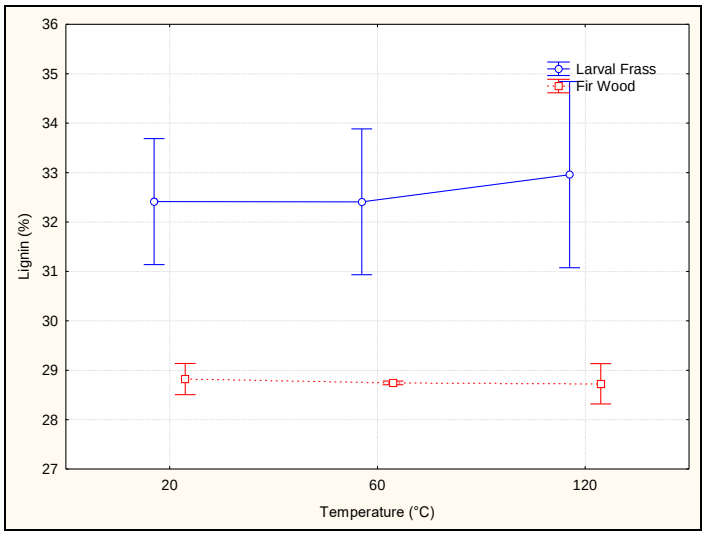

Fig. 6 Influence of temperature on content of lignin in wood and larval frass samples with 95% confidence intervals.

Thermal analysis

Three regions were determined on thermograms of wood samples according to the approximate starting and ending points of the DTG curve which represents a thermal breakdown of the individual components of wood. The first region corresponded to the degradation of the extractives and hemicelluloses, the second one corresponded to the degradation of the cellulose, and the last one showed the degradation of the lignin.

The highest activation energies were calculated for the second region (Tab. 2). The activation energy of wood samples decreased with the treatment temperature in the first region and increased in the second region. In the third region, it decreased. In the first and third regions of larval frass samples activation energy increased after an initial decrease. It was reversed in the second region. The activation energies calculated by other authors (CHEN *et al.* 2015; GASPAROVIC *et al.* 2012; YORULMAZ and ATIMTAY 2009) are higher than our results. We assume it is because we used smaller particles for analysis and air atmosphere instead of nitrogen atmosphere which was used by other authors.

Tab. 1 Data for activation energies calculation.

Sample	w_0 (mg)	w (mg)	w_f (mg)	T (°C)	T (K)	α	ln	1/T	c
W201	6.02	6.01	0.37	20	293.15	0.00	–17.70	0.0034	0
W202	6.02	5.86	0.37	193	466.15	0.03	–15.83	0.0021	–1472
W203	6.02	2.21	0.37	375	648.15	0.67	–12.83	0.0015	–4984
W204	6.02	0.41	0.37	552	825.15	0.99	–11.84	0.0012	–3000
W601	6.85	6.84	0.40	20	293.15	0.00	–17.83	0.0034	0
W602	6.85	6.71	0.40	203	476.15	0.02	–16.16	0.0021	–1276
W603	6.85	2.49	0.40	383	656.15	0.68	–12.85	0.0015	–5738
W604	6.85	0.42	0.40	616	889.15	1.00	–11.83	0.0011	–2570
W1201	6.28	6.27	0.35	20	293.15	0.00	–17.75	0.0034	0
W1202	6.28	6.16	0.35	203	476.15	0.02	–16.20	0.0021	–1182
W1203	6.28	2.26	0.35	385	658.15	0.68	–12.85	0.0015	–5753
W1204	6.28	0.36	0.35	652	925.15	1.00	–11.72	0.0011	–2586
F201	6.18	6.17	0.75	20	293.15	0.00	–17.66	0.0034	0
F202	6.18	6.00	0.75	199	472.15	0.03	–15.69	0.0021	–1523
F203	6.18	2.30	0.75	398	671.15	0.71	–12.79	0.0015	–4609
F204	6.18	0.75	0.75	798	1071.15	1.00	–11.88	0.0009	–1649
F601	6.33	6.32	1.12	20	293.15	0.00	–17.62	0.0034	0
F602	6.33	6.17	1.12	206	479.15	0.03	–15.80	0.0021	–1373
F603	6.33	2.38	1.12	394	667.15	0.76	–12.65	0.0015	–5349
F604	6.33	1.13	1.12	798	1071.15	1.00	–12.05	0.0009	–1068
F1201	6.66	6.65	0.60	20	293.15	0.00	–17.77	0.0034	0
F1202	6.66	6.48	0.60	201	474.15	0.03	–15.82	0.0021	–1497
F1203	6.66	2.54	0.60	387	660.15	0.68	–12.85	0.0015	–4987
F1204	6.66	0.61	0.60	798	1071.15	1.00	–11.82	0.0009	–1775

Note: W201 – F1204 denoted samples treated at temperatures 20, 60, and 120 °C.

Relationships between wood components contents and activation energies (Tab. 3) in wood samples show high correlation coefficients between all chemical characteristics (extractives, holocellulose, cellulose, and lignin) and activation energies in all three regions of thermal decomposition. On the other hand, in frass samples, high correlations were found

only for holocellulose and cellulose, probably due to different structures of main wood components in frass samples.

Tab. 2 Activation energies of analysed samples.

Sample	Treatment Temperature (°C)	Activation Energy (kJ/mol)		
		First region	Second region	Third region
Fir Wood	20	12.235	41.433	24.944
Fir Wood	60	10.605	47.704	21.363
Fir Wood	120	9.828	47.831	21.498
Larval Frass	20	12.661	38.321	13.713
Larval Frass	60	11.411	44.468	8.879
Larval Frass	120	12.446	41.464	14.755

Tab. 3 Equations and correlation coefficients of wood components contents vs. activation energies plots.

		First region	Second region	Third region
Fir Wood	Extractives	y = 6.02x – 8.97 r = 0.9797	y = –16.00x + 98.44 r = 0.8746	y = 8.62x – 5.83 r = 0.8490
	Holocellulose	y = 4.01x – 289.58 r = 0.9797	y = –10.66x + 844.34 r = 0,8746	y = 5.74x – 407.57 r = 0.8489
	Cellulose	y = –2.63x + 108.76 r = 0.9649	y = 6.83x – 208.57 r = 0.8417	y = –3.66x 158.91 r = 0.8133
	Hemicellulose	y = 1.59x – 48.99 r = 0.9713	y = –4.17x + 202.66 r = 0.8553	y = 2.24x – 61.73 r = 0.8279
	Lignin	y = 20.19x – 569.09 r = 0.9487	y = –63.35x + 1865.80 r = 0.9998	y = 35.14x – 986.94 r = 0.9994
Larval Frass	Extractives	y = –2.05x + 20.24 r = 0.3543	y = –0.35x + 42.79 r = 0.0141	y = –17.30x + 80.48 r = 0.6369
	Holocellulose	y = 1.29x – 78.51 r = 0.9909	y = –5.82x + 450.24 r = 0.9713	y = 5.46x – 371.55 r = 0.8945
	Cellulose	y = –0.78x + 41.15 r = 0.8237	y = 2.45x – 48.97 r = 0.5587	y = –4.30x + 171.29 r = 0.9626
	Hemicellulose	y = 0.11x + 8.06 r = 0.1229	y = –1.96x + 115.13 r = 0.4778	y = –0.85x + 44.54 r = 0.2040
	Lignin	y = 0.68x – 10.10 r = 0.3543	y = 0.12x + 37.64 r = 0.0141	y = 5.77x – 175.49 r = 0.6369

CONCLUSIONS

Only the amount of hemicelluloses changed significantly during the heat sterilisation of silver fir wood. In larval frass samples, the holocellulose amount decreased during the heat treatment.

The hemicellulose yields in the larval frass samples were about 12% lower in average than in the wood samples. Yields of the holocellulose were about 6% lower in average in the larval frass samples than in the wood samples. As a result, the amounts of extractives and the lignin in the larval frass samples were higher than in the silver fir wood samples.

The activation energies of thermal degradation of wood and frass samples changed negligibly. Correlations between wood components and activation energies were found to be high for wood samples but not for larval frass samples. Therefore, heat sterilisation does not deteriorate fire safety of wooden parts of buildings and can be used without significant alteration of the wood composition and properties.

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