

THERMOGRAVIMETRIC ANALYSIS, DIFFERENTIAL SCANNING CALORIMETRY AND TIME-TO-IGNITION OF WOOD MATERIALS TREATED WITH WATER GLASS FLAME RETARDANTS

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ABSTRACT

The paper investigates efficiency of silicate flame retardants on wooden fibres (used for production of medium-density fibreboards) and spruce boards. A simultaneous thermal analysis and cone calorimetry were used for the assessment. Specimens were treated with three different types of silicate water glass: sodium silicate, potassium silicate and modified silicate. Along with these, one sample of untreated fibres was tested in order to provide a baseline sample. As a result of the simultaneous thermal analysis, the ability of the treatments to increase the residual amount of biological char in samples during pyrolysis and ability of the flame retardant to be retained in these wood fibres was used to assess the efficiency of each flame retardant on the wood fibres.

KEYWORDS: Simultaneous thermal analysis, cone calorimetry, time of ignition, mass loss, fibreboard, spruce, pyrolysis.

INTRODUCTION

Wood as a thermally thick construction material is widely used in various types of buildings globally. One of the weaknesses of wooden materials are the fire resistant properties, therefore, modifications to improve these properties can be beneficial. Application of flame retardants is one of the possible material modifications that is common thanks to its efficiency (Eremina et al. 2020, Gasparik et al. 2017, Lu et al. 2019, Baysal et al. 2019).

We use water glass flame retardants for these experiments. This type of flame retardation is popular for its simple preparation and application on and into the material surface and is used not only in combination with wood materials but also with a variety of others such as coal (Fan et al. 2020) or graphite (Ustinov et al. 2017, Kasymov et al. 2019). To ensure thorough impregnation of the flame-retarding agent a vacuum/pressure impregnation is commonly used for wood materials (Lou et al. 2018, Clause et al. 2010). This was avoided in the following experiments to investigate material behavior with slender layer of flame retardant on and underneath the surface.

Testing of fire resistant properties of materials has evolved and changed through time (Makovicka Osvaldova et al. 2013, 2015) from which both rather traditional and novel methods were used, i.e. methods of cone-calorimetry and simultaneous thermal analysis (hereinafter referred to as STA). STA integrates two measurement methods, i.a. thermogravimetric analysis (hereinafter referred to as TGA) and differential scanning calorimetry (hereinafter referred to as DSC), which are widely used with wood materials (Dineff et al. 2014, Azizi et al. 2017, Grønli et al. 2002.). Results will help to assess the efficiency of silicates used as flame retardants and extend the knowledge of biochar forming in pyrolysis previously investigated by Kloss et al. (2012), Haseli et al. (2012) and Qi et al. 2016, England and Iskra (2020).

MATERIALS AND METHODS

The reaction to heat of treated hardwood and wood fibres was investigated in this paper. The density of the MDF material was about $160 \text{ kg}\cdot\text{m}^{-3}$. The STA was used to evaluate the effect of the different treatments. Water soluble silicates (water glass) flame retardants were used for treating: sodium silicate - type N37/40, potassium silicate - type K4009, and modified silicate - type K1530, all provided by a Danish company Bollerup Jensen A/S. Microscopic images of the samples in different phases of experiment are included accompanied by a brief commentary.

Sodium silicate (type N37/40) was marked as N (Danish: "Natron vandglas") for easier labelling of the samples that was also used during the experiment. Sodium silicate is made by melting the sand together with sodium carbonate which will end up as glass that is dissolved in water under high temperature and pressure resulting in the production of water glass. Its consistency is best described as a thick and clear liquid. The chemical formula of sodium silicate is $\text{Na}_2\text{Si}_3\text{O}_7$ with a pH value >12 . The density was measured to be $1.27 \text{ g}\cdot\text{ml}^{-1}$.

Potassium silicate (type K4009) was marked as K (Danish: "Kali vandglas"). It is a versatile inorganic chemical produced by melting sand together with potassium carbonate, dissolved in water afterwards. Potassium silicate is also thick and clear but it is easily soluble in water. It also achieves higher temperature tolerance, however it is more expensive. Chemical formula of potassium silicate is $\text{K}_2\text{Si}_3\text{O}_7$ and its pH value is > 12 (Bollerup Jensen A/S 2016). Its density was measured at $1.24 \text{ g}\cdot\text{ml}^{-1}$.

Modified silicate (type K1530) will be marked as S (silica sol). It is a liquid with a mat opaque colour and sharp odour. Just like the previous silicates, it is soluble in water. Modified silicate is more expensive than the other treatments, however its lower pH value is favourable for wood penetration. Its pH value was between 3-5. Its density was measured at $1.12 \text{ g}\cdot\text{ml}^{-1}$.

The first of two base materials were wood fibres typically used in MDF (medium density fibreboards). These were used for the simultaneous thermal analysis. Experiments with 4 differently treated samples were conducted. One sample consisted of untreated fibres to provide the basic data for further comparison of changes with different treatments. Other three samples were treated with various types of water glass, specifically with sodium silicate (type N37/40),

potassium silicate (type K4009) and modified silicate (type K1530). Wood materials tend to produce biochars with rather basic pH values in pyrolysis as indicated by Kloss et al. (2012) on spruce and poplar specimen. All densities were measured weighing 10 ml of the treatment and scales with resolution of 0.01 g.

Cone calorimetry was used to investigate the effect of silicate water glass flame retardants on spruce board evaluating the time to ignition at different heat fluxes (20/25/30/40/50 kW·m⁻²) and estimating the critical heat flux for each type of treatment, including untreated specimens. The spruce board was treated with the flame retardants by soaking under atmospheric pressure which was expected to impregnate specimens creating a thin layer on and under the surface. Pressure impregnation was not incorporated in this study.

For the cone calorimetry a specimen of spruce (*Picea abies*) was used. Very important property of wood is its density as it has direct impact on the flame retardant efficiency (Makovická Osvaldová et al. 2016). Average density of the spruce board was estimated to be 560 kg·m⁻³ for thickness ranging from 17 to 21 mm. Specimens were standardly square-shaped with an exposed surface area to heat flux of 100 cm². Fibres and spruce specimens were treated with all the three water glass flame retardants using the same conditions. The tests include pure (untreated) spruce specimens that undergo the same procedure for further comparison of changes.

Sample preparations

Four small bowls were prepared for all treatments (pure/N/S/K). A certain amount of MDF fibres was picked for each sample and weighed afterwards to find the real weight of basic material. One gram of the agent was diluted in approximately 5 ml of distilled water. The solution was then poured into the bowl containing fibres and stirred for approximately 5 min to achieve equal distribution of treatment into the entire sample. Afterwards, all the samples were inserted into a heating cabinet for a 24 hour drying process. A constant temperature of 100°C ± 5°C was used. Samples were cut into finer fibres and stirred again to achieve higher homogenisation. Obviously, this whole preparation process is not necessary for the pure fibres. However, the pure sample was wetted with water and dried in the heating cabinet as well, as we wanted to keep the undergoing process equal for all tested samples. The spruce board underwent the same drying process as the fibres being exposed to 100°C ± 5°C in 24 hour time span. Soaking in the water-diluted flame retardants was conducted afterwards. Specimens were soaked approximately 1 cm deep in flame retardant solution for 90 min and left for a week to dry again naturally. An optical microscope with installed camera was used to take microscopic pictures of the samples in 3 different phases of the testing, i.a. before, after STA and after furnace experiments, using the 20x magnification. The same samples were used for all phases of the microscopic photos. For the simultaneous thermal analysis, consisting of thermogravimetric analysis and differential scanning calorimetry, a STA 449 F3 Jupiter device manufactured by Netzsch was used. Samples were weighed with resolution of 1 µg. Temperature range of the measurements was set to 35-1000°C with heating rate set to 10 K·min⁻¹. Before starting the STA temperature ramp the sample holder was evacuated. The pyrolysis atmosphere during the measurements consisted of 100% pure nitrogen. For further analysis of the results Proteus 6.1.0 software was used. Correction of TGA for phenomenon of buoyancy effect and friction force from vertically moving gas was done using TGA-BeFlat® procedure which is part of the mentioned software.

RESULTS AND DISCUSSION

First of all, the results of thermogravimetric analysis (TGA) are shown in Fig. 1, compile all samples for clear comparison of the data. The residual masses after the pyrolysis consist of biochar from the MDF fibres and the solid residues of the silicates. In order to estimate the fractions of the biochar and the silicate residues, all samples were placed in a furnace at 950°C for 2 hours in air to remove the carbonized residue (biochar) of the MDF fibres. Assuming a high heat stability of the silicates, we estimated the fraction of silicate residues and thus also the ratio between these two components after the pyrolysis.

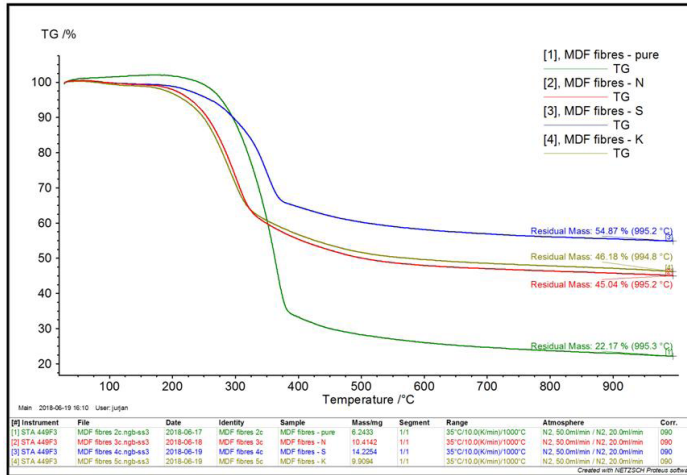


Fig. 1: Thermogravimetric analysis of the treated MDF fibres.

The results showed significant difference between treated and untreated MDF fibres as seen in Fig. 1. All the treatments (curves 2-3-4) change the pyrolysis behaviour relative to the pure MDF fibres (curve 1). Based on residual mass, modified silicate (S) achieved the best result surpassing residual mass of pure fibres by 32.7%. Sodium silicate (N) and potassium silicate (K) show a very similar progress (curves 2-4) with residual masses differing only by 1.14%. Behaviour of the sodium silicate (N) and potassium silicate (K) samples show an interesting progress during the first third of measurement interval (Fig. 1) as they seem to initiate rapid mass loss earlier (at lower temperature) than the other samples, even relative to pure fibres. The residual masses after the pyrolysis consisted of biochar from the MDF fibres and the solid residues of the silicates. In order to estimate the fraction of the biochar, all samples were exposed to 950°C in a furnace for 2 hours in air and weighed afterwards (Tab. 1).

Tab. 1: Composition of samples before and after pyrolysis.

MDF	Pure	+N	+S	+K
Fibres (mg)	6.2433	8.6909	9.2824	7.7049
Treatment (mg)	-	1.7233	4.943	2.2045
Biochar residue (mg) / (%)	1.3841 / 22.17	2.9673 / 34.14	2.8625 / 30.84	2.3717 / 30.78
Biochar (%) / Treatment (%)	-	63.26 / 36.74	36.67 / 63.33	51.83 / 48.17

Biochar residue mass values are given in mg, as shown in Tab. 1, and express the mass of the biochar in the sample after pyrolysis. The additional percentage value is its fraction in the pure fibres. The respective percentages of the biochar and the treatment are given in the bottom of Tab. 1 and are based on the mass of the STA pyrolysis measurement (Fig. 1). Comparing the biochar residue (Tab. 1) we can see that sodium silicate (N) achieved the best result from all the treatments with 34.14% residue of the former mass of fibres. Modified silicate (S) and potassium silicate (K) acquired almost identical values of 30.84% and 30.78%. The increase is evident in treatments compared to biochar residue of pure MDF fibres which acquired 22.17% residue of former mass before the pyrolysis.

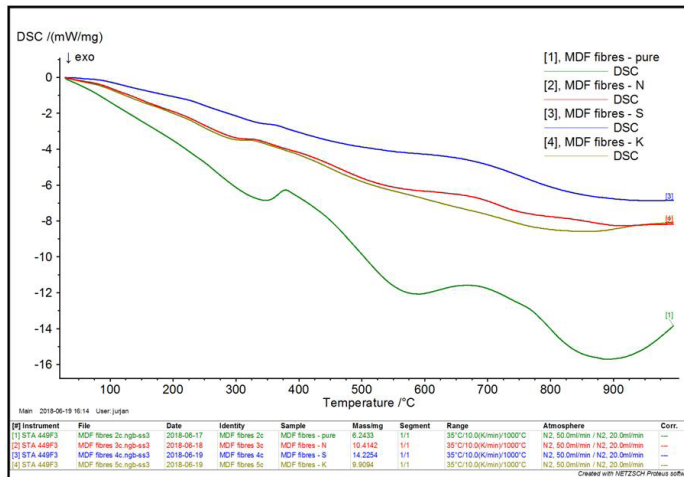


Fig. 2: Differential scanning calorimetry of the treated MDF fibres.

In addition to thermogravimetric analysis a differential scanning calorimetry (DSC) analysis was conducted and results are shown in Fig. 2. Note that the DSC results are mostly exothermic except for a signal of pure fibres where two significant parts between peaks 347.7-378.5°C and 590.9-668.7°C show the reaction is endothermic. It can be also seen at the last part of measurement on the same curve. Production of volatile by-products of the pyrolysis caused the sample to absorb energy which makes the reaction endothermic (Anca-Couce 2016). The applied treatments appear to reduce this production. Imperfection of the measuring device can influence the results as well which can be seen especially in the end of measurement on the green curve representing pure fibres. This phenomenon does not occur to such an extent in other signals where treatments moderate the progress of pyrolysis. The same samples that underwent the STA were subsequently placed in a furnace for 120 min at a constant temperature of 950°C with air atmosphere to remove the carbonized fibres (biochar) produced during the pyrolysis to find the share of biochar and treatment in sample after the STA. Microscopic images were made for the samples in 3 phases of the experiment and are shown in the Figs. 3-6. The first image was of the sample before the STA, the second image was the sample after the STA and the last one shows the sample after the furnace exposure.

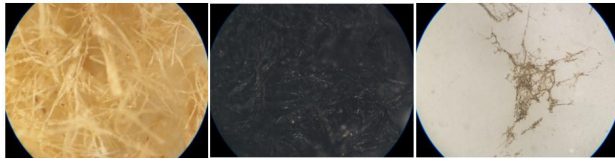


Fig. 3: Sodium silicate (N): a) original, b) after STA, c) after furnace.

As we can see in the third phase (Fig. 3), there are almost no remains of fibres after the furnace exposure, demonstrating that most of it combusted completely. The amount left is negligible.

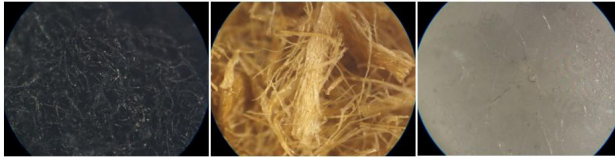


Fig. 4: Pure fibres: a) original, b) after STA, c) after furnace.

Compared to pure fibres a difference in the first phase is visible (Figs. 3-4) as fibres are more bound and a colour shade appears to be a bit darker likely caused by the pH value of the treatment. The treatment formed a glass layer as shown at last phase of Fig. 4. There are no visible remains of fibres in the last phase.

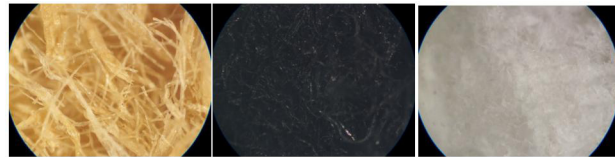


Fig. 5: Modified silicate (S): a) original, b) after STA, c) after furnace.

For the modified silicate (S) fibres in the first phase we can also see binding (Fig. 5), however the shade appears a bit brighter compared to sodium silicate (N) in Fig. 4. The third phase shows that the treatment attained a white crystalized form.

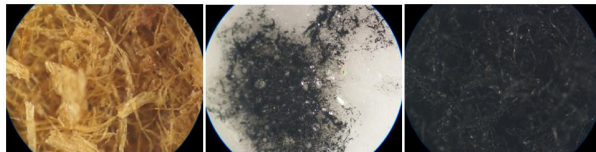


Fig. 6: Potassium silicate (K): a) original, b) after STA, c) after furnace.

At the phases of potassium silicate (K) (Fig. 6) we can see similarities with phases in Fig. 4, however in the third phase of Fig. 6 a minor remains are visibly melted into the glass formed by the treatment which we should take into account for it causes a small inaccuracy in the results. Time to ignition measurements are shown in Tab. 2. The results displayed represent average value as every measurement was done twice to achieve higher precision.

Tab. 2: Measured times of ignition of the specimens at different heat fluxes.

Specimen	Time of ignition (s)	Specimen	Time of ignition (s)	q_e'' (kW·m ⁻²)
Pure 1	137.5	N 1	104	20
Pure 2	60	N 2	55	25
Pure 3	51	N 3	38	30
Pure 4	24.5	N 4	23.5	40
Pure 5	14	N 5	11	50
S 1	122.5	K 1	103.5	20
S 2	68	K 2	59.5	25
S 3	40	K 3	35.5	30
S 4	20	K 4	20.5	40
S 5	10.5	K 5	13	50

From the raw data we can see that untreated specimens exhibited the longest time to ignition in general, while treatments seem to reduce it. Ignition delay time $1/t_{ig}^{1/2}$ (1/s^{1/2}) is calculated according to Eq. 1 for thermal thick behavior where $k\rho c$ is thermal inertia, q_e'' is incident heat flux, T_{ig} is temperature of ignition and T_0 is the ambient temperature which was measured to be 25 °C.

$$\frac{1}{\sqrt{t_{ig}}} = \frac{2}{\sqrt{\pi}} \sqrt{k\rho c} \frac{q_e''}{(T_{ig} - T_0)} \quad (1)$$

These results are displayed in Fig. 7 and for a more detailed insight also in Tab. 3 with all the source data included. Along with the ignition delay time the critical heat flux q_{0ig}'' is estimated. For the description of deviation of results a standard deviation and standard error are evaluated.

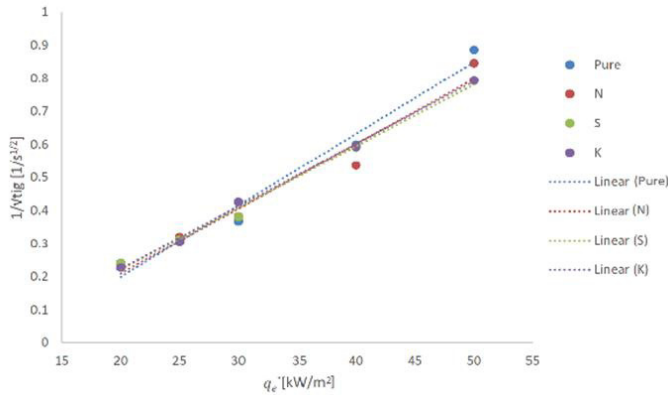
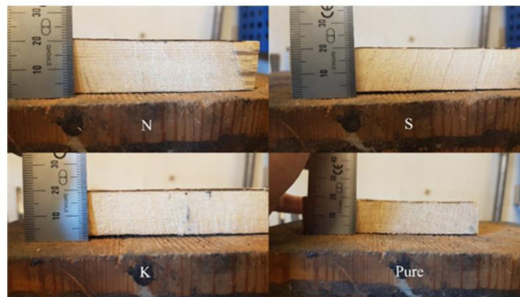


Fig. 7: Ignition delay time for different heat fluxes.

Tab. 3: Ignition delay time, critical heat flux and standard deviation of critical heat flux.

Specimen	$1/\sqrt{t_{ig}}$ (1/s ^{1/2})	Specimen	$1/\sqrt{t_{ig}}$ (1/s ^{1/2})	q_e'' (kW·m ⁻²)
Pure 1	0.241	N 1	0.236	20
Pure 2	0.309	N 2	0.320	25
Pure 3	0.368	N 3	0.382	30
Pure 4	0.600	N 4	0.537	40
Pure 5	0.886	N 5	0.846	50
Slope	0.022 ± 0.002	Slope	0.0196 ± 0.002	-
Intercept	-0.23 ± 0.07	Intercept	-0.18 ± 0.07	-
R²	0.977	R²	0.966	-
q_{0ig}'' (kW·m⁻²)	11 ± 3	q_{0ig}'' (kW·m⁻²)	9 ± 4	-
Specimen	$1/\sqrt{t_{ig}}$ (1/s ^{1/2})	Specimen	$1/\sqrt{t_{ig}}$ (1/s ^{1/2})	q_e'' (kW·m ⁻²)
S 1	0.240	K 1	0.228	20
S 2	0.313	K 2	0.305	25
S 3	0.382	K 3	0.427	30
S 4	0.591	K 4	0.594	40
S 5	0.792	K 5	0.794	50
Slope	0.0187 ± 0.0008	Slope	0.0189 ± 0.0005	-
Intercept	-0.15 ± 0.07	Intercept	-0.16 ± 0.02	-
R²	0.994	R²	0.998	-
q_{0ig}'' (kW·m⁻²)	8 ± 2	q_{0ig}'' (kW·m⁻²)	8 ± 1	-

The estimates of the critical heat flux q_{0ig}'' have shown that pure specimens achieve value of 11 ± 3 kW·m⁻² which roughly corresponds to critical heat flux of wood in general. Different types of wood achieve values around 12 kW·m⁻². A decrease to 9 ± 4 , 8 ± 2 and 8 ± 1 kW·m⁻² in treated specimens seems odd as it appears to be counter-intuitive yet it matches the trending data when compared. Results of all of the measurements appear to be very similar which can be clearly seen visualized in Fig. 7. A gap of 2 – 3 kW·m⁻² is minor, however should not be caused by error as standard deviations and standard errors of every type of treatment are almost identical, which means that no extreme values interfering data appeared or appeared equally in all measurements. Specimens were cut in half after the experiments conducted using cone calorimeter in order to investigate carbonized layer. Captures of specimens that were exposed to 50 kW·m⁻² heat flux are shown in Fig. 8.

Fig. 8: Carbonized layer of spruce specimens - 50 kW·m⁻² heat flux.

Approximately, a 0.5 mm thick carbonized layer occurred in all of the treated specimens. This, however, does not apply to the pure sample where carbonization occurred solely on the surface in significantly lesser extent. Furthermore, the pure specimen exhibited the longest time to ignition (Tab. 2a). It appears that small amount of these water glass flame retardants applied with thin impregnation layer accelerated the process of carbonization at high heat flux rates. This phenomenon did not occur in samples exposed to a lower heat flux of $20 \text{ kW}\cdot\text{m}^{-2}$, where the carbonized layer thickness occurred equally in all of the tested specimens. Increased carbonization in spruce specimens after usage of mono-ammonium phosphate flame retardant was also reported by (Hagen et al. 2009) as we as for many other flame retarding agents as reported by (Schaffer 1974) and (Nussbaum 1988). The surface temperature at ignition varied from 240 to 271°C in these specimens at $50 \text{ kW}\cdot\text{m}^{-2}$ heat flux, which is on the edge of onset temperatures range for pyrolysis of lignin (227 - 550°C) (Tan et al. 2006), cellulose (250 - 350°C) (Yeng et al. 2015) and hemicellulose (220 - 315°C) (Yang et al. 2007). It could also be that other organic compounds may help create the flammable mixture over the surface. So called extractives are common organic compounds found in wood, however their content in spruce wood reaches around 2% (Hillis 1971, Shebani et al. 2008). Their possible contribution to a flammable mixture creation in influential extent was therefore highly questionable and improbable. It is important to note that it was not a question of determining the course and development of the charred layer, but the final value after the end of the experiment. All measurements were in the same time interval.

CONCLUSIONS

The results of this experiment have shown the differences between tested treatments including no treatments. All of them have proved to have a significant effect on the pyrolysis of MDF fibres. According to biochar residue, sodium silicate (N) has shown the best results acquiring 34.14% of the former fibre mass of the sample. This is an interesting finding from the commercial point of view as it is the cheapest of the evaluated treatments and it improves also other properties of wood such as hardness, bending strength etc. (Ming-Li et al. 2019). Sample of MDF fibres treated with modified silicate (S) achieved the best results in thermogravimetric analysis (Fig. 1) with 54.87% of residual mass, however most of this residue (63.33%) consists of the treatment itself and biochar residue acquires the value of 30.84% of the former fibre mass (Tab. 1). Low pH value of modified silicate (S) enabled it to be retained in the MDF fibres most effectively compared to other treatments with 4.943 mg of treatment in 9.2824 mg of MDF fibres. Potassium silicate (K) acquired almost identical result of biochar residue like modified silicate (S) with value of 30.78%, however the share of treatment in the sample after pyrolysis was lower, achieving 48.17% which shows better efficiency at lower concentration compared to modified silicate (S). Both of these results are however outperformed by sodium silicate (N) having 36.74% share of treatment in the sample after pyrolysis and achieving best results in biochar residue as mentioned.

All three types of used water glass flame retardants show decrease in the onset temperature of pyrolysis which was also reported by (Hagen et al. 2009). The critical heat flux decrease from estimated $11 \text{ kW}\cdot\text{m}^{-2}$ to $9 \text{ kW}\cdot\text{m}^{-2}$ for sodium silicate (N) and $8 \text{ kW}\cdot\text{m}^{-2}$ for modified silicate (S) and potassium silicate (K) is most probably caused by application method where only thin layer of flame retardant in specimens was created. Authors that used vacuum pressure impregnation for wood materials report an increase in critical heat fluxes, see e.g. (Hagen et al. 2009). Cause of this decrease in fire resistant properties of flame retarded specimens might be that low amount of

thinly penetrated flame retardant might serve as empowering initializer of pyrolysis, as indicated before, yet failing to provide sufficient protection due to its inadequate impregnation. Another reason for the occurrence of this phenomenon might be that layer of flame retardant might obstruct the absorbed energy from cone to radiate partly back through the surface which would result in accelerated heating of the material on and under the surface accumulating heat, thus decreasing also the time to ignition. These are just hypotheses and further research is necessary in order to prove or disprove them.

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