

Wood facade materials ageing analysis by FTIR spectroscopy

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Attenuated total reflection Fourier transform infrared spectroscopy is used in this study to quantify wood degradation. Nine different types of wood materials were studied. All types of wood were exposed in an Atlas solar simulator against artificial climate conditions. Additionally, natural climate exposure was performed for all types of wood at an outdoor test site in Trondheim, Norway, for 4 years. Surface treatment enhanced the durability of the wood and their performance against weathering. Rough surfaces might reduce the resistance against degradation in comparison with planed surfaces. The degradation during climate exposure created loss of lignin and accumulation of cellulose at the surface.

Notation

T	transmittance
A'	absorbance
I	intensity

1. Introduction

Wood materials are often preferred in facades or as claddings on buildings owing to the light weight, easy workmanship and low thermal conductivity. However, most construction companies and users are aware of the performance-related failures that occur in wooden claddings. Determination of environmental impact, moisture problems, preservative efficiency, degradation, coating failure and predicting service life of woods are challenging research areas (Gobakken *et al.*, 2008; Gupta *et al.*, 2011b; Masters, 1985). Service life of a wood material exposed to natural climate is limited because of its degradation against solar ultraviolet (UV) radiation, especially against the UV-B component of the radiation (Lionetto *et al.*, 2012). Moisture-induced decay in wood and wooden cladding materials is a big problem (Brischke *et al.*, 2008; Brischke and Rolf-Kiel, 2010). Interstitial condensation and water penetration through wooden cladding materials is a major challenge for water tightness of the building envelope (Sahal and Lacasse, 2005; Sahal and Lacasse, 2008). Decay at indoor and outdoor conditions can drastically reduce service lives of residential buildings (Kalamees, 2002). Fungus mould growth on wooden components at high humidity condition is an added problem (Gobakken and Westin, 2008; Gupta *et al.*, 2011a). Furthermore, types of wooden joint affect the service life of the structure (Highley, 1995).

Wood is chemically complex and composed of cellulose (40–55%), hemicellulose (24–40%), lignin (18–35%) and extraneous materials or extractives (0.1–10%) (Lewin and Goldstein, 1991; Pettersen, 1984; Sjöström, 1993). Cellulose is a carbohydrate of linked 1, 4- β -D-glucoglycan units having degree of polymerization (DP) 5000–10 000. Lignin, on the other hand, has predominant β -O-4-linked aromatic units as building blocks having DP 100–1000. Hemicellulose is non-glucose pentose and hexose sugars having DP 150–200. The distribution of the wood components varies between species and depends on the amount of heartwood/sapwood content. For example, it has been reported that the lignin content of Norway spruce decreases in the radial direction from heartwood (28.3%) to sapwood (27.7%) (Bertaud and Holmbom, 2004; Raikila *et al.*, 2007).

Mechanical, electromagnetic, thermal, chemical and biological agents are the key degradation agents affecting the service life of building materials, components and structures (ISO 15686-8 2008; ISO, 2008). Analysing wood degradation processes in natural outdoor environment and in artificial laboratory conditions yields results that can be modelled for service life planning (SLP) of wood materials (Gupta *et al.*, 2010; Lisø *et al.*, 2006; Morris, 2005; O'Connor, 2004; Sjöström and Brandt, 1991). Enhancement of the service life of wood materials is possible by application of surface coatings (Hovde *et al.*, 2008).

Opting best design and clever construction practices would imply a durable building envelope with minimal facade maintenance. Wood modification and chemical treatments affect the performance of wood components in a complex way

(Bull, 2000; Kollmann, 1968). Since successful preservatives such as chromated copper arsenate (CCA), creosote, coal tar and chromium-based preservatives have been barred from use at places of human skin contact as specified by the American Wood Protection Association (AWPA), Health and Safety Executive (HSE) of United Kingdom and European standards (EN 351-1; CEN, 2007). Innovative methods like furfurylation have become an alternative for the wood preservative industries (Lande *et al.*, 2004; Lande *et al.*, 2008). Furfuryl alcohol is a bio-based product that is used to impregnate wood to impart toxicity against bio-organisms (Pilgard *et al.*, 2010).

Attenuated total reflection (ATR) Fourier transform infrared (FTIR) spectroscopy is a non-destructive technique that has been successfully employed in characterisation of polymer structures and of polymer and wood degradation (Feist and Hon, 1984; Himmelsbach *et al.*, 2006; Jelle *et al.*, 2008; Jelle and Nilsen, 2011; Jelle *et al.*, 2012; Jelle, 2012; Jelle, 2013; Korner *et al.*, 1992; Rodrigues *et al.*, 1998). Common FTIR bands for wood are provided in Table 1 (Faix, 1986; Faix, 1988; Feist and Hon, 1984; Himmelsbach *et al.*, 2006; Hinterstoisser and Salmen, 1999; Jelle *et al.*, 2008; Korner *et al.*, 1992; Lewin and Goldstein, 1991; Pandey and Pitman, 2003; Robotti *et al.*, 2007; Tolvaj and Faix, 1995).

The European Union Construction Product Regulation (305/2011), that replaces the existing Construction Products Directive (89/106/EEC, as amended by Directive 1993/68/EEC – the CPD) requires a declaration from the manufacturer for the performance of the construction products and carry the CE mark. Subsequently, performance analysis of wood as a construction material is getting increasing importance (Gupta *et al.*, 2011b; Steer, 2001).

The objective of this study was to evaluate the performance and the degradation processes occurring in nine types of wood materials at (a) natural outdoor exposure and (b) artificial laboratory exposure conditions, that may provide valuable input to service life estimation (ISO 15686-2 2001; ISO, 2001). SLP requires the description/design of exposure programmes, identification of possible degradation mechanisms and of possible effects of degradation (ISO 15686-1 2000; ISO, 2000). In addition, categorisation of degradation processes occurring in a treated wood compared to untreated wood would provide valuable information regarding the degradation processes in the same climate conditions. In this study aged materials were characterised by the ATR-FTIR spectroscopy as a fast characterising tool to evaluate degradation and durability of wood.

2. Experimental

2.1. Materials

Nine types of wood materials, namely, rough sawn Norway spruce (*Picea abies* [L] Karst.), planed surface Norway spruce

(*Picea abies* [L] Karst.), scots pine heartwood (*Pinus silvestris* L.), aspen (*Populus tremula* L.), larch (*Larix decidua* Mill.), Kebony Furu (*Pinus silvestris* L., Scots Pine, untreated heartwood and sapwood treated with furfuryl alcohol), Kebony SYP (*Pinus taeda*, Southern yellow Pine, sapwood treated with furfuryl alcohol), linseed oil-impregnated scots pine (*Pinus silvestris* L.) and pressure-treated scots pine (*Pinus silvestris* L.) were exposed to outdoor and artificial climate laboratory conditions. Details of the wood samples were reported previously (Rüther, 2011; Rüther and Jelle, 2013). The dried woods material specimens were stored in a desiccator at ambient temperature $\sim 22^{\circ}\text{C}$. The furfurylated woods were used as supplied by Kebony ASA, Norway. The furfurylation process includes impregnation of wood with a 22% solution of furfuryl alcohol in water (furfuryl alcohol : water = 30 : 100, acidic catalysts and process additives). The impregnated woods were then cured at 100°C (Gobakken and Westin, 2008).

2.2 Long-term outdoor exposure

Natural weathering of the cladding materials (50 cm \times 15 cm \times 2 cm specimens) was performed at an outdoor field test site in Trondheim, Norway ($63^{\circ}25'\text{N}$, $10^{\circ}26'\text{E}$). The specimens were vertically mounted on a wall facing south for up to 1322 days. The climate of Trondheim can be classified into 'D' as per the Köppen classification system having cold/snow climate and boreal forest type of vegetation (Kleidon *et al.*, 2000; Smith *et al.*, 2002). The annual mean precipitation is greater than the dryness threshold or the hydrological area that separates evaporated water from the climate regions. The test site is equipped with meteorological facilities from the Norwegian Meteorological Institute to collect data of daily precipitation, wind direction and solar radiation. The climate in Trondheim is affected by the North Atlantic Oscillations (NAO) accompanied by occasional changes in sea level pressure gradient (Werner *et al.*, 2000). Ageing was performed mostly on the tangential side of the specimens. The specimens were exposed to daily solar radiation, wind, snow, rain and other wind-driven particles. Outdoor exposure has a distinct advantage that the natural weather conditions invites attack by biological agents. Importantly, the local microclimate has a particular effect on the exposed specimens which is difficult to reproduce in laboratory conditions.

2.3 Short-term artificial exposure

Artificial ageing was performed by exposing the wood test-blocks (15 cm \times 15 cm specimens) at horizontal laying position in an ATLAS SC600 MHG solar simulator. At 100% lamp power intensity the solar radiation intensity was 1200 W/m^2 at a specified distance. Temperature and relative humidity (RH) were constant, namely, 63°C at 50% RH during the solar radiation exposure and 10°C at 100% RH during the water spray period. Deionised water was sprayed from two nozzles at a rate of $0.5 \text{ dm}^3/\text{min}$ per nozzle; creating an even, horizontal water mist

IR band	Cellulose (cm ⁻¹)	Lignin (cm ⁻¹)	Wood (cm ⁻¹)	Studied peak (cm ⁻¹)
Free OH, weakly absorbed water, O—H stretching of water bound by hydrogen bonds (Hinterstoisser <i>et al.</i> , 2001; Luna <i>et al.</i> , 2012; Olsson and Salmen, 2004; Popescu <i>et al.</i> , 2006)	3500–3100, 3600	3425–3400, 2940, 2920, 2880–2850	3568, 3561, 3499–3468, 3430–3423, 3419	3600–3000
Cellulose I β, 6—OH·O—3 intermolecular H-bond (Hinterstoisser <i>et al.</i> , 2001; Popescu <i>et al.</i> , 2007)	3270, 3310–3230	–	3278, 3277, 3271	
Asymmetric CH, CH ₂ stretching, OCH ₃ stretch (Hinterstoisser <i>et al.</i> , 2001; Luna <i>et al.</i> , 2012; Popescu <i>et al.</i> , 2006)	2980–2835	–	2937–2929, 2924–2906	
Symmetric CH and CH ₂ stretching (Hinterstoisser <i>et al.</i> , 2001; Popescu <i>et al.</i> , 2006)	2980–2835	–	2908–2904	
Scissoring and rocking vibrations of water (Olsson and Salmen, 2004)	2100	–	–	
Non-conjugated carbonyl groups associated with pectin (Luna <i>et al.</i> , 2012)	–	–	1738–1726	1734
H—O—H angle vibration of adsorbed water (Olsson and Salmen, 2004)	1650	–	–	1650
C=O stretching (Popescu <i>et al.</i> , 2006)	1660–1610	1330–1325, 1275–1270	1749–1740, 1665–1655	
Aromatic skeletal vibrations (Luna <i>et al.</i> , 2012; Popescu <i>et al.</i> , 2006)	–	1605, 1595, 1505, 1515–1510, 1430, 1425	1515–1505	1510, 1505
CH ₂ scissoring (Hinterstoisser <i>et al.</i> , 2001; Popescu <i>et al.</i> , 2006)	1480–1400	–	1463	
C—C—H and C—O—H bending (Marechal and Chanzy, 2000)	1500–1300	–	–	
Alkane and CH deformation of polymers (Luna <i>et al.</i> , 2012)	–	–	1426	
Holocellulose (Li <i>et al.</i> , 2010)	–	–	1736, 1372	
CH deformation of acetyl groups (Luna <i>et al.</i> , 2012)	–	–	1376–1372	
Asymmetric C—H deformation (Tolvaj and Faix, 1995)	–	1470–1460	1465–1457, 1428–1426	1465, 1423
Symmetric C—H deformation (Luna <i>et al.</i> , 2012; Tolvaj and Faix, 1995)	–	1370–1365	1380–1372, 1376–1372	1360, 1375
Ring breathing (guaiacyl 'G') (Popescu <i>et al.</i> , 2006)	–	1275, 1270, 1230	1270	1270
Ring breathing (syringyl 'S') (Luna <i>et al.</i> , 2012; Popescu <i>et al.</i> , 2006)	–	1330–1325, 1235–1230	1321, 1317, 1323–1320	1310
C—O—C asymmetric stretching (Hinterstoisser <i>et al.</i> , 2001; Popescu <i>et al.</i> , 2006)	1165	–	1174–1167	
Symmetric C—O—C stretching, pectin (Hori and Sugiyama, 2003)	1158	–	1160, 1158, 1137–1132	1158
Aromatic C—H in-plane deformation (Popescu <i>et al.</i> , 2006)	–	1145, 1140, 1130, 1035–1030	1030, 1032, 1033, 1034	1030
C—O, C—C, —C—H stretching of alcohol, pyranoside rings from glucose, cellulose and acetylglucomannan (Hori and Sugiyama, 2003; Luna <i>et al.</i> , 2012; Pandey and Pitman, 2003)	1030	1085	1023, 1030, 1132, 1134, 1136, 1137, 1049–1043	1024, 1030
C—C stretching	1030–1000	–	–	
=CH out of plane deformation	–	970	–	

Table 1. FTIR bands assigned to cellulose^a, lignin^a, wood^a and the IR bands of particular interest (continued on next page)

IR band	Cellulose (cm ⁻¹)	Lignin (cm ⁻¹)	Wood (cm ⁻¹)	Studied peak (cm ⁻¹)
C–H deformation (Popescu <i>et al.</i> , 2006; Tolvaj and Faix, 1995)	897	–	896, 900–898	898
Aromatic CH out-of-plane deformation (Popescu <i>et al.</i> , 2006)	–	915, 860–855, 815, 770–750	839, 838, 835	
Epoxy groups (Luna <i>et al.</i> , 2012)	–	–	898–893	
Meta-di-substituted benzene groups (Luna <i>et al.</i> , 2012)	–	–	779–774	
=CH ₂ rocking vibration (Popescu <i>et al.</i> , 2006)	715	–	716–713	
–OH out-of-plane vibration or rotational vibration of water (Olsson and Salmen, 2004)	700	–	–	

^aOther literature sources are cited in the introduction and discussion sections.

Table 1. Continued

distribution in the exposure chamber. A steel perforated mesh was used as a solar radiation filter to reduce the oxidative solar radiation stress on the specimens by 50% of original radiation. Ageing was performed for 42 days where each 6 h ageing cycle was divided into: 5 h (63°C) solar radiation exposure and 1 h (10°C) water spray. Irradiance was emitted from a 2.5 kW metal halide global lamp at an intensity of 600 W/m² at the sample surface (using the perforated mesh). Ageing was performed on the tangential side of the wood. Each wood specimen was marginally tilted from horizontal position to allow the flow of water.

2.4 Attenuated total reflectance spectroscopy

A Thermo Electron Nicolet 8700 Fourier transform infrared spectrometer with a Smart Orbit accessory was used to obtain ATR-FTIR spectra of the wood specimens that were conditioned at room temperature 22°C and room humidity. Adequate touch of the specimen surface with the diamond crystal was obtained by pressing a rotating knob fitted with the accessory against the specimen surface. The mid-IR regions of 4000–400 cm⁻¹ were evaluated to characterise the specimens. OMNIC software was used to average 32 scans (fixed incidence angle of 45°) for a single specimen spot. In total, a minimum of three spectra were collected for each specimen. No further spectral correction was performed. Absorbance is specified by Beer-Lambert's law

$$1. \quad A' = \text{absorbance} = \log_{10}(1/T)$$

where T is the transmittance. A carbonyl index (C.I.) is calculated by normalising the peak intensities of the C=O group at 1718 cm⁻¹ ($I_{1718 \text{ cm}^{-1}}$) with the C–H deformation and CH₃ group at 1375 cm⁻¹ ($I_{1375 \text{ cm}^{-1}}$) according to previous research

work (Andrady *et al.*, 1993; Lionetto *et al.*, 2012; Pandey and Pitman, 2003):

$$2. \quad \text{C.I.} = \left[\frac{I_{1718 \text{ cm}^{-1}}}{I_{1375 \text{ cm}^{-1}}} \right]$$

Similarly, a lignin index (L.I.) is calculated by normalising the peak intensities of the lignin group at 1505 cm⁻¹ ($I_{1505 \text{ cm}^{-1}}$) with the C–H/CH₃ groups at 1375 cm⁻¹ ($I_{1375 \text{ cm}^{-1}}$):

$$3. \quad \text{L.I.} = \left[\frac{I_{1505 \text{ cm}^{-1}}}{I_{1375 \text{ cm}^{-1}}} \right]$$

The hypothesis for conducting this test of measuring and calculating normalised indices was to calculate the variation of the surface cellulose and lignin amount related to changes in the wood material during the ageing.

2.5 Statistics

The absence or presence of a relationship between the two random variables, namely, cellulose and lignin, were measured by correlation. The correlation coefficient, ρ_{XY} of two random variables, X and Y , is defined by:

$$4. \quad \rho_{XY} = \frac{\text{Cov}(X, Y)}{\sigma_X \sigma_Y}$$

where $\text{Cov}(X, Y)$ is the covariance, σ_X^2 is the square of variance for variable X and σ_Y^2 is the square of variance for variable Y (Casella and Berger, 2002). If large values of X tend to appear with the observation of large values of Y , then $\text{Cov}(X, Y)$ will be positive. Contrarily, if large values of X tend to be observed

with small values of Y , then $\text{Cov}(X,Y)$ will be negative. Consequently, a value of ρ_{XY} of 1.0 or -1.0 will indicate a perfect linear correlation. Spearman's rank correlation coefficient is used in this study ($\alpha = 0.05$). It has been hypothesised that the arithmetic mean of two sample types is the same if the p -value is greater than 0.05. Goodness of fit for a plot is provided by the coefficient of determination R^2 , which measures the total variation that is explained by the fitted line.

3. Results and discussion

Inspections of the wood samples were performed twice a year, once in October just before the start of the snow season and once in June during the summer season. The idea was to get maximum information from seasonal changes. Aged and non-aged specimens were inspected for visual grading. It was found that the ageing both at natural outdoor and artificial laboratory exposure conditions imparts discoloration due to stain and/or due to chemical degradation. Furthermore, outdoor aged specimens showed cupping, knot failure and cracking.

3.1 FTIR spectroscopy

Figure 1 shows the FTIR spectra of the non-aged, fresh wood samples and those aged at natural outdoor conditions for 1322 days. For all samples, there is a broad band at 3600–3000 cm^{-1} that can be assigned to the loosely associated water or moisture absorption. The bands for asymmetric and symmetric stretching of methyl and methylene groups from organic extractives have been assigned to the peaks at 2920 and 2850 cm^{-1} (Poletto *et al.*, 2012). The fingerprint region of 1800–600 cm^{-1} , including the absorbed O–H vibration, C–H deformation in lignin and carbohydrates, C–H deformation in cellulose and

hemicellulose, C–O stretch in lignin, C–O–C vibration in cellulose and hemicellulose and C–O stretch in cellulose and hemicellulose, were of particular interest. Previously, researchers (Li *et al.*, 2010) observed gradual decrease of the intensity of absorbance bands at 1736 and 1372 cm^{-1} assigned to hemicellulose, and an increase in the intensities of bands at 1510 and 1225 cm^{-1} assigned to lignin, with ageing. Various absorbance peaks in this study are given in Table 1, where a comparison to literature values is also performed.

3.1.1 Results from natural exposure

Figure 1(b) shows the absorbance spectra of the samples aged at natural outdoor conditions for 1322 days. Lignin peaks at 1720, 1505, 1423 and 1310 cm^{-1} are distinctive. The broadened peak at 1640 cm^{-1} is assigned to bound water. The spectra demonstrate that there is a difference between the pattern of the nine wood types. Furthermore, there is a difference in spectra between plane surface spruce and rough surface spruce. The cellulosic O–H absorbance peak intensity at 1024 cm^{-1} from non-aged conditions changed to the following values after 1322 days of natural ageing exposure: pressure treated scots pine (0.16 to 0.44), larch (0.23 to 0.32), scots pine (0.24 to 0.29), aspen (0.28 to 0.26), plane surface spruce (0.19 to 0.24), Kebony SYP (0.11 to 0.20), rough surface spruce (0.16 to 0.17), linseed oil-treated scots pine (0.21 to 0.14) and Kebony Furu (0.37 to 0.06). This shows that the exposure effect on treated wood is higher.

The double peak at 3000–2800 cm^{-1} has been assigned for C–H stretching vibrations from impregnated wood by previous researchers (Jelle *et al.*, 2008). Consequently, this IR region was monitored. The splitting of the band was observed

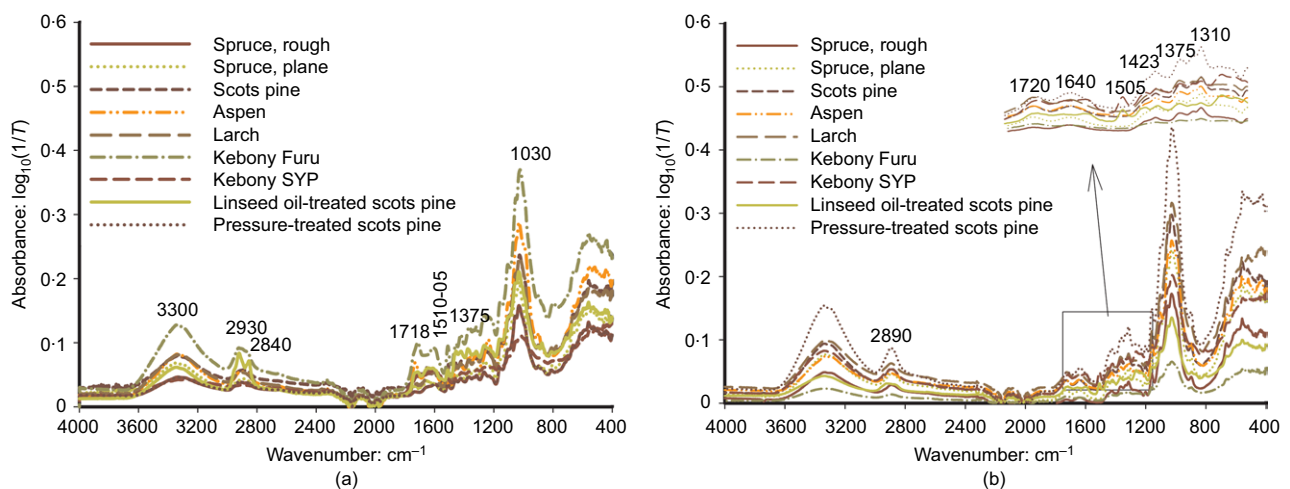


Figure 1. FTIR spectra showing: (a) non-aged, fresh wood before exposure; (b) natural outdoor ageing of wood after 1322 days

for linseed oil treated wood in Figure 1(a). It was observed that the intensity for C–H stretching vibration from the linseed oil impregnation which is assigned at $\sim 2890\text{ cm}^{-1}$, was highest for pressure treated scots pine in Figure 1(b). Untreated larch showed second highest absorbance at 2890 cm^{-1} , followed by scots pine, Kebony SYP, plane surface spruce, aspen, linseed oil-treated scots pine, rough surface spruce and minimum for Kebony Furu. Wood resin, poly(oxyethylene) types associated with C–O–C stretching vibrations, shows intense bands at 2860 and 1100 cm^{-1} owing to symmetrical stretching of CH_2 and asymmetrical C–O–C stretching (Jensen *et al.*, 1992).

The differences in the amount of absorbance between wood types, namely, the rough surface/plane surface and treated/untreated surfaces were distinguishable. Pressure-treated scots pine showed the highest intensities at the $3000\text{--}3600\text{ cm}^{-1}$ region which is assigned for the loosely associated water, at the lignin fingerprint region at 1505 cm^{-1} and for the cellulose at 1024 cm^{-1} . Furthermore, plane surface spruce showed higher intensities throughout the observed spectral region compared to rough surface spruce. This may be because the planed surfaces obtained better surface contact with the ATR crystals, that is, not necessarily due to the material itself.

Photodegradation is believed to be the primary form of degradation during outdoor natural exposure. Apart from the photo- and thermal degradations, there could be deterioration in lignocellulosic components at the surface, by different fungal species through enzymatic hydrolysis.

3.1.2 Results from artificial exposure

The results from artificial laboratory exposures of the nine wood types are shown in Figures 2 to 10. Artificial exposures of wooden specimens were performed to accelerate the failure

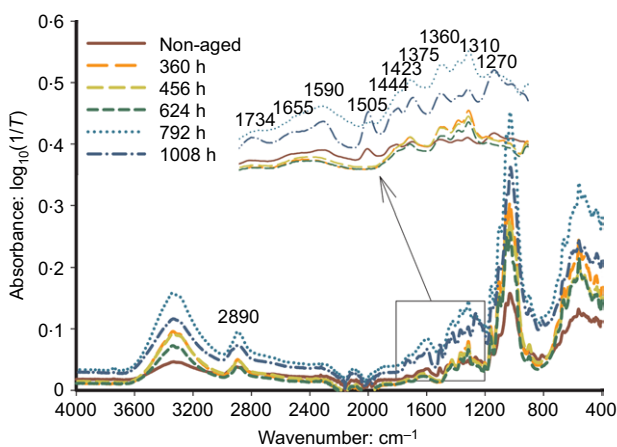


Figure 2. FTIR spectra showing artificial ageing of rough surface spruce

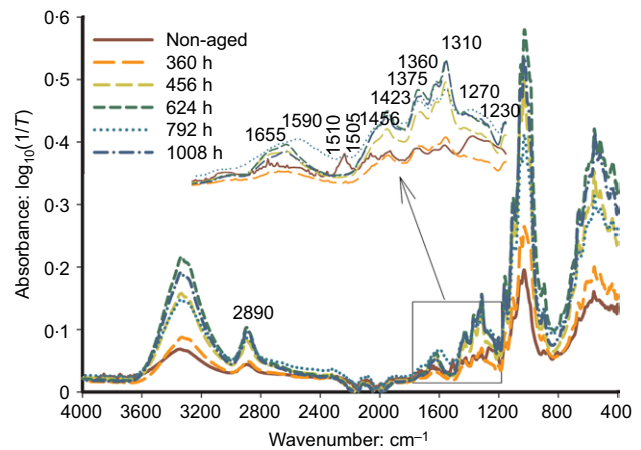


Figure 3. FTIR spectra showing artificial ageing of plane surface spruce

and determine the amount of degradation at controlled conditions.

Untreated wood materials. Figure 2 shows the absorbance spectra of rough surface Norway spruce. Limited irregularities were observed probably because of the roughness of the surface that possibly had created more chemical heterogeneity in the surface. However, rough surfaces may have had less contact with ATR crystal, depending on how soft the wood is and if there will be air cavities when pressed towards the ATR crystal.

The absorbance spectra of plane surface spruce are shown in Figure 3. For non-aged wood, there is a sharp peak at

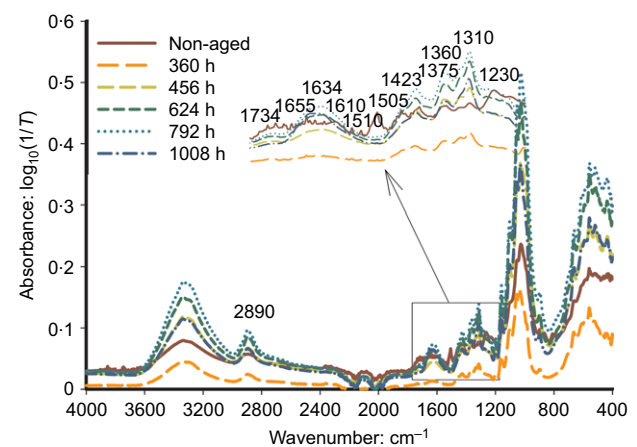


Figure 4. FTIR spectra showing artificial ageing of scots pine heartwood

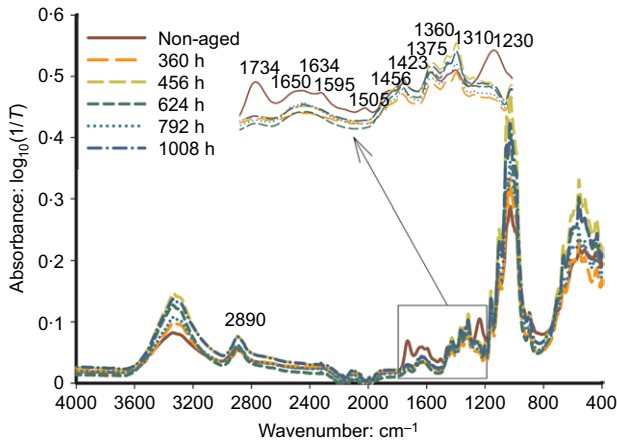


Figure 5. FTIR spectra showing artificial ageing of aspen

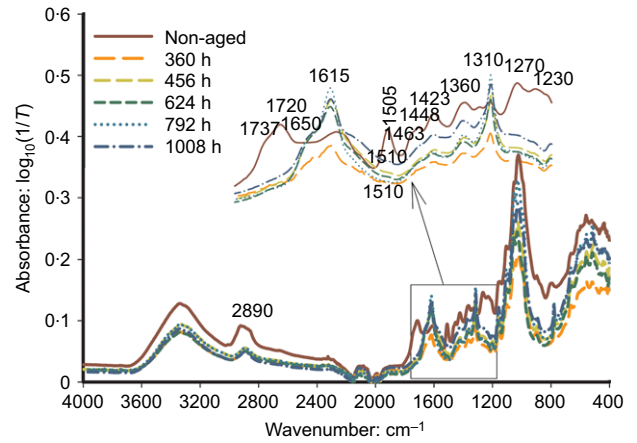


Figure 7. FTIR spectra showing artificial ageing of Kebony Furu

1505 cm^{-1} and a small shoulder at 1510 cm^{-1} showing the presence of lignin. With ageing, this lignin peak disappears first and reappears again at 792 h of ageing. Additionally, the non-aged wood shows a broad peak at 1270 cm^{-1} which can be assigned to G-lignin from coniferyl alcohol (Table 1). However, at higher levels of ageing, namely 1008 and 792 h, the broadness of this peak disappears. Instead, a sharper peak closer to 1275 cm^{-1} forms, indicating the accumulation of the C=O groups from lignin in a greater amount at the wood surface (Table 1). At micro-level, surface irregularities or roughness may have possibly induced more heterogeneity in chemical composition during ageing exposure as streaming water flow on a rough surface would not have been the same as on a machined, polished and plane surface. Plane surface spruce showed much more regularity in FTIR spectra.

Absorbance intensities were higher than the intensities observed for the rough surface spruce.

Figure 4 shows the absorbance spectra of scots pine heartwood. In general, several IR absorbance intensities increased with increasing ageing period. No new peaks resulted from ageing. Figure 5 shows the spectra of aspen. The cellulosic peak at 1024 cm^{-1} is sharp, similar to that found in the scots pine and spruce spectra. However, a broad band around 3300 cm^{-1} , assigned for water absorption, shows a difference between ageing periods which may be because of a variable amount of moisture absorption. Figure 6 shows the absorbance spectra of larch. All spectra of larch follow the same IR intensities in terms of absorbance; there are only minor differences in ageing for different exposure durations.

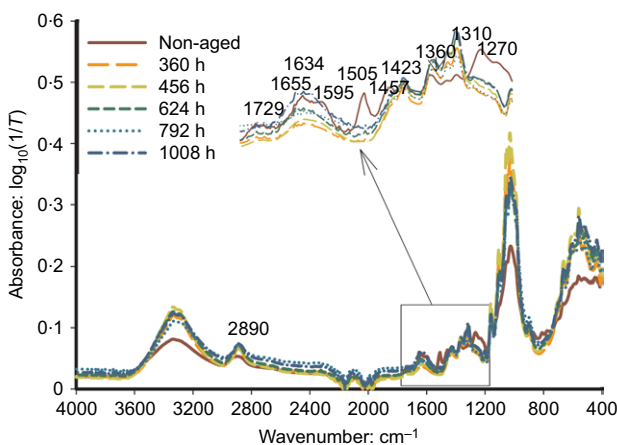


Figure 6. FTIR spectra showing artificial ageing of larch

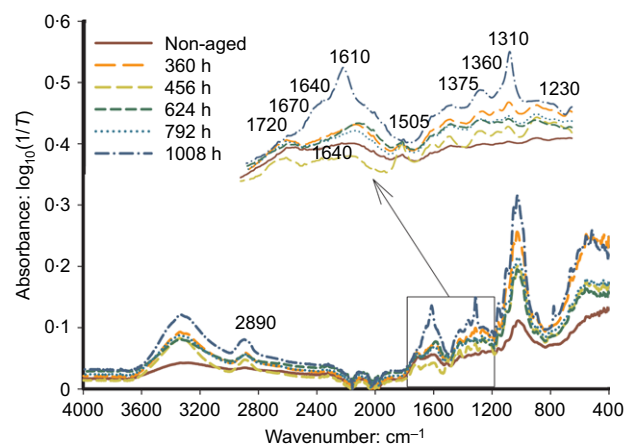


Figure 8. FTIR spectra showing artificial ageing of Kebony SYP wood

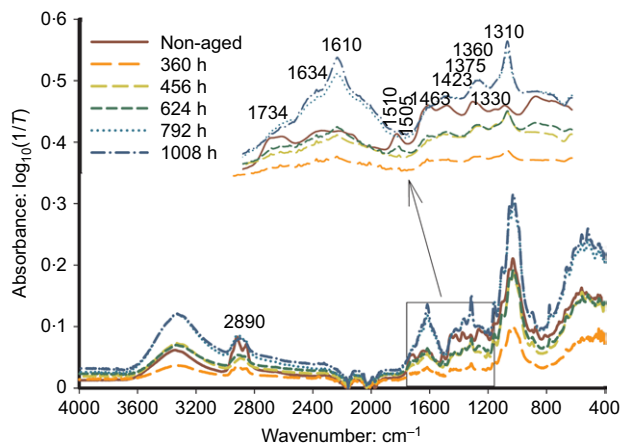


Figure 9. FTIR spectra showing artificial ageing of linseed oil-treated scots pine

No particular trend was observed in the survey spectra of untreated spruce, scots pine, aspen and larch woods. In general, maximum intensities for water absorption and cellulose peaks were observed at 792 h exposure for rough surface spruce, 624 h exposure for plane surface spruce, 792 h of exposure for scots pine, 456 h exposure for aspen and none for larch. Guaiacyl lignin (G-lignin) yields strong absorbance at 1510, 1465 and 1270 cm^{-1} band regions due to aromatic $-\text{O}$ and aromatic $-\text{OCH}_3$ stretch. In most spectra, the lignin band at 1510 cm^{-1} disappeared at longer exposure periods suggesting fast removal of G-lignin from substrate due to degradation. Cellulose shows a stronger absorbance near the 1024 cm^{-1} band. The intensity of the lignin band at 1505 cm^{-1} varied during exposure periods. The conjugated carbonyl group

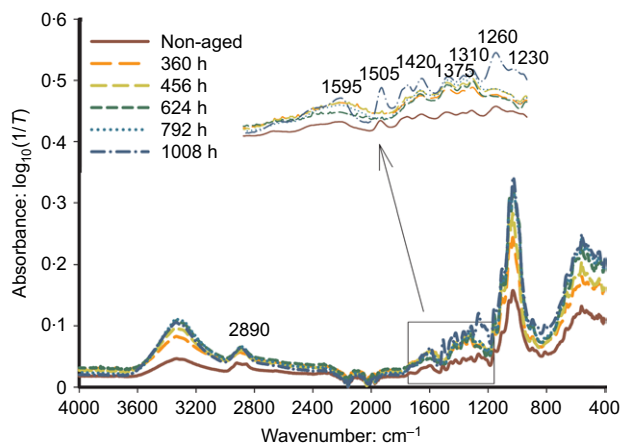


Figure 10. FTIR spectra showing artificial ageing of pressure-treated scots pine

associated with lignin shows a strong absorbance near the 1640 cm^{-1} band. However, the broadness of the band near 1640 cm^{-1} suggests that it could be from the residual bound water from the cell walls. Spruce with rough and plane surfaces showed prominent absorbance at the 1590 cm^{-1} band which represents characteristic absorbance by Klason lignin (Raiskila *et al.*, 2007). The strong absorbance near 1734 cm^{-1} is possibly due to the $\text{C}=\text{O}$ absorbance band from hemicelluloses associated with lignin.

Treated wood materials. In contrast to the untreated woods, the treated woods demonstrated a large difference in the absorbance spectra when observed specifically at the fingerprint regions.

Kebony Furu, shown in Figure 7 at longer durations of ageing, demonstrates a shoulder at 1650 cm^{-1} assigned to the adsorbed $\text{O}-\text{H}$ group (Olsson and Salmen, 2004). This indicates a greater amount of bound water present in the sample. Previous researches concluded that the peak at 1650 cm^{-1} disappears at a higher level of ageing of untreated wood (Anderson *et al.*, 1991). The shoulder at 1463 cm^{-1} , assigned to CH_2 scissoring, is present in non-aged wood and absent in aged woods. Lignin peaks at 1720, 1615, 1510, 1423, 1360, 1310 and 1270 cm^{-1} were observed for all levels of ageing. New peaks appeared at 780, 660, 590, 517 and 490 cm^{-1} at longer ageing time.

For Kebony SYP wood (Figure 8), the peaks at 1610 and 1310 cm^{-1} are prominent only after 1008 h of ageing. This peculiarity separated the spectra from Kebony Furu. Less prominent was the absorbance near the 1640 cm^{-1} band. Instead, lignin shoulders were prominent at 1720 and 1670 cm^{-1} at longer ageing time. Previously, in an artificial ageing of wood study, it was found to be difficult to locate the consistency in absorbance at 1640 cm^{-1} during ageing (Anderson *et al.*, 1991). The peak at 1375 cm^{-1} was prominent at all exposures of ageing.

Linseed oil-treated scots pine showed an increase in absorbance intensities for the fingerprint region (Figure 9). The double peak in the 3000–2800 cm^{-1} region for non-aged wood disappeared with ageing. A similar result was observed in spectroscopic evaluation for treated woods degradation by previous researchers (Jelle *et al.*, 2008). The $\text{C}=\text{O}$ absorbance from hemicelluloses was observed at 1734 cm^{-1} . Lignin peaks at 1634, 1610, 1510 cm^{-1} (not marked in figure), 1505, 1463, 1423, 1375, 1360 and 1310 cm^{-1} were prominent. In particular, the peak at 1610 cm^{-1} becomes sharper and distinguished after 792 h of ageing. The shoulder at 1330 cm^{-1} assigned to ring breathing was present in non-aged wood that fades up after 624 h of ageing. This peak has probably resulted from the linseed oil. Maximum absorbance intensity was obtained for

specimens aged up to 1008 h. New peaks appeared at 780, 660, 590, 558, 518, 490, 480 and 440 cm^{-1} . The high level of absorbance from lignin peaks is expected as linseed oil itself contains an average of 70% lignin materials.

In general, for pressure-treated scots pine, ageing increased the absorbance intensities (Figure 10). The double peak at the 3000–2800 cm^{-1} region for non-aged wood disappeared with ageing. Apart from the cellulose peak at 1024 cm^{-1} , lignin peaks were visible at 1505, 1420, 1375, 1310 and 1260 cm^{-1} . The peak surrounding 1375 cm^{-1} assigned to C–H deformation becomes more distinct in aged wood. New peaks appeared at 660, 558 and 519 cm^{-1} . Prominent peaks in the fingerprint region of pressure-treated scots pine were near 850, 746, 731, 550 and 490 cm^{-1} for chromates, sulfates, Cr-OH-Cr, $\text{CH}_2(\text{OH})$ and aryl-H, respectively.

In conclusion it may be said that generally the treated wood showed spectra of sharp absorbance intensities at various parts of the fingerprint region that are typical for lignin, cellulose and inorganic salts.

3.2 Spectral analysis and degradation of nine types of studied wood

Table 2 shows the carbonyl (C=O, H–C=O) index (C.I.) as calculated for the substrate surface from FTIR spectroscopy. There is a decreasing trend in C.I. with increasing degradation period. However, a linear regression model demonstrated that the fit was not significant ($R^2 = 0.16$). The number of oxygen atoms in the empirical molecular formula of lignin can be 3 to 9 (O/C = 0.33) whereas for cellulose the number of oxygen

atoms is a constant 5 (O/C = 0.83) (Li and Reeve, 2004). It is known that bleaching or removal of lignin would indicate O/C ratio shift towards 0.8 (Li and Reeve, 2004). So, neglecting the minor amounts of extractive contents, it may be said that the decrease in C.I. has possibly occurred due to the removal of amorphous content and lignin. This is because in cellulose, O atoms are bound in C–O–C or C–O–H form (Li and Reeve, 2004). A rigid conclusion cannot be drawn since researchers have found that lignin may be present on the wood surface in patches within the detectable limits of the characterising instruments. Kebony SYP remained to be on the higher side, compared to other woods, of the trend line obtained in the linear regression model ($R^2 = 0.16$). Overall, it might be concluded that unlike pure polymers, which show continuous increase in C.I. with the progress of degradation (Andrady *et al.*, 1993), wood degradation process is complex. Moreover, apart from variation of individual amounts of cellulose and C.I., other effects such as the distribution of the lignin and cellulose at the exposure surface and the influence of wood treatment chemicals act simultaneously. The surface roughness and colour change is random for artificial exposures as has been observed by others (Nzokou *et al.*, 2011).

The L.I., namely the surface concentration of lignin during artificial ageing exposure is shown in Table 2. The peak at 1505 cm^{-1} was found to be the best calibration fit ($R^2 = 0.98$) for a regression model in a previous study (Rodrigues *et al.*, 1998). First-order linear regression, when plotted for nine different types of wood, showed a decreasing trend ($R^2 = 0.10$) as the ageing period increased. The decreasing

Time of exposure: h	Rough spruce	Plane spruce	Pine	Aspen	Larch	Kebony Furu	Kebony SYP	Linseed oil	Pressure
Carbonyl index (I_{1718}/I_{1375})									
0	0.50	0.59	0.73	0.81	0.50	0.83	0.94	0.60	0.50
360	0.29	0.42	0.32	0.45	0.38	0.55	0.69	0.74	0.55
456	0.33	0.31	0.41	0.36	0.32	0.44	0.62	0.61	0.50
624	0.33	0.25	0.40	0.28	0.40	0.41	0.70	0.69	0.49
792	0.46	0.40	0.38	0.37	0.53	0.37	0.74	0.61	0.41
1008	0.51	0.26	0.38	0.37	0.46	0.42	0.57	0.58	0.41
Lignin index (I_{1505}/I_{1375})									
0	0.77	0.82	0.92	0.51	0.79	0.76	0.82	0.61	0.77
360	0.26	0.37	0.29	0.37	0.36	0.57	0.66	0.65	0.65
456	0.30	0.29	0.37	0.30	0.31	0.58	0.84	0.56	0.60
624	0.31	0.26	0.36	0.23	0.41	0.54	0.73	0.69	0.53
792	0.54	0.48	0.34	0.32	0.50	0.44	0.72	0.50	0.49
1008	0.82	0.27	0.39	0.32	0.46	0.59	0.56	0.55	0.89

Table 2. Carbonyl index (C.I.) and lignin index (L.I.) for treated and untreated wood species

amount of lignin confirms the appearance of an increased amount of cellulose at the surface. A similar observation of removal of non-crystalline entities due to natural and accelerated exposures of Norway spruce has been reported in another research where the band ratio has been evaluated in the opposite direction, that is, I_{1375}/I_{1512} (Raiskila *et al.*, 2007). However, an increase in L.I. at longer periods of exposure was observed in this study. When the L.I.s of all woods were plotted on the same graph, the L.I. of Kebony SYP was found to be the outlier in the linear regression model ($R^2 = 0.10$) after 400 h of ageing. Although, $R^2 = 0.10$ is a poor fit; however, it is possible that this has happened because of the effect of furfurylation,

3.3 Implications for durability evaluation

The onset of degradation and decay would limit the performance and reduce service life of a component. Progress of decay, however, would definitely reduce the performance and service life. Timber durability is, therefore, a key issue. To evaluate durability, five peaks, namely, 1505, 1423, 1158, 1023 and 898 cm^{-1} were selected to verify the nature of the collected spectra. Table 1 shows the chemical bonds that have been assigned for the above wavenumbers. Table 3 shows the Spearman's rank correlation coefficients. Interestingly and predictably, the cellulosic peak pairs of 1423, 1158, 1023 and 898 cm^{-1} correlate well ($\rho_{XY} > 0.6$, $p < 0.05$). The peak at 1505 cm^{-1} assigned to lignin was not in good fit with the peaks assigned purely for cellulose. Therefore, it can be said that larger values in intensities of one peak of cellulose is associated with larger values of cellulose peaks at other wavenumbers. As explained before, there is no statistically significant correlation between the cellulosic peaks and the lignin peaks ($p > 0.05$). Therefore, from a physicochemical point of view, it may be said that surfaces with high cellulose content may not have high lignin content.

4. Conclusion

Degradation of different species of wood with and without preservatives has been tested by natural outdoor climate exposure as well as artificial accelerated climate laboratory exposure. The materials were characterised by ATR-FTIR spectroscopy. The following results were obtained:

1. C.I. had a decreasing trend, not statistically significant, with increasing degradation period.
2. Treated woods demonstrated better performance than untreated woods as observed from the L.I. study.
3. Statistically, there was no significant correlation between the cellulosic peaks and the lignin peaks at 1505 cm^{-1} .
4. L.I. and C.I. indices showed that after degradation, treated woods had less changes in chemical composition compared to the untreated woods.
5. Rough surface spruce had small spectral irregularities during the exposure period compared with the plane surface spruce, as evaluated in the FTIR study.
6. Preservative treatment and impregnation of chemicals to wood certainly enhanced the service life at accelerated ageing conditions as was also observed at the natural outdoor weathering conditions.

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Variables	Cellulose (1023 cm^{-1})	Cellulose (1158 cm^{-1})	Cellulose, lignin (1423 cm^{-1})	Lignin (1505 cm^{-1})
Cellulose (898 cm^{-1})	0.800 (0.006)	0.933 (0.000)	0.717 (0.024)	0.483 (0.169)
Cellulose (1023 cm^{-1})	–	0.750 (0.016)	0.683 (0.036)	0.267 (0.462)
Cellulose (1158 cm^{-1})	–	–	0.750 (0.016)	0.567 (0.099)
Cellulose, lignin (1423 cm^{-1})	–	–	–	0.750 (0.016)

^aNumber of samples: 9.

The pair(s) of variables tends to increase together, if they have positive correlation coefficients and p -values below 0.050. For pairs with p -values greater than 0.050, there is no significant relationship between the two variables.

Table 3. Spearman rank order correlation showing correlation coefficient and p -value (in parentheses) for cellulose and lignin absorbance peaks^a

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