

Accelerated Climate Ageing of Building Materials and their Characterization by Fourier Transform Infrared Radiation Analysis

Bjørn Petter Jelle^{ab*}, Tom-Nils Nilsen^c, Per Jostein Hovde^b and Arild Gustavsen^d

^a SINTEF Building and Infrastructure, Department of Materials and Structures,
NO-7465 Trondheim, Norway.

^b Norwegian University of Science and Technology, Department of Civil and Transport Engineering,
NO-7491 Trondheim, Norway.

^c Norwegian University of Science and Technology, Department of Chemical Engineering,
NO-7491 Trondheim, Norway.

^d Norwegian University of Science and Technology, Department of Architectural Design, History and Technology,
NO-7491 Trondheim, Norway.

*Corresponding author: bjorn.petter.jelle@sintef.no (e-mail), 47-73-593377 (phone), 47-73-593380 (fax).

Abstract

Building materials and their durability and ageing are essential to the performance of buildings and their building physical aspects over time. Accelerated climate ageing investigations are carried out in order to study the durability of various building materials in a substantial shorter time span than natural weather ageing would have allowed. Climate parameters like temperature including freezing/thawing cycles, relative air humidity, water spray amount, solar and/or ultraviolet radiation and exposure duration are controlled in different climate ageing apparatuses. Various ageing processes in the building materials, ageing both by natural and accelerated climate exposure, may be studied in an attenuated total reflectance (ATR) Fourier transform infrared (FTIR) radiation analysis following the decomposition and/or formation of chemical bonds in these building materials and products.

The ATR-FTIR experimental method represents a powerful measurement tool on various materials. This method may be applied on both solid state materials, liquids and gases with none or only minor sample preparations, also including materials which are non-transparent to infrared radiation. Such a facilitation is made possible by pressing the sample directly onto various crystals with high refractive indices, e.g. diamond, in a special reflectance setup. Thus ATR saves time and enables the study of materials in a pristine condition, i.e. the comprehensive sample preparation by pressing thin KBr pellets as in traditional FTIR transmittance spectroscopy is avoided.

However, the ATR-FTIR analysis of different building material samples exhibit various levels of experimental difficulties. In this investigation we have studied the FTIR spectra of climate weathered wood, wood rot, mould fungus on wood, mould fungus on plaster board and plastic degradation by ultraviolet radiation. Both qualitative and quantitative results may be obtained by an ATR-FTIR analysis using regular recordings of FTIR spectra. In order to determine the ageing progress and assess the effective lifetime of materials, components or products, FTIR spectra should be recorded both before, during and after the climate ageing. Hence, FTIR may be developed as an important tool to track the condition of various materials, components and products during the lifetime of a building, and thereby be applied as a tool for condition assessment, selection of maintenance intervals and service life prediction of buildings.

Keywords: Accelerated climate ageing, Durability, Lifetime, Building material, Fourier transform infrared, FTIR, Attenuated total reflectance, ATR.

1. Introduction

The climate ageing of various building materials represents a substantial strain to the buildings, both on a material, component and structural basis. The climate exposure factors may be divided into the following:

- Solar radiation, i.e. ultraviolet (UV), visible (VIS) and near infrared (NIR) radiation.
- Ambient infrared (IR) heat radiation (the resulting elevated temperature increases the rate of chemical degradation reactions, and also the rate of growth of rot and fungus up to limiting temperatures).
- High and low temperatures.
- Temperature changes/cycles (relative temperature movements between different materials, number of freezing point passes during freezing/thawing).
- Water, e.g. moisture, relative air humidity, rain (precipitation) and wind-driven rain.
- Physical strains, e.g. snow loads.
- Wind.
- Erosion (also from above factors).
- Pollutions, e.g. gases and particles in air.
- Microorganisms.
- Oxygen.
- Time (determining the effect for all the factors above to work).

Note that wood rot and mould fungus specifically, may not be listed as climate exposure factors, i.e. the wood rot and mould fungus are in this context viewed as (unwanted) results of the specific climate factors moisture and temperature for a certain exposure time with a sufficient supply of nourishment (e.g. wood). In fact, although commonly not regarded as so, the availability of fungal spores may be seen as a special climate exposure factor. Fungal spores are almost always present, except under sterile conditions.

Infrared spectroscopy may be utilized as an interesting and powerful tool during investigations of the degradation and durability of building materials or products. Various works have been carried out applying infrared spectroscopy in studies of wood weathering and wood photodegradation, e.g. by Anderson (1991), Colom (2003), Humar (2006), Mohebbi (2005), Pandey (2003, 2005), Sudiyani (2003) and Yamauchi (2004) and co-workers. Anderson et al. (1991) studied the effects of artificial weathering of softwoods and hardwoods with water alone, sunlight (xenon lamp) alone and both water and sunlight applied in conjunction. ATR analyses were performed by Humar et al. (2006) and Mohebbi et al. (2005). Miscellaneous works are performed by applying infrared spectroscopy in various studies of microorganisms, e.g. by Fischer (2006), Irudayaraj (2002), Kos (2002), Mohebbi (2005), Naumann (2005), Ngo-Thi (2003), Orsini (2000), Pandey (2003, 2004) and Wenning (2002) and their co-workers. Jelle et al. (2008), Jelle and Nilsen (2011), Kos et al. (2002), Mohebbi (2005) and Orsini et al. (2000) performed their works by employing the ATR-FTIR spectroscopical technique, which they describe as promising and with many advantages in these types of investigations. Several works are also carried out using infrared spectroscopy in various studies of weather ageing and photodegradation of different polymer materials, e.g. by Commereuc (1997), Croll (2003), Gerlock (1998) and Muasher (2006) and their co-workers. More general literature concerning photodegradation of polymers may be found in Pospíšil et al. (2006), Rånby and Rabek (1975) and Rabek (1995, 1996). The common practice and most widely applied experimental method is the traditional KBr pellet sample technique in infrared transmittance modus. Although there exists many experimental challenges using the ATR-FTIR method on various solid state materials (e.g. wood and fungus), this method is nevertheless very promising and with many advantages and is therefore the subject of our investigations in this work.

The various building materials have different resistance and durability towards the different climate exposure factors. It is important to protect the assorted materials versus the climate influence as a single material failure due to climate ageing may lead to failure of the whole building component and in the worst case even jeopardize the whole building structure. A material's resistance towards climate strains has therefore a direct impact on both economical and safety issues during the whole lifetime of a building.

That is, building materials and their durability and ageing are essential to the performance of buildings and their building physical aspects over time. Various strategies exist to protect the materials versus deterioration, e.g. paints, varnishes and impregnation for wooden claddings and UV stabilizers for polymers like polyethylene, polypropylene and others.

Natural climate ageing processes take long time to proceed, and often the buildings in question are supposed to be erected in the close future, i.e. it is impossible to wait for results from a long-term outdoor natural exposure test. To provide results fast enough, and within economical limits, accelerated climate ageing tests may be conducted. It is important *not* to induce any changes or chemical reactions in the materials that would not occur during an outdoor natural ageing process. That is, in an accelerated ageing test, only processes which would also occur in an outdoor natural ageing should be initiated and accelerated. For example, UV radiation with lower wavelengths than what exists in natural solar radiation should definitely not be applied in an accelerated climate ageing apparatus, as the short-wave UV radiation may bring about a degradation and chemical reactions that would never take place in nature. Earlier accelerated ageing apparatuses were in fact too often employing larger amounts of UVB radiation with too low wavelengths.

In this work we are attempting to utilize the attenuated total reflectance (ATR) Fourier transform infrared (FTIR) radiation experimental technique in order to study degradation of various building materials exposed to accelerated climate ageing. The ATR-FTIR technique makes it possible to study materials which are non-transparent to infrared radiation in a pristine condition. That is, the extensive, time-consuming and often cumbersome sample preparation by pressing thin KBr pellets as in traditional FTIR transmittance spectroscopy is avoided. The traditional technique might even change the sample material in question. The ATR technique is based on a special reflectance setup where the sample is pressed directly onto various crystals with high refractive indices, e.g. diamond. One goal is to be able to quantitatively determine the building material decay by performing ATR-FTIR analysis. Fortunately, for life time predictions, it is not necessary to identify what species are formed/degraded, as it is sufficient to determine quantitative relative changes.

Experimentally the task is challenging. Firstly, for some materials it may be difficult to differentiate the degradation products on the attacked sample from the substrate material itself. Secondly, it may be complicated to be able to distinguish between various degradation products. Thirdly, some building materials are not homogenous, which may complicate the measurements. Finally, with the ATR technique it is important to achieve and ensure a good contact with no air pockets between the sample and the ATR crystal in order to obtain correct quantitative results. Several building materials have so relatively hard and rough surfaces that such a good contact might be difficult to achieve, and especially to ensure that the actual contact is the best one attainable.

Five different building material degradation categories are chosen in the work presented here, namely:

- Climate weathered wood.
- Wood rot.
- Mould fungus on wood.
- Mould fungus on plaster boards.
- Plastic degradation by ultraviolet radiation.

As we will see in the following, these five building material degradation categories exhibit various experimental difficulties.

2. Experimental

2.1. Sample Materials

The results presented here is part of a larger study where several different building material samples are being subjected to both natural weather ageing and various accelerated climate ageing apparatuses. However, in order not to overburden the reader, only one selected sample from each category is shown in the following:

Wood	Pine heartwood, <i>Pinus silvestris</i> L., measured dry mass density of 0.48 kg/dm ³ . Raw, non-impregnated sample.
Wood Rot	Wood rot on wood, area on wood piece heavily infected with wood rot.
Wood Fungus	Mould fungus on wood. Mould fungus scraped off from wood, i.e. measured on a small mould fungus powder pellet.
Plaster Fungus	Mould fungus on plaster board. Mould fungus scraped off from GU plaster board, i.e. measured on a small mould fungus powder pellet. GU = exterior grade gypsum board, i.e. "outdoor use".
Plastic	High density polyethylene (HDPE), intended for application as water barriers/repellants around building foundation walls.

The small sample materials for the FTIR measurements have been collected from the larger samples depicted in Figure 1. For the Plastic sample the FTIR measurements have been performed directly on the sample shown in Figure 1.



Figure 1. Wood, Wood Rot, Wood Fungus, Plaster Fungus and Plastic samples. Small pieces are cut from the samples for use in the FTIR investigations, except the Plastic sample which is used directly.

2.2. Accelerated Ageing in QUV Apparatus

The accelerated ageing of the Plastic sample shown here was performed in a QUV apparatus, Weathering Tester Horizontal Option with Ponding and Water Spray (The Q-Panel Company, Cleveland, Ohio, USA), which subjected the sample to UV radiation at a constant air temperature of 50°C. The UVA and UVB intensities are averaged to 28 W/m² and 2.8 W/m², respectively. No water spray was applied. The shortened form QUV is applied throughout the text as an abbreviation for this specific exposure.

2.3. Accelerated Ageing in Atlas Solar Simulator

The accelerated ageing of the Wood sample shown here was carried out in an Atlas SC600 MHG Solar Simulator climate chamber with a 2500 W MHG lamp. The wood sample was tilted a bit from the horizontal ($8 \pm 2^\circ$) in order to let the water run off the surface. The sample was placed in the climate chamber with a distance of approximately 55 cm from the climate chamber glass ceiling to the samples, where the solar radiation intensity is reported to be 1200 W/m² at 100 % lamp power intensity. During the ageing period the UVA and UVB radiation intensity was measured at various times to be lying within the interval 60-80 W/m² and 3-6 W/m², respectively. The UV measurements were performed with a radiometer/photometer Model IL 1400A (International Light) with an UVA sensor and an UVB sensor. Note that a 6 % UVA fraction (like in sunlight) of the total solar intensity (1200 W/m²) yields 72 W/m² UVA radiation. The exposure duration consisted of 115 whole cycles of 24 hours, each cycle divided into 20 hours with a solar radiation intensity of 1200 W/m² and 4 hours with water spray and no solar radiation exposure. The two water spray nozzles gave each 0.5 dm³/min, i.e. 1 dm³/min in total, which gives approximately 1.7 dm³/(m²min) assuming an even horizontal water distribution at the sample location in the whole climate chamber. Integrated up, the solar radiation (MHG lamp) energy is 24 kWh/m² per day and 2760 kWh/m² during 115 days. Likewise, assuming 70 W/m² as an average, the UVA radiation energy is 1.4 kWh/m² per day and 161 kWh/m² during 115 days. The temperature and relative air humidity were held constant at 63°C and 50 % RH during the solar radiation exposure and at 10°C and close to 100 % RH during the water spray application. The shortened form Atlas is applied throughout the text as an abbreviation for this specific exposure.

2.4. FTIR Measurements

The FTIR material characterization was carried out with a Thermo Nicolet 8700 FTIR spectrometer with a Smart Orbit accessory, i.e. a horizontal attenuated total reflectance (ATR) accessory (single reflection) with a diamond crystal, in the wavelength range 4000 cm⁻¹ (2.5 μm) to 400 cm⁻¹ (25 μm) in an atmosphere with minimalized CO₂ and H₂O content through purging by a Parker Balston 74-5041 FTIR Purge Gas Generator. Each FTIR spectrum presented is based on a recording of 32 scans at a resolution of 4 cm⁻¹.

The principle of the FTIR material characterization is that infrared radiation is absorbed by different atomic and molecular bonds at different wavelengths or wave numbers, e.g. by miscellaneous atomic and molecular vibrations. Neighbouring chemical bonds are also influencing each other with respect to their absorption of the infrared radiation, e.g. wavelength shifts for the same chemical bond with different neighbouring chemical bonds. Hence, each chemical compound will have an unique FTIR transmittance or absorbance spectra, i.e. an unique fingerprint of the material.

Traditionally, FTIR transmittance, or even the older dispersive IR transmittance method, has been employed as the tool for material analysis. However, the infrared transmittance techniques require that it is actual possible to get some infrared radiation through the material sample, which for most solid state samples is not the case. Thus, such samples have to be diluted by various means, e.g. by mechanical grinding or chemical dissolving and subsequently at a low concentration mixed with a base material mainly transparent to IR radiation (e.g. KBr), which are finally pressed into e.g. KBr sample pellets. This process is rather time-consuming and may also change the sample material somewhat.

The ATR measurement technique however, allows FTIR spectra recording directly on the pristine material surface without any sample preparation, where slicing off sample layers also allows the study of the bulk material in its pristine condition. Both solid state materials, liquids and gases are readily studied. The principle of the ATR technique is that when there is no sample pressed towards the ATR crystal, all the incident IR radiation is reflected back from the crystal/air interface due to the large difference in refraction indices between the crystal (e.g. diamond) and air, i.e. total reflection at that specific angle of incidence (e.g. 45°). However, when a sample with refraction index larger than air is pressed towards the ATR crystal, some of the IR radiation will penetrate into the ATR crystal and get absorbed there, while most of the IR radiation will be (total) reflected as normal, i.e. attenuated total reflectance. Thus, the absorbance is measured and calculated as the difference between the reflected IR radiation with and without a sample pressed onto the ATR crystal.

In order to ensure satisfactory contact between the ATR diamond crystal and the sample, three or more FTIR spectra were recorded at various locations on the sample. Relatively hard surfaces complicate accurate quantitative measurements (height of absorbance peaks) due to varying contact with the ATR crystal for the different samples. Air between sample and ATR crystal results in a weaker absorbance signal. Unless other conditions indicate otherwise (e.g. inhomogenities, impurities, etc.), the FTIR curves with the largest absorbance peaks represent the most correct measurements on one and the same sample with equal ageing time, and hence these curves are chosen as they are assumed to be the most correct ones. Qualitative measurements (location of absorbance peaks at wave numbers) do not represent a problem as long as the contact area is large enough to ensure a sufficient strong measurement signal.

The FTIR spectra given in this work have not been ATR corrected, neither with respect to penetration depths nor absorbance band shifts, which both are dependent on the refractive indices of the sample and the ATR crystal (diamond in this case) and the angle of incident radiation. The penetration depth is in addition also dependent on the radiation wavelength, and increases with increasing wavelength (decreasing wave number). That is, non-corrected ATR spectra have much stronger absorbance bands at longer wavelengths (smaller wave numbers) than at shorter wavelengths (larger wave numbers). Note that it should always be stated if an ATR-FTIR spectrum has been ATR corrected or not, e.g. important during computerized database spectra comparison searches. As we in this work are solely comparing the ATR-FTIR spectra measured within this work, there is no need for performing any ATR corrections. Besides, the raw ATR-FTIR data in either transmittance or logarithmic absorbance mode is usually preferred. It should also be noted that one often do not know the refractive indices of the samples which are measured, thus errors might be introduced in the ATR corrected spectra as the refractive index of the sample is an input parameter in the ATR correction. Figure 2 shows a small wood piece from the wood sample pressed onto the diamond crystal in the ATR accessory connected to the FTIR spectrometer.

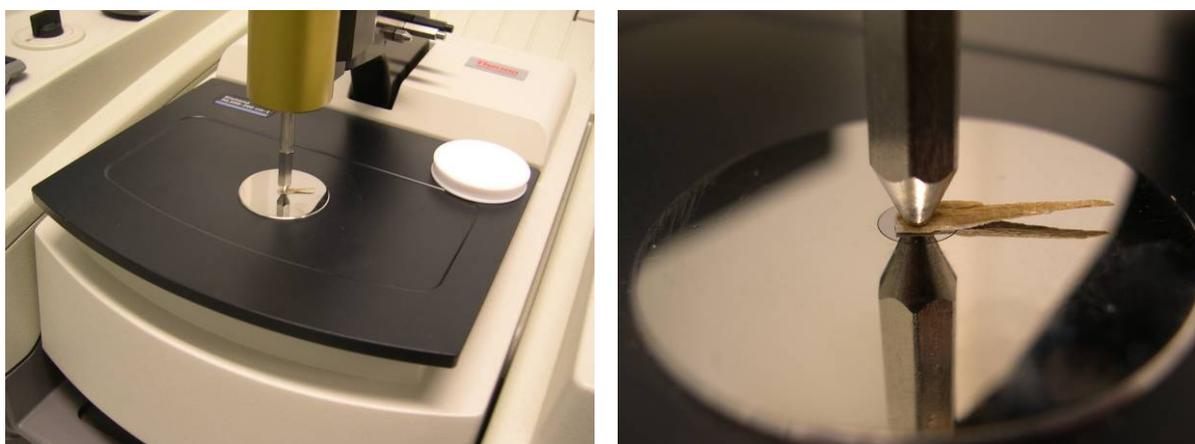


Figure 2. A small wood piece from the wood sample was pressed onto the diamond crystal in the ATR accessory connected to the FTIR spectrometer. In the close-up photo to the right, the lower steel pin is only a reflection of the above steel pin in the polished mirror-like sample plate.

3. Results and Discussion

FTIR transmittance spectra versus wave number between $4000\text{-}400\text{ cm}^{-1}$ for the different building material samples at various ageing levels are presented in Figs.3-5 (left). Further close-ups of these spectra shown as FTIR absorbance versus wave number are also depicted in Figs.3-5 (right). Furthermore, an unexposed wood sample and an unexposed GU plaster board sample are shown in Figure 4. In addition in Figure 4, as a reference to avoid any misinterpretations, the distilled water, non-dried gypsum and dried gypsum spectra are also plotted. Note that the irregularities in the FTIR spectra between $2200\text{-}1900\text{ cm}^{-1}$ (Figs.3-5) are due to the very large absorption in the ATR diamond crystal between these wave numbers, which represents the weak point in an otherwise excellent material choice for ATR applications.

As the absorption of electromagnetic radiation, e.g. infrared radiation, follows the Beer-Lambert law, i.e. the radiation is decreasing exponentially with the penetration depth in the actual material, it is often helpful to plot the spectra on a logarithmic absorbance scale vs. wavelength. Hence, a representative spectrum is chosen from each of the samples and plotted on a logarithmic absorbance scale for quantitative studies. Mathematically and physically it follows that a doubling of the logarithmic absorbance, also called optical density, is interpreted as a doubling of material thickness or a doubling of concentration of absorption active agents.

The building material decay makes the sample material undergo chemical changes and may then as a result of the chemical reactions change the actual thickness of the sample. In this work the experiments are conducted by applying the ATR equipment with the FTIR spectrometer. Hence, the infrared radiation is only penetrating into a thin surface layer of the actual sample. With respect to the experiments carried out in this work, the material thickness will then be regarded as approximately constant, i.e. the change in the infrared absorbance is explained by an increase or decrease of absorption active species within the sample material undergoing the chemical transformation. Also note that different adjacent neighbour atoms in a compound, e.g. in a polymer chain, will shift the wave number somewhat for the absorbance peak corresponding to the chemical bond in question.

By inspecting the FTIR spectra in Figure 3 (and other spectra not depicted here) more closely it is found that several absorbance peaks in the fresh, non-aged wood samples are diminished and finally vanished during the accelerated ageing in the Atlas Solar Simulator. These absorbance peaks are located around $1730\text{-}1710\text{ cm}^{-1}$ (varying with different wood specimens), 1510 cm^{-1} , 1260 cm^{-1} and 810 cm^{-1} . The peak around $1730\text{-}1710\text{ cm}^{-1}$ are attributed to carbonyl (C=O) stretching in hemicellulose, whereas the peak around 1510 cm^{-1} arises from the C=C stretching of the aromatic ring in lignin. These two peaks located around $1730\text{-}1710\text{ cm}^{-1}$ and 1510 cm^{-1} represent good candidates for studying wood decay by climate ageing through FTIR analysis. For the Wood sample (Figure 3) there are some other changes (i.e. increase and/or decrease) in the absorbance peaks during the ageing period also, but generally no completely disappearance of peaks and neither appearance of new peaks. Note that the water absorbance peaks are located at 3265 cm^{-1} (broader peak, -OH) and 1637 cm^{-1} (narrower peak, -O-), and changes in the FTIR spectra around these wave numbers might also be due to various moisture levels in the samples.

In Figure 4 the narrow, large ellipse encircles FTIR absorbance peaks which may be related to mould fungus and/or wood rot, but this is more uncertain due to the water peak at this location. Note that any microbiological growth may increase the water content. Furthermore, in order to quantify any potential pollution (error) from any astray gypsum powder, the gypsum peaks are also depicted, as these falls in the same wave number range. The thick, small ellipse in Figure 4 encircles FTIR absorbance peaks which are found to be characteristic for the mould fungus and wood rot. These peaks, also including the other samples not shown here, lie between 1558 cm^{-1} and 1535 cm^{-1} . That is, the chemical bonds which is the source for the absorbance peaks at these wave numbers, are found to be present and characteristic for the various mould fungus and wood rot products studied in this work.

An inspection of Figure 5 reveals that due to the ageing processes large absorbance peaks are growing up at wave numbers around 1732 cm^{-1} and 1713 cm^{-1} and also around 1200 cm^{-1} . The peaks around 1732 cm^{-1} and 1710 cm^{-1} are attributed to carbonyl ($\text{C}=\text{O}$) stretching, i.e. an oxidation of the polymer occurs during UV exposure in the QUV apparatus.

Experimentally, all these solid state building material samples had so hard and rough surfaces that it could be difficult to obtain a good contact with no air pockets between the sample and the ATR crystal, and especially to ensure that this contact in fact was the best one attainable. The Plastic sample had the smoothest surface, and was therefore the easiest sample in this respect. In addition, the Plastic sample was homogenous, i.e. you are assured to be measuring on the same material over the whole sample surface. For the Wood, Wood Rot, Wood Fungus and Plaster Fungus samples it could be rather difficult and time-consuming to differentiate the degradation products on the attacked samples from the substrate materials themselves and to be able to distinguish between various degradation products.

Hence, in this study it has been demonstrated that the ATR-FTIR spectroscopical technique may be applied in order to detect different levels of climate induced building material decay or ageing. Further studies and experimental investigations are needed in order to elaborate the suitability, the reliability, the limitations and the future potential possibilities of this method. Note that miscellaneous building material samples exhibit various levels of experimental difficulties in the ATR-FTIR analysis. In future applications of this experimental method as a detection tool capable of differentiating between various building material decay or ageing levels, there is a need to build up large reference databases containing FTIR spectra of the different species.

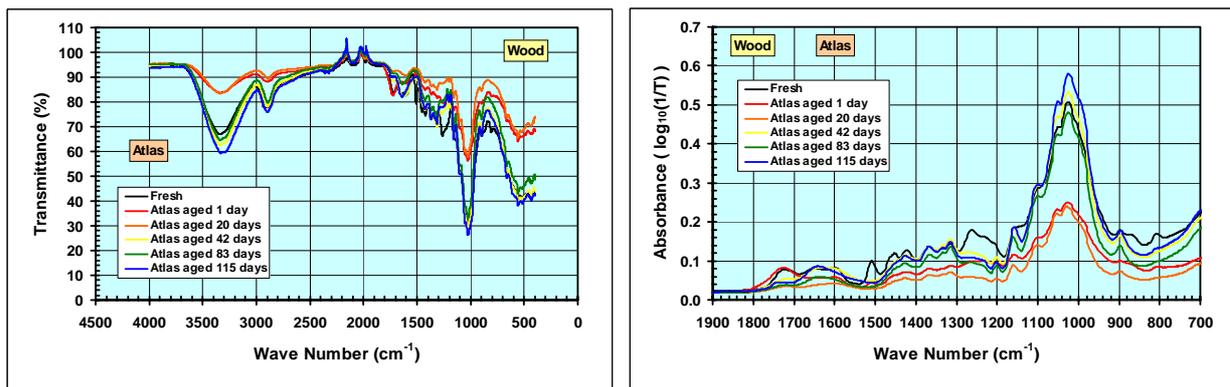


Figure 3. Transmittance vs. wave number between $4000\text{--}400\text{ cm}^{-1}$ (left) and absorbance (logarithmic) vs. wave number between $1900\text{--}700\text{ cm}^{-1}$ (right) for the Wood sample during accelerated ageing in an Atlas Solar Simulator.

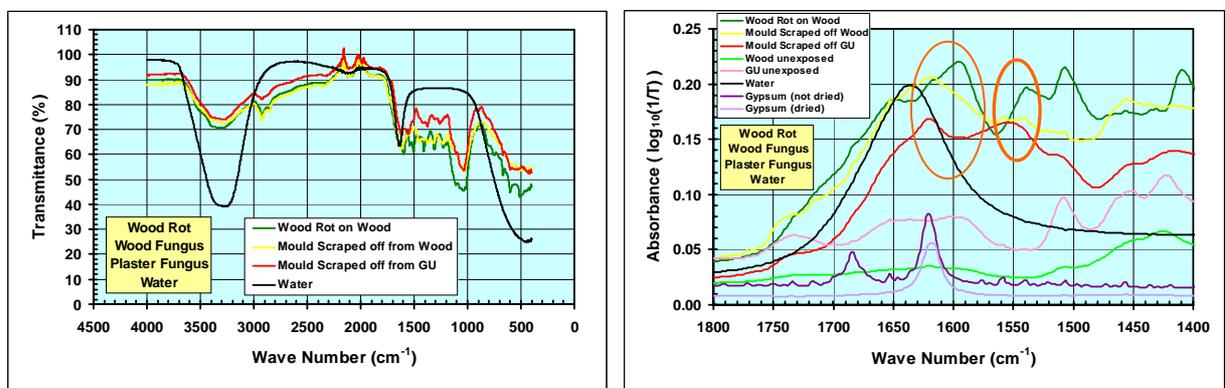


Figure 4. Transmittance vs. wave number between $4000\text{--}400\text{ cm}^{-1}$ (left) and absorbance (logarithmic) vs. wave number between $1800\text{--}1400\text{ cm}^{-1}$ (right) for the Wood Rot, Wood Fungus and Plaster Fungus samples. In addition, the water spectrum is included as a reference. In the close-up spectra (right),

unexposed wood, unexposed GU plaster board, water, non-dried gypsum and dried gypsum spectra are also plotted as a comparison. See discussion in text about the FTIR absorbance peaks within the narrow, large (left) and thick, small (right) ellipse encircling the absorbance peaks.

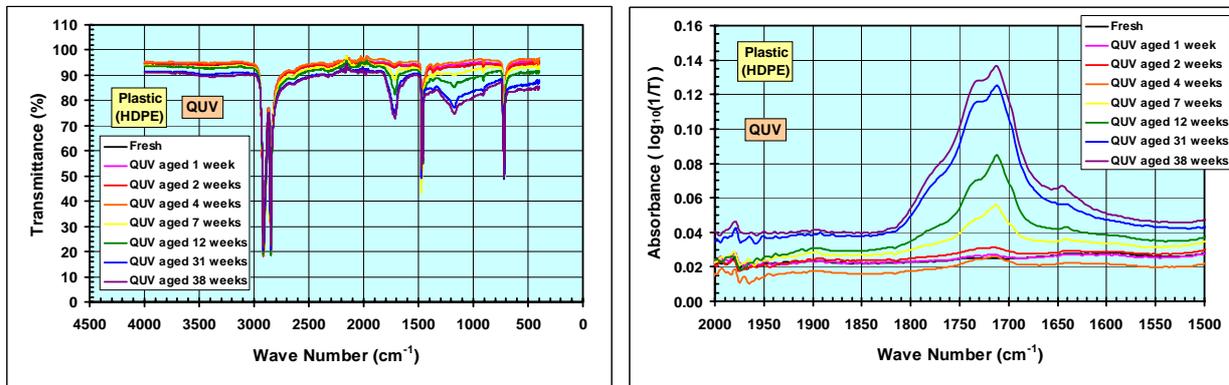


Figure 5. Transmittance vs. wave number between 4000-400 cm^{-1} (left) and absorbance (logarithmic) vs. wave number between 2000-1500 cm^{-1} (right) for the Plastic sample during accelerated ageing in a QUV apparatus.

The outcome or long time benefits from such experiments could be early detection of initial ageing processes and further extrapolation of the ageing processes quantitatively and thereby estimation of the effective lifetime of a building material or product. That is, one may ultimately avoid long time outdoor natural and accelerated artificial ageing exposure experiments, saving both time and costs. In addition, a correlation between various material properties, e.g. tensile strength, and FTIR spectra may be sought and found. Thus, some material properties testing may be replaced with the less time-consuming and more cost-effective FTIR analysis. Successful utilization of accelerated artificial climate ageing and estimation of effective lifetime of materials, components and structures will be crucial in the development of robust and durable building products, including aspects like future climate changes towards harsher climates and the development of new and advanced building materials and solutions. Any calculation of a total and absolute acceleration factor and the possibilities of exact lifetime predictions are rather difficult to perform, exhibiting many complications at several levels. Hence, relative lifetime estimations and comparisons of building product samples are thus more feasible than absolute predictions of lifetimes.

4. Conclusions

Miscellaneous building material samples exhibit various levels of experimental difficulties in an attenuated total reflectance (ATR) Fourier transform infrared (FTIR) radiation spectroscopical analysis. Nevertheless, this study has demonstrated that the ATR-FTIR technique may be applied in order to detect different levels of climate induced building material decay or ageing. The development of robust and durable building products depends on a successful utilization of accelerated climate ageing and estimation of effective lifetime of materials, components and structures, e.g. also with respect to future climate changes towards harsher climates and development of new and advanced building materials and solutions. However, further studies and experimental investigations are needed in order to elaborate the suitability, the reliability, the limitations and the future potential possibilities of this method.

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