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Durability of Vacuum Insulation Panels in Alkaline Environment

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The English abstract is found on the first page of the article:

Norsk sammendrag:
Betongelementer (sandwichelementer) og Lecablokker (isoblokker) er mye brukt i byggeindustrien i dag. Ved å erstatte tradisjonelle isolasjonsmaterialer, som ekspandert polystyren (EPS), ekstrudert polystyren (XPS) og polyuretan (PUR) med vakuumisolasjonspaneler (VIP), er det mulig å redusere den termiske ledningsevnen uten å øke veggtykkelsen. Sammenlignet med dagens sandwichelementer og isoblokker, kan slankere elementer fremdeles oppnå U-verdier som tilfredsstiller passivhus-standarder. På denne måten kan man også redusere energibruken og utslippet av CO_2 i byggeindustrien Dette er en av grunnene til at VIP muligens kan være et godt alternativ til de tradisjonelle isolasjonsmaterialene. Men det er allikevel enkelte problemer relatert til VIP, spesielt i kombinasjon med betong. Det alkaliske miljøet i betongen vil kunne reagere med aluminiumen i VIP-folien, noe som vil kunne ødelegge folien, og ende med at panelet punkterer. For å undersøke innvirkningen av alkalisk miljø på VIP ble forskjellige eksperimenter utført i laboratoriet hos SINTEF Byggforsk. Det ble gjennomført forsøk på VIP i forskjellige pH-løsninger og temperaturer. Panelene viste forskjellige fysiske og termiske aldringstegn, avhengig av hvilke påkjenninger de ble utsatt for. Generelt viste det seg at temperatur var den mest avgjørende faktoren, da de panelene som var plassert i høy temperatur i varmeskap mistet vakuumet etter kun kort tid, mens de panelene som var plassert i romtemperatur viste seg å være relativt bestandige, nesten uavhengig av pH-verdien på løsningene de var utsatt for. Det ble også utført et forsøk som gikk direkte på VIP-folien, der folien ble separert fra resten av panelet, og eksponert for sterk alkalisk løsning over tid. Resultatet fra dette forsøket var ganske overraskende, da folien viste tydelige aldringstegn etter relativt kort tid.

Keywords:

1. Vacuum Insulation Panel
2. Concrete
3. Alkaline
4. Ageing

(sign.)

Preface

This Master of Science thesis is the last compulsory activity required of graduate students and was carried out throughout my 10th semester at the Norwegian University of Science and Technology (NTNU). The work is delivered under the Department of Civil and Transport Engineering, in close co-operation with SINTEF Building and Infrastructure. This work has been written as an article to be submitted to a relevant scientific journal yet to be decided.

The work started up during fall 2011 (9th semester), when a project thesis on the topic "Durability of Vacuum Insulation Panels in Alkaline Environment" was worked out. This was an experimental task, where experiments on VIPs in different alkaline environment and at different temperatures were carried out. However, the time frame of these experiments was not sufficient, thus it was desired to take the same topic into a Master of Science thesis. New experiments, similar to the ones that were carried out during the 9th semester were carried out, over a time frame of 12 weeks. When discussing the development of the VIPs after only three weeks of testing, it was desired to have a closer look at the VIP envelope in particular as well. Thus, a VIP envelope specimen experiment was also carried out in addition to the VIP experiments.

Correspondence with Franco Bløchlinger and Metallplan AS, the provider of VIP, was established during fall 2011 and carried on through the work with my Master of Science thesis. In a short time they provided VIPs in the right sizes for my experiments. Franco deserves gratitude for his goodwill and co-operation in relation with my work.

I want to extend a special reward to Egil Rognvik at SINTEF and Ole Aunrønning at NTNU for great help in connection with the practical challenges with the experiments. Tao Gao at NTNU also deserves a great gratitude for helping me out with the scanning electron microscope (SEM) in relation with the VIP envelope specimen experiment. I would like to thank Samuel Brunner who has contributed constructively, both practical and theoretical, with this work. Last, but not least, I want to extent my rewards to my supervisor at SINTEF and NTNU Bjørn Petter Jelle for always helping me out and setting aside time for me.

It has been an interesting and powerful learning experience.

Trondheim, May 2012

Synne Chr. Helgerud

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Durability of Vacuum Insulation Panels in Alkaline Environment

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Abstract

Concrete and lightweight concrete elements are today used in various building applications to a great extent globally. Replacing traditional thermal insulation such as expanded polystyrene (EPS), extruded polystyrene (XPS) and polyurethane (PUR) by vacuum insulation panels (VIPs) is discussed in order to increase the thermal resistance without increasing the wall thickness. Compared to traditional concrete and lightweight concrete elements, slimmer elements may still achieve U-values low enough to fulfil passive house or zero energy building requirements. Thus, sandwich elements with VIPs may be an alternative to the traditional solutions. However, there may be some challenges related to the use of VIPs in such concrete elements. The alkaline environment in concrete may lead to reactions with the aluminium in the multi-layered laminate used as the VIP envelope, and destroy its barrier function. To investigate the influence of the alkaline environment on the durability of VIPs in general, and the VIP envelope in particular, various VIP and VIP envelope specimen experiments have been carried out. The VIPs were subjected to alkaline solutions at different pH-values and temperatures, with and without direct contact with the liquid alkaline solutions. A worst-case scenario was investigated when any additional protection of the VIPs was disregarded. The results from the VIP experiments showed various degrees of degradation effects. Depending on the temperature and the pH-value of the alkaline environment the VIPs were exposed to, physical changes were observed on some of the test specimens, while others were more or less unaffected during 12 weeks of exposure. In general, elevated temperatures proved to be the most significant strain. The VIPs in the heating cabinet showed much greater signs of degradation than the VIPs at room temperature, more or less independent of the pH-value of the alkaline solution they were exposed to. Noteworthy results were also obtained from the VIP envelope specimen experiment, where the VIP envelope showed signs of degradation after only a short time in alkaline solution.

Keywords: Vacuum insulation panel, VIP, Concrete, Alkaline, Ageing, Degradation, Durability, Laminate

1 Introduction

A steadily increase in required energy efficiency in buildings has led to today's considerably strict requirements. Due to the increased social consciousness of 'going green' and scientific knowledge to support the innovation of energy efficient construction projects, technical regulations will continually demand stricter requirements. The EU has enforced five headline targets as a part of their Europe 2020 goals, where one of the five headline targets is to cut the greenhouse gas emissions from year 1990 with 20 %, increase the energy efficiency by 20 % and to obtain 20 % of the energy from renewables (European Commission 2012). Consequently, these demands will potentially result in thicker walls, roofs and floors. By using 'insulated' concrete and lightweight concrete elements, required wall thickness would be over 450-500 mm (Weber Saint Gobain 2011), or when looking at the thermal insulation thickness only, typically 300 mm of mineral wool (Passipedia 2012) would be needed.

In suburban and urban areas, the plots are often small and expensive, which requires optimal use of the available area. Thus, more effective insulation materials with reduced thermal conductivity are desirable. A vacuum insulation panel (VIP) is a type of effective insulation material with a 'centre-of-panel' thermal conductivity of approximately 4 mW/(mK) in pristine conditions. Ideally, VIPs are typically 5 to 10 times more effective than traditional insulation such as mineral wool (Baetens et al. 2009). Due to much lower thermal conductivity of VIPs, the required thickness of the insulation layers and thus the total wall thickness, may be strongly reduced compared to a traditional construction.

The vacuum insulation panel technology is based on the principle that heat in the form of gas conduction and convection cannot be conducted in the absence of gas, for example air, even though radiation still occurs. The absence of molecules makes it impossible for the gas conduction to pass through a given distance, which makes vacuum a good insulator (Fricke et al. 2008). A VIP consists of a micro porous core structure and an air and vapour tight envelope. The main core constituent is fumed silica powder, while other significant components are opacifiers for minimizing infrared radiation (Porextherm 2011). The panels are heat sealed with a multilayer high barrier envelope to maintain the vacuum (Willems and Schild 2005). Various envelope configurations may be found in Wegger et al. (2011).

VIPs have been used in consumer products such as refrigerators since the 1990s (Fricke et al. 2008), but it was not until the 21st century that it was adopted to the building technology (Simmler et al. 2005). Some aspects of the use of VIPs in construction and buildings have previously been investigated. Simmler and Brunner (2005) have studied the application of VIPs in buildings considering ageing effects where VIPs between pH-values of 3.0 and 8.5 are highlighted as a 'safe zone' regarding the chemical reaction between the VIP envelope and an aqueous solution. Sveipe et al. (2011) have studied the use of VIPs in connection with retrofitting timber frame walls and Wegger et al. (2011) have studied different accelerated ageing effects on VIPs. Various core and envelope materials have also been studied. Alam et al. (2011) have reviewed studies on the production of VIPs using different core materials such as glass fibre, foams, perlite and fibre/powder composites to make it more suitable for building applications, whilst Schwab et al. (2005) have studied the gas transmission rates of different high barrier laminates, such as laminated metal films (AF) and multilayer metalized films (MF), for air and water vapour transport. Caps and Fricke (2000) tested various powder-fillings for load-bearing VIPs, such as micro porous silica powders.

However, there have been few studies investigating the use of VIPs in concrete and lightweight concrete elements. As of today no common understanding of the durability of VIPs in alkaline constructions seems to exist. The topic has so far slightly been discussed in the IEA/ECBCS Research Programme (2005), where a lightweight concrete wall insulated with EPS encapsulated VIPs on the external side was tested regarding the U-value (Simmler et al. 2005). In Germany a VIPBON project has investigated the use of VIPs in concrete elements, which lead to a demonstration building that was built up of such elements in 2005 (ZAE Bayern 2008). Also Grynning and Jelle (2010) have mentioned the matter by generally presenting the practical use and challenges of using VIPs near concrete in buildings. Hence, there is a need for investigating the durability of VIPs in an alkaline environment. Thus, this work investigates the behaviour and durability of VIPs placed in an alkaline environment, e.g. in concrete elements. A variety of ageing experiments have been carried out; VIPs have been exposed to different alkaline solutions as well as at different temperatures. Some VIPs were exposed to an alkaline solution through capillary suction, while others were submerged in an alkaline solution. To investigate whether it was possible for hydroxide (OH^-) to be transported through air, and thus degrade the VIP without direct liquid contact with the alkaline solution, a distillation experiment of alkaline solution was implemented. An experiment concerning the degradation of the VIP laminate in particular has also been carried out.

2 Experimental

2.1 Material Samples

2.1.1 Description of VIP

The VIPs used in this experiment are of the type Vacuspeed, from the producer Porextherm (Porextherm 2011). Due to the size of the available test containers, the smallest standard size of Vacuspeed had to be investigated, with the dimensions of 250 mm x 250 mm x 20 mm (nominal length x width x thickness), see Figure 2(b). The VIP envelope is stated to consist of three layers of metallized polyethylene terephthalate (PET) with a sealing layer of polyethylene (PE) on the inside, with a total thickness of 97 µm (Porextherm 2012), whereas the actual VIP envelope used in this work was measured to be 138 µm according to scanning electron microscope (SEM) analysis. The build-up of the envelope is equivalent to MF4, as described in Figure 1(a) (Wegger et al. 2011 and Brunner et al. 2006) except two additional layers towards the outside of the VIP envelope, see Figure 1(b). The second outmost layer is an additional fire retarder layer containing aluminium hydroxide ($Al(OH)_3$). The third layer is an additional organic layer of which the composition is not available so far.

A configuration of the cross-section of a non-aged VIP (photo taken with a SEM) is shown in Figure 2(a). The words ‘envelope’ and ‘laminate’ are used interchangeably.

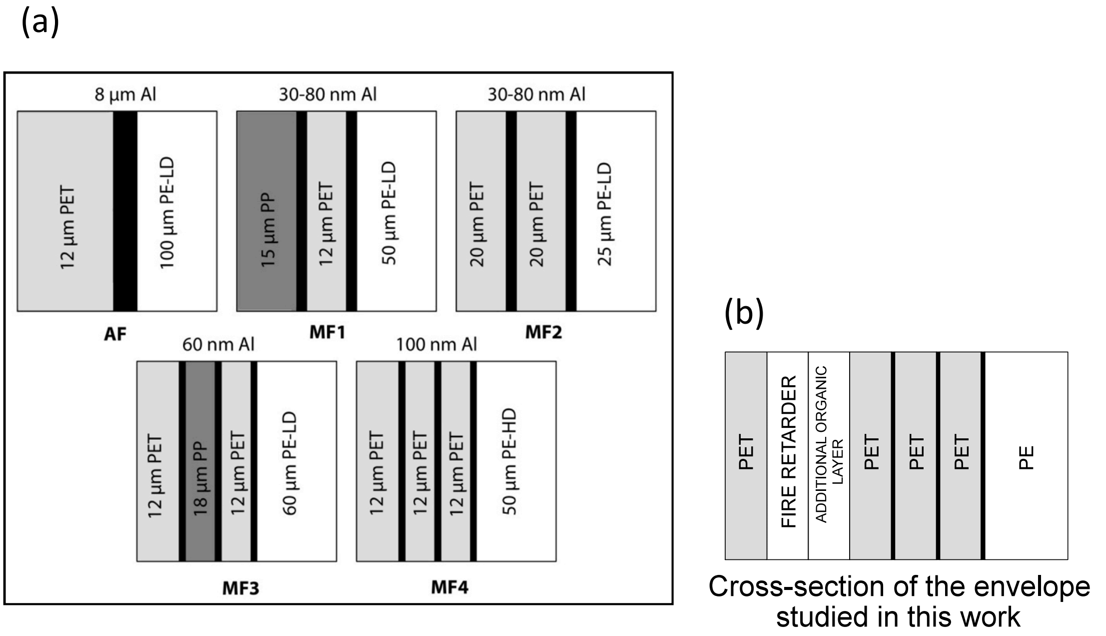

Figure 1. Left: Cross-sections of various envelope solutions for application in VIPs. Note: The thickness of the Al-layer denotes the thickness of each separate layer (Wegger et al. 2011). The 'outside' of the laminate is the PET-layer whilst the 'inside' of the laminate is the PE-LD/PE-HD layer. Right: Cross-section of the VIP envelope used in this work. Note: The cross-sections and the various layers are not drawn to scale.

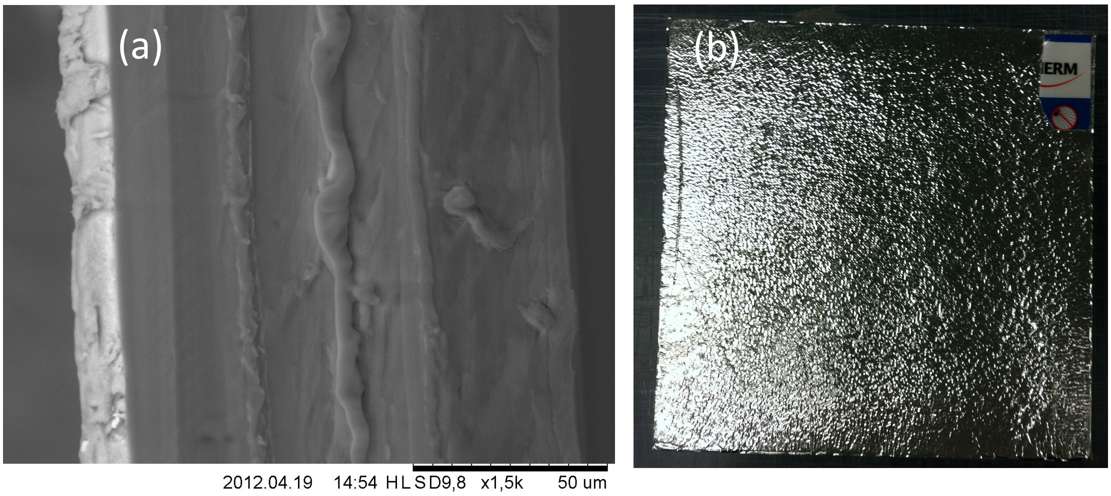

Figure 2. (a) Scanning electron microscope (SEM) cross-section of a non-aged VIP envelope, showing the multi-layered VIP laminate prepared with razor cutting. The 'outside' of the laminate is to the left. (b) A photo of a non-aged VIP.

2.1.2 Description of Concrete

The concrete used in this work was produced according to EN 1333 (170 mm x 270 mm x 1000 mm). The concrete was cast and then stored under a plastic foil for the first 24 hours. Thereafter, the concrete was cut into slices of 170 mm x 270 mm x 50 mm by using a diamond concrete saw, and then submerged in water for 5 days. Finally, the concrete slices were cured in laboratory climate for 22 days before they were ready for use. The concrete mix ratio and the actual measures used in the concrete mix are given in Table 1.

Table 1. Left: The concrete mix ratio. Right: The measures used in the concrete mix giving 50 dm^3 concrete.

Cement	1	Norcem Standard cement	17.00 kg
Fine aggregate, 0-8 mm	3.2	Årdal fine aggregate, 0-8 mm	54.40 kg
Coarse aggregate, 2-8 mm	1.8	Årdal coarse aggregate, 2-8 mm	30.60 kg
Water	0.53	Water	9.01 kg

2.2 Apparatus and Methods

2.2.1 General

Preliminary experiments have been carried out over a period of three months to obtain a deeper understanding of the experimental progress. Based on these experiments, systematic VIP ageing procedures were developed.

Containers with glass covers and gaskets were used for all the VIP experiments, which implies that the RH inside each container was 100 %. Thus, as with the VIP experiments submerged in alkaline solution, the RH of the VIP experiments that were moistened through capillary suction was also 100 %. This is shown in Figure 4. For the VIP envelope specimen experiment, one part of the envelope was submerged in alkaline solution, while the other part was over the alkaline solution, with RH ≈ 20 – 28 %, as shown in Figure 10.

2.2.2 Heat Flow Meter and Scanning Electron Microscope

A heat flow meter (HFM) apparatus was used to measure the change in thermal conductivity of the VIPs every three weeks during the 12-week testing period. Due to the limited size of the VIPs used in the experiment (250 mm x 250 mm), a customised setup for smaller test samples was applied, see Figure 3. The actual heat flow meters used were made of 1 mm thick, flexible, circular plate of polyurethane (PUR), with an outer diameter of 100 mm and a circular measuring area of 50 mm on each side of the specimen. To obtain stable conditions, with one constant warm side and one constant cold side, the whole metering area of the HFM apparatus (600 mm x 600 mm) had to be covered. Thus, the VIPs were placed in a frame of polyethersulfone (PES), see Figure 3. The measurements were performed in accordance with NS-EN 12667 (2001) and ISO 8301 (1991). A constant distance between the heating and cooling plates was used throughout the 12-week testing period, when the change of the thickness of the VIP panels proved to be insignificant.

Figure 3. The customized setup for smaller test pieces, used in the heat flow meter.

A scanning electron microscope (SEM, TM3000 TableTop Microscope, Nor Fab 2012) was used to investigate the configuration of the VIP envelope and possible degradations every three weeks during the VIP envelope specimen experiment, which lasted for nine weeks.

2.2.3 Degradation of VIP in Alkaline Environment

The VIPs in building application are required to have entirely satisfactory functions for a long period of time due to the fact that it is often expensive and/or almost impossible to replace a built-in component (Simmler and Brunner 2005). To investigate how the contact with moist concrete substantially reduces the long-term durability of the VIPs, the NT POLY 161, 1993, Method A was used. To simulate the potential properties of the VIPs after a long period of time methods of ageing VIPs at extreme pH conditions, lab tests were implemented. Figure 4 visualises the schematic setup of the different VIP experiments carried out. Also a reference test was carried out, shown in Figure 5, to obtain a better basis for comparison. The experimental investigations performed during the testing period are presented in Table 2. They are also described in detail in the following chapters.

Table 2. The experimental investigations carried out during the 12-week testing period.

Ageing time intervals (weeks)	0	3	6	9	12
Experimental investigations	• Visual observations (digital photos) • Dimensions • Mass • Thermal conductivity				

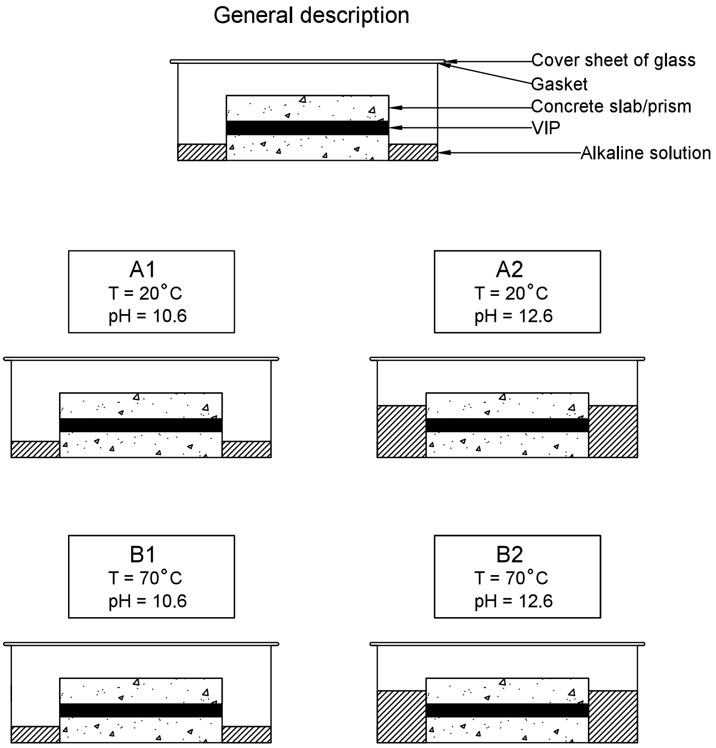

Figure 4. Schematic setup of the different experiments.

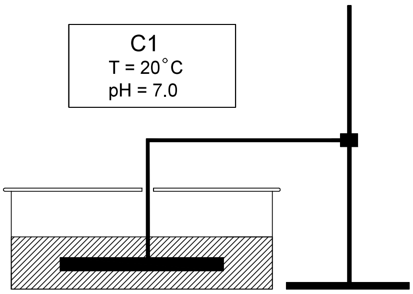

Figure 5. Setup of the reference experiment, C1. The concrete slabs were replaced with a plastic cylinder mounted to a support stand to keep the VIP under water. The plastic cylinder did not affect the pH-value of the solution, which was kept close to 7.0.

Experiment A1 and A2 – Degradation of VIP at Room Temperature

To evaluate the effect of alkaline environment on VIPs in general, a test was designed to expose the VIPs to different alkaline solutions at room temperature, T = 20°C.

'Normal' procedure to protect the VIP against the alkaline environment in the concrete is to place a protective plastic film between the VIP and the concrete. However, as a worst-case scenario the VIPs might get in direct contact with the concrete. That may be if the protective plastic film between the concrete and the VIP is absent, or has large cuts, as visualised in Figure 6. The objective of this experiment was to measure the change in thermal conductivity of the VIPs in direct contact with the concrete (without the protective plastic film), and to estimate how long the VIPs will survive subjected to a given alkaline environment.

With Experiment A1 the water level was 15 mm under the upper edge of the *underlying* concrete slab, to create capillary suction. With Experiment A2 the VIP was fully submerged in alkaline solution. See Figure 7 for a schematic setup of the two experiments. To keep the water level constant, it was, when necessary, refilled with alkaline solution throughout the experiment.

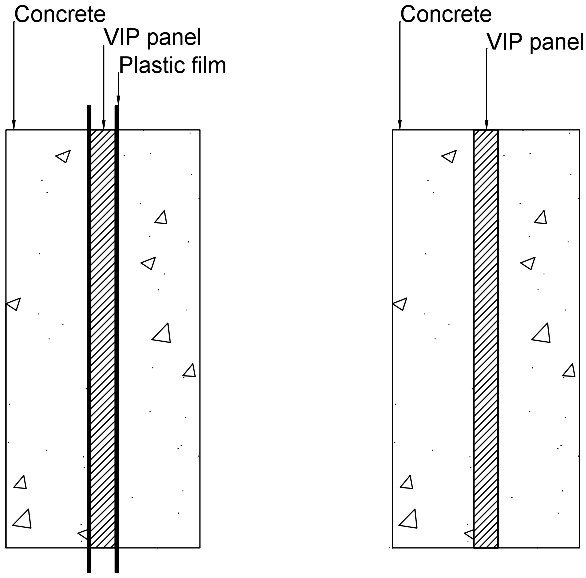

Figure 6. The figure to the left illustrates the ideal scenario, with a plastic film between the concrete and the VIP. The figure to the right illustrates a worst-case scenario, where the protective plastic film between the concrete and the VIP is absent.

Two different situations were investigated, as shown in Figure 7:

A1. pH = 10.6. The alkaline solution was taken from a container where the concrete was stored, and is therefore denoted 'natural concrete water'. The VIP was pressed to a moist concrete surface, continuously moistened with alkaline solution through capillary suction.

A2. pH = 12.6, aqueous calcium hydroxide $(CaOH)_2$ solution (~ 0.03M). This alkaline solution was made in the laboratory and represents an even worse situation than A1. The VIP was submerged in alkaline solution.

Three parallel A1 and A2 experiments were carried out in case of unforeseen events.

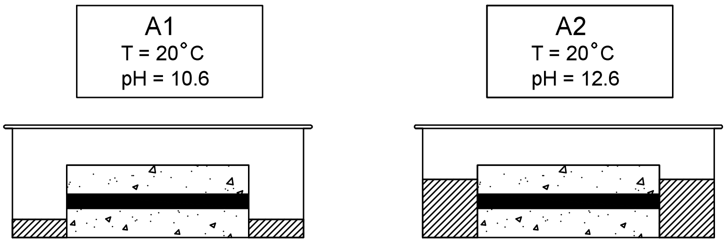

Figure 7. Schematic setup of experiment A1 and A2.

Experiment B1 and B2 – Degradation of VIP at 70°C

One method for testing accelerated ageing effects on VIPs in alkaline constructions is the Nordtest Method NT POLY 161 (1993) Method A, which this experiment is done according to. The procedures have been altered somewhat, by reducing the temperature in the heating cabinet from 90°C to 70°C, and by replacing the PE-foil (polyethylene foil) with a VIP. The manufacturer-declared maximum service temperature is 80°C, when applying a RH of 80 % at the same time. In this work a RH of 100 % was applied, and thus the temperature reduced to 70°C. The VIP replaced the PE-foil (polyethylene foil) in the Nordtest Method NT POLY 161 (1993) Method A due to the fact that it was the VIP that was important to investigate in this setting.

Even though the VIP is separated from the concrete with a plastic film, it may still get in contact with the concrete, and thus the alkaline environment, either due to diffusion through the plastic film, or through cuts. The objective of this experiment was to measure the change in thermal conductivity of the VIPs in direct contact with the concrete, subjected to a given alkaline environment (without the protective plastic film) and high temperature.

Both with Experiment B1 and B2, the test piece was stored in a heating cabinet at a constant, elevated temperature of 70°C. This represents an extreme condition, which may occur in especially unfavourable cases, on e.g. dark coloured building façades heated through solar radiation. With Experiment B1 the water level was 15 mm under the upper edge of the *underlying* concrete slab, to create capillary suction. With Experiment B2 the VIP was fully submerged in alkaline solution. See Figure 8 for a schematic setup of the two experiments. To keep the water level constant, it was, when necessary, refilled with alkaline solution throughout the experiment.

As with Experiment A, two different situations were investigated, as shown in Figure 8:

B1 pH = 10.6. The alkaline solution is taken from a container where the concrete was stored, and is therefore denoted 'natural concrete water'. The VIP was pressed to a moist concrete surface, continuously moistened with alkaline solution through capillary suction.

B2 pH = 12.6, aqueous calcium hydroxide $(CaOH)_2$ solution (~ 0.03M). This alkaline solution was made in the laboratory and represents an even worse situation than B1. The VIP was submerged in alkaline solution.

Three parallel B1 and B2 experiments were carried out in case of unforeseen events.

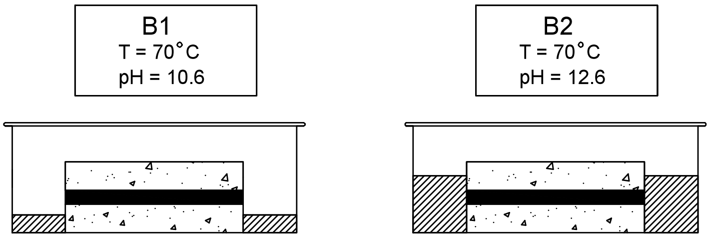

Figure 8. Schematic setup of experiment B1 and B2.

2.2.4 Degradation of VIP Envelope Specimen in Alkaline Environment

The most important aspect of the durability of the VIP is the laminate, how well it works as an air- and vapour tight barrier, see Figure 9. Degradation and delamination of the laminate will reduce the durability of the VIP, as it allows for air and vapour to seep into the core material, which will lead to increased thermal conductivity. The objective of the experiment was to obtain a better understanding of the ageing of the VIP envelope exposed to alkaline solution, expecting deterioration that could occur towards the end of the lifetime of the VIP envelope.

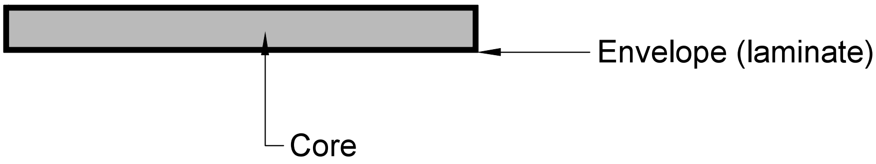

Figure 9. VIP configuration showing the envelope and the core.

A VIP envelope was separated from the VIP core. To make the experiment as realistic as possible, the backside of the envelope was sealed with lacquer. This was to represent exposure of the alkaline solution only from the outer side of the VIP, when the backside of the VIP originally is faced against the core, and is therefore not directly exposed to the alkaline solution. The VIP envelope specimen were immersed in 0.03 M $Ca(OH)_2$ aqueous solution with a pH-value of about 12.6 during a testing period of nine weeks, see Figure 10. The specimen was examined by SEM every three weeks.

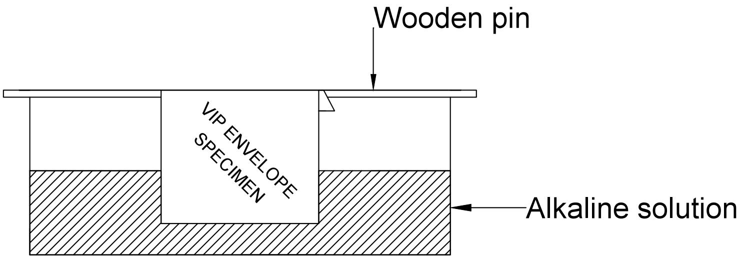

Figure 10. Schematic setup of the VIP laminate specimens experiment. The investigated part of the VIP envelope is the part that was kept under the alkaline solution.

2.2.5 Transport of Hydroxide

It may be obvious that alkaline liquid water that gets in *physical* contact with VIPs may cause destruction. However, if hydroxide (OH^-) may be transported through air, e.g. connected with water moisture as small water particles, this may also lead to degradation of VIPs. Therefore, a possible transport of OH^- through air was desired to have a deeper look into i.e. to verify whether such a transport of OH^- may occur.

To evaluate a possible transport of OH^- through air, a distillation experiment was carried out. The objective of the experiment was to determine whether it was possible for OH^- to be transported in air, and if so, determine the amount that was transported, and evaluate the consequences.

A distillation apparatus was used to evaporate the alkaline water. The bottle with alkaline water was placed in a bowl of silicone oil and heated to a temperature of 96°C. From this bottle of alkaline water, the solution was evaporated through a pipeline, which was cooled by running water. In the other end of the distillation apparatus, the solution was investigated by using a pH-meter.

3 Results and Discussion

3.1 Degradation of VIP in Alkaline Environment

3.1.1 General

The dimensions, weight and thermal conductivity of the VIP specimens were measured every three weeks during the 12-week testing period, except a second round of the B2 experiments, denoted the 'additional B2 panels' that were measured weekly during a three weeks testing period (explained later).

As the distance between the heating and cooling plates in the HFM was kept constant during the 12-week testing period, any possible thickness change of the panels throughout the period did not influence the thermal conductivity measurements.

The VIPs in the heating cabinet at 70°C were to a great extent affected by the alkaline environment and the high temperature they were subjected to. Each of these VIPs needed a very long time to achieve stable conditions in the HFM apparatus, with one constant warm side and one constant cold side. Due to the capacity in the laboratory it was not possible to let these VIPs stay in the HFM apparatus long enough to reach the final stabilisation. The thermal conductivity values obtained from the measurements may therefore, to some extent, be subjected to a relatively large uncertainty. Moreover, the uncertainty of the thermal conductivity measurements increased throughout the testing period, as the conditions of the VIPs became worse. An estimate of the uncertainty of the measured thermal conductivity of these panels is therefore set to be ± 10 %. However, the uncertainty of the measurements of the VIPs at room temperature is estimated to be ± 3 %. The measurements of weight and dimensions, as well as the thermal conductivity, may also be influenced by human errors.

Some alkaline solution gathered in the customized PES frame in the HFM apparatus for every testing period. The frame was dried out between every testing period, but this may however have influenced the thermal conductivity results.

3.1.2 Measurements on VIP at Room Temperature

Experiment A1 with pH = 10.6

Dimensions

For all the VIP experiments, it was difficult to measure change in any of the three dimensions (nominal length, width and thickness). A slide calliper was used. The measured changes were considered insignificant. The thickness changes for the experiments at room temperature (A1, A2 and C1) are shown in Table 3 and Figure 15. However, these changes were all within the uncertainty of the measurements, which is estimated to be ± 0.5 mm.

Mass

From Table 6, Table 7 and Figure 17 it is apparent that the A1 experiments followed almost the same pattern as the C1 reference experiment, and the weight increase for all the three A1 experiments and the C1 experiment was very small. The splices on the VIPs were taped around the surface from the producer, and therefore some water was gathered in the air pocket created by the tape. After the 12-week testing period, the tape was opened up and the panels were dried out. Measurements showed that the weight increase for all the A1 experiments and the C1 experiment was due to the water gathered in these pockets, see Table 6 and Table 7.

Thermal conductivity

The initial thermal conductivity for all the three A1 experiments was measured to be between 4.8 and 5.2 ± 0.1 mW/(mK). Throughout the testing period of 12 weeks the A1 experiments and the C1 experiment showed a minor increase in thermal conductivity, as given in Table 12, Figure 19 and Figure 20. The change in thermal conductivity for all the A1 experiments was actually less than 0.1 mW/(mK), which falls within the uncertainty of the measurements (estimated to be ± 3.0 %). As this minor change in thermal conductivity indicates, the VIPs showed no significant, visible signs of degradation, as visualised in Figure 11.

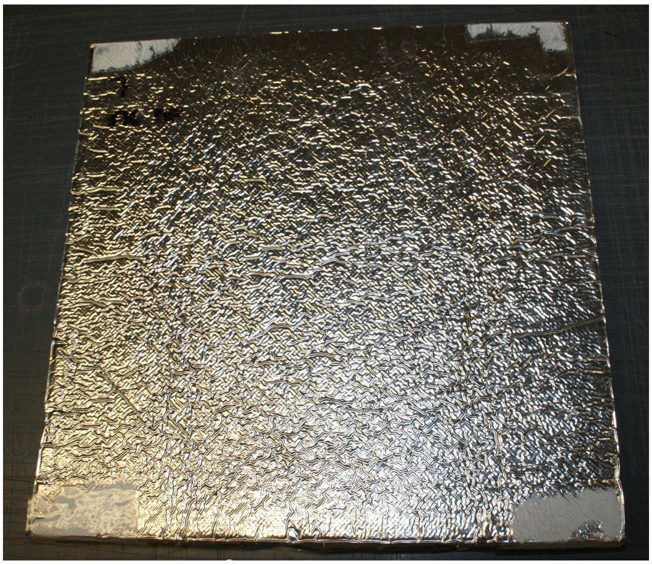

Figure 11. Experiment A1 after 9 weeks at room temperature, exposed to alkaline solution, pH = 10.6 through capillary suction. No visible signs of degradation on the VIP. The white areas around the corners of the VIP are due to the glue from the protective tape that protects the corners from mechanical damage. The tape also helps the panels to keep a squared shape for installation. An example of a non-aged VIP is shown in Figure 2.

Experiment A2 with pH = 12.6

Dimensions

See the dimensions chapter under experiment A1.

Mass

The A2 experiments followed the same weight increase pattern as the A1 experiments (Table 6, Table 7 and Figure 17) except that the A2 panels experienced a slightly greater weight increase during the 12-week testing period. Also here most of the weight increase can be explained by the water gathered in the air pockets created by the tape around the panels. Furthermore, the rest of the weight increase may be explained by the thin film of $CaCO_3$ that emerged on the surface of the panels, which comes from a combination of added $Ca(OH)_2$, $CaCO_3$ in the water and $CaCO_3$ from the concrete, as shown in Figure 12.

Thermal Conductivity

The A2 experiments followed the same thermal conductivity pattern as the A1 experiments, as shown in Table 12, Figure 19 and Figure 20. Different from the A1 panels, a thin, white film of calcium carbonate ($CaCO_3$) emerged on the surface of the A2 panels after three weeks, as shown in Figure 12. However, it seemed that the fire retarder layer and the three Al layers were still intact after the 12-week testing period (observations done by the naked eye), see Figure 12. It was first expected that the A2 experiments would show greater signs of degradation, in the form of increased thermal conductivity, compared to the A1 experiments as the A2 experiments were subjected to stronger alkaline solution than the A1 experiments. However, that proved not to happen within the investigated time frame. Thus, it may seem that the VIPs degrades slowly in combination with solutions with pH less than 12.6, at room temperature.

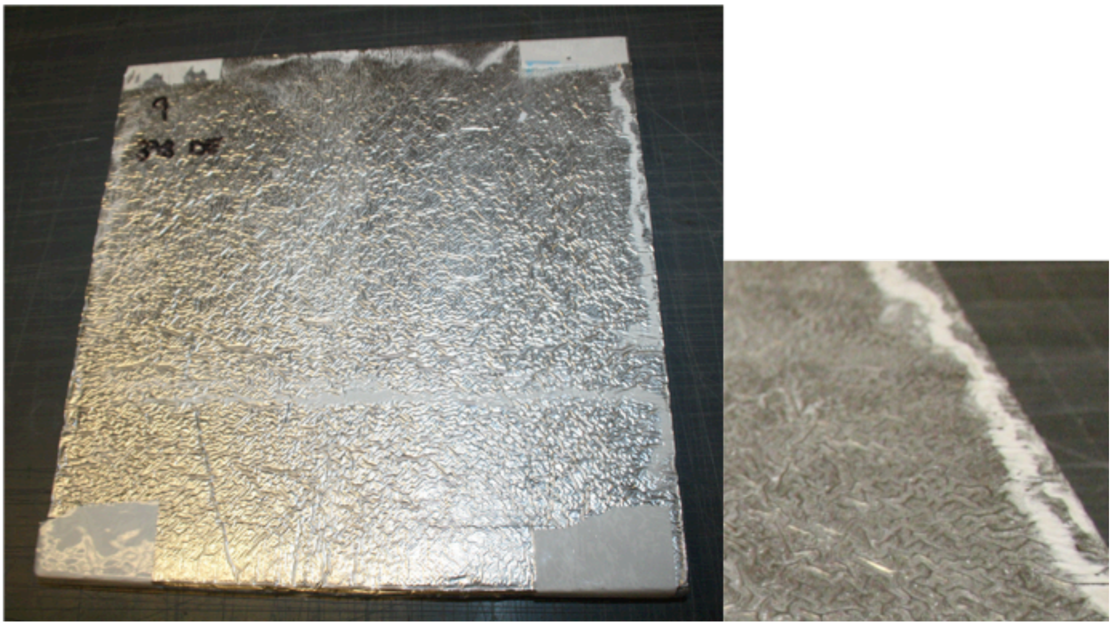

Figure 12. Experiment A2 after 9 weeks at room temperature, submerged in alkaline solution, $Ca(OH)_2$ with pH = 12.6. A thin layer of $CaCO_3$ is visible, but nevertheless no signs of degradation. Note: the white line across the panel is due to the overlying concrete, which was divided in two parts. The white line is the split between the two concrete prisms, where some $CaCO_3$ gathered. Right: A close-up of the VIP where the white layer of $CaCO_3$ is visible. An example of a non-aged VIP is shown in Figure 2.

3.1.3 Measurements on VIP in Heating Cabinet at 70°C

Experiment B1 with pH = 10.6

Dimensions

For all the VIP experiments, it was difficult to measure change in any of the three dimensions (nominal length, width and thickness). A slide calliper was used. The measured changes were considered insignificant. The thickness changes for the experiments in heating cabinet at 70°C (B1 and B2) are shown in Table 4, Table 5, Figure 15 and Figure 16. However, these changes were all within the uncertainty of the measurements, which is estimated to be around ± 0.5 mm.

Mass

The change in weight of the B1 panels developed different from the experiments at room temperature (A1 and A2). The B1 experiments showed a significant, even weight increase throughout the whole testing period. Since the weight increase with the B1 panels was much greater than the amount of water gathered in the air pockets created by the tape around the panels, most of the increase was due to vapour permeation through the VIP envelope. The dry gases N_2 and O_2 only have a small influence of 1.3 g in a fully vented panel. The results from the weight measurements for the B1 panels are summarised in Table 8, Table 9 and Figure 17.

Thermal Conductivity

The degradation of the thermal conductivity performance of the B1 experiments showed a different development from the experiments carried out at room temperature (A1 and A2). Already after three weeks one of the three B1 panels had punctured ($B1_1$). The two remaining B1 panels both showed increased thermal conductivity, one greater than the other. Further development of the two “unvented” panels showed rapid deterioration, and both of them punctured after nine weeks in the heating cabinet. The full development of the different B1 panels is found in Table 13 and Figure 19. All the three B1 experiments showed pronounced marks on the surface. Two of the three panels went almost totally white, where the fire retarder layer and possibly the Al layer(s) presumably had oxidised, see Figure 13, whereas the remaining panel had major white marks on the surface. For all the three B1 panels, the top PET layer was delaminated from the VIP surface.

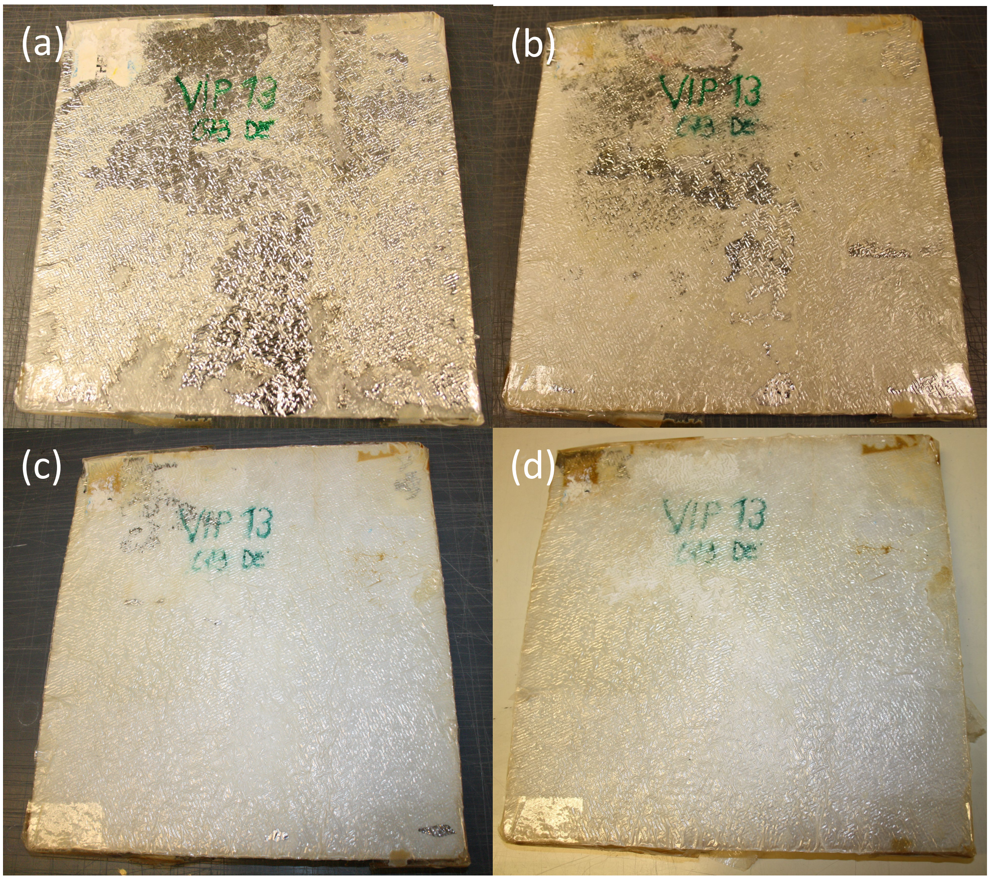

Figure 13. (a) Experiment B1 after 3 weeks in the heating cabinet at 70°C, exposed to alkaline solution with pH = 10.6 through capillary suction. Visible signs of physical degradation on the VIP envelope; the top fire retarder layer has oxidised on large surface areas. (b) B1 after 6 weeks in the heating cabinet. Only small areas with shiny Al are left. (c) B1 after 9 weeks in the heating cabinet. There are only minor spots with visible Al left on the upper left corner and the lower right corner of the VIP. (d) B1 after 12 weeks in the heating cabinet. No visible Al left, the whole surface is white. The fire retarder layer and presumably the Al layer(s) have oxidised. An example of a non-aged VIP is shown in Figure 2.

Experiment B2 with pH = 12.6

Dimensions

See the dimensions chapter under experiment B1.

Mass

Two of the three B2 panels were totally destroyed after only three weeks of testing ($B2_2$ and $B2_3$). Therefore, only one panel was continued testing throughout the last nine weeks ($B1_1$). To compensate for the destroyed panels, two new panels exposed to the same conditions as the original B2 panels were initialised ($B2_4$ and $B2_5$). These panels, denoted 'additional B2 panels', were tested weekly, throughout a testing period of totally three weeks, as it was assumed that they would puncture after only a short while.

The remaining B2 panel followed almost the same pattern as the B1 panels, and experienced a weight increase much greater than the amount of water gathered in the air pockets created by the tape around the panels throughout the testing period, as shown in Table 8, Table 9 and Figure 17. Most of the increase was due to vapour permeation through the VIP envelope as the dry gases N_2 and O_2 only have a small influence of 1.3 g in a fully vented panel.

The additional B2 panels experienced almost the same weight increase as the remaining B2 panel did during the first three weeks of the 12-week testing period, see Table 10, Table 11 and Figure 18. Also this may indicate that they follow the same pattern, and that this development is a 'normal' development of a VIP subjected to pH = 12.6 in heating cabinet at 70°C, contrary to the development of the destroyed VIPs.

Thermal Conductivity

As visualised in Table 13 and Figure 19, the remaining B2 panel experienced a significant increase in the thermal conductivity throughout the testing period, and were punctured after nine weeks. The panel also showed great signs of physical degradation, see Figure 14. As with the B1 panels, top PET layer were delaminated from the surface of the B2 panel. At the end of the testing period, no Al was visible on the surface of the B2 panel (the fire retarder layer and the Al layer(s) had presumably oxidised). During the last three weeks of the testing period the panel was slightly deformed, as shown in Figure 14.

Contrary to expectations, the additional B2 panels showed a steady development, and only a small change in thermal conductivity was measured after three weeks, as shown in Table 14 and Figure 21. Both the remaining B2 panel and the additional B2 panels obtained the same thermal conductivity value after three weeks of testing (approximately 6 mW/(mK)). This may indicate that this is a 'normal' development of a VIP panel subjected to pH = 12.6 in heating cabinet at 70°C, contrary to the development of the destroyed VIPs.

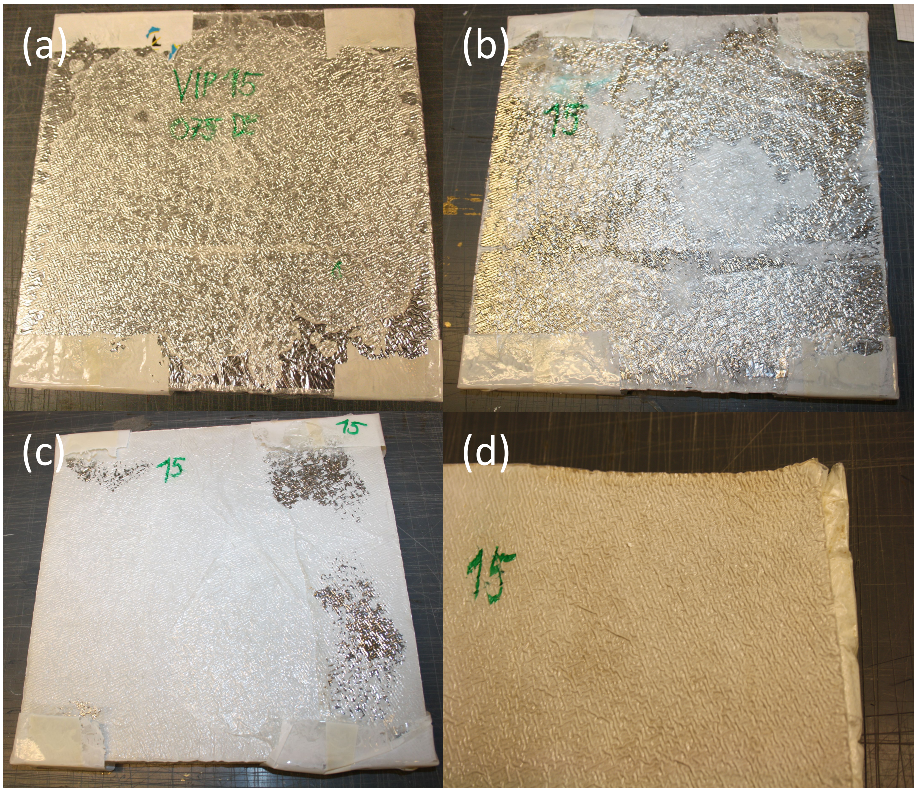

Figure 14. (a) The remaining B2 panel after 3 weeks in the heating cabinet at 70°C submerged in alkaline solution, $Ca(OH)_2$ with pH = 12.6. Visible signs of physical degradation on the VIP envelope; the top PET layer was delaminated from the surface of the VIP, and the top fire retarder layer has oxidised on large surface areas. (b) B2 after 6 weeks in the heating cabinet. The shiny Al surface has started to disappear. (c) B2 after 9 weeks in the heating cabinet. Only small areas with shiny Al left. (d) B2 after 12 weeks in the heating cabinet. No visible Al on the surface of the panel, the fire retarder layer and presumably the Al layer(s) have oxidised. An example of a non-aged VIP is shown in Figure 2.

3.2 Summary of VIP experiments

The following Table 3, Table 4, Table 5, Table 6, Table 7, Table 8, Table 9, Table 10, Table 11, Table 12, table 13 and Table 14 summarises the results from the thickness, mass and thermal conductivity measurements.

Dimensions

Table 3. Thickness (mm) versus time for the VIP experiments at room temperature, including the reference experiment.

Time aged (weeks)	$A1_1$	$A1_2$	$A1_3$	$A2_1$	$A2_2$	$A2_3$	C1
0	20.50	20.60	20.40	20.60	20.50	20.30	20.35
3	20.52	20.65	20.45	20.63	20.53	20.30	20.39
6	20.64	20.68	20.55	20.65	20.58	20.32	20.39
9	20.64	20.68	20.47	20.65	20.58	20.25	20.41
12	20.68	20.53	20.46	20.73	20.63	20.22	20.41

Table 4. Thickness (mm) versus time for the VIP experiments in the heating cabinet at 70°C.

Time aged (weeks)	$B1_1$	$B1_2$	$B1_3$	$B2_1$	$B2_2$	$B2_3$
0	20.50	20.00	20.00	20.30	20.00	20.00
3	20.56	19.94	20.08	20.34	19.00	19.00
6	20.69	19.95	20.08	20.36	-	-
9	20.63	19.95	20.08	20.36	-	-
12	20.44	19.93	20.02	20.30	-	-

Table 5. Thickness (mm) versus time for the additional B2 panels (that were started up when the $B2_2$ and $B2_3$ panels failed) in the heating cabinet at 70°C.

Time aged (weeks)	$B2_4$	$B2_5$
0	20.00	20.00
1	19.92	19.88
2	19.80	19.88
3	19.80	19.85

Mass

Table 6. Mass (g) versus time for the VIP experiments at room temperature, including the reference experiment. Note: Panels $A1_3$, $A2_3$ and C1 were punctured when opening up the tape for the panels to dry out and is therefore coloured in red.

Time aged (weeks)	$A1_1$	$A1_2$	$A1_3$	$A2_1$	$A2_2$	$A2_3$	C1
0	273.2	264.9	258.9	264.7	264.4	271.2	271.8
3	278.7	269.8	264.5	274.6	274.0	283.0	283.0
6	281.2	271.1	267.0	274.2	274.9	285.9	289.1
9	280.7	270.8	265.6	273.8	274.2	284.9	290.0
12	281.0	270.6	265.2	274.8	275.1	286.3	289.6
After drying out	*273.5*	*266.6*	*260.6*	*265.5*	*265.4*	*274.2*	*274.9*

Table 7. Weight-% versus time for the VIP experiments at room temperature, including the reference experiment. Note: The panels A1₃, A2₃ and C1 were punctured when opening up the tape for the panels to dry out and is therefore coloured in red.

Time aged (weeks)	$A1_1$	$A1_2$	$A1_3$	$A2_1$	$A2_2$	$A2_3$	C1
0	0 %	0 %	0 %	0 %	0 %	0 %	0 %
3	2.0 %	1.8 %	2.2 %	3.7 %	3.6 %	4.4 %	4.1 %
6	2.9 %	2.3 %	3.1 %	3.6 %	4.0 %	5.4 %	6.4 %
9	2.8 %	2.2 %	2.6 %	3.4 %	3.7 %	5.0 %	6.7 %
12	2.9 %	2.1 %	2.5 %	3.8 %	4.0 %	5.5 %	6.5 %
After drying out	*0.1 %*	*0.6 %*	*0.7 %*	*0.3 %*	*0.4 %*	*1.1 %*	*1.2 %*

Table 8. Mass (g) versus time for the VIP experiments in the heating cabinet at 70°C.

Time aged (weeks)	$B1_1$	$B1_2$	$B1_3$	$B2_1$	$B2_2$	$B2_3$
0	261.4	267.2	271.1	257.0	272.6	266.1
3	311.5	314.0	291.1	288.3	1038.3	996.6
6	327.9	331.9	329.2	321.3	-	-
9	334.3	350.9	367.1	320.3	-	-
12	336.8	341.3	367.5	387.8	-	-

Table 9. Weight-% versus time for the VIP experiments in the heating cabinet at 70°C.

Time aged (weeks)	$B1_1$	$B1_2$	$B1_3$	$B2_1$	$B2_2$	$B2_3$
0	0 %	0 %	0 %	0 %	0 %	0 %
3	19.2 %	17.5 %	7.4 %	12.2 %	280.9 %	274.5 %
6	25.4 %	24.2 %	21.4 %	25.0 %	-	-
9	27.9 %	31.3 %	35.4 %	24.6 %	-	-
12	28.8 %	27.8 %	35.5 %	50.9 %	-	-

Table 10. Mass (g) versus time for the additional B2 panels (that were started up when the B2₂ and B2₃ panels failed) in the heating cabinet at 70°C.

Time aged (weeks)	$B2_4$	$B2_5$
0	272.8	270.0
1	288.6	283.8
2	311.8	303.6
3	319.8	302.7

Table 11. Weight-% versus time for the additional B2 panels (that were started up when the B2₂ and B2₃ panels failed) in the heating cabinet at 70°C.

Time aged (weeks)	$B2_4$	$B2_5$
0	0 %	0 %
1	6 %	5 %
2	14 %	12 %
3	17 %	12 %

Thermal conductivity

Table 12. Thermal conductivity (mW/(mK)) versus time for the VIP experiments for the VIP experiments at room temperature, including the reference experiment.

Time aged (weeks)	A1_1	A1_2	A1_3	A2_1	A2_2	A2_3	C1
0	5.170	4.825	4.965	4.941	5.121	4.969	4.940
3	5.210	4.973	5.148	5.059	5.177	5.003	5.004
6	5.234	5.000	5.147	5.028	5.149	5.000	4.998
9	5.235	4.965	5.143	5.082	5.175	5.026	5.025
12	5.302	5.058	5.332	5.058	5.152	4.970	5.001

Table 13. Thermal conductivity (mW/(mK)) versus time for the VIP experiments in the heating cabinet at 70°C. Note: The grey indicates that the panel has punctured.

Time aged (weeks)	B1_1	B1_2	B1_3	B2_1	B2_2	B2_3
0	4.818	4.788	4.849	4.701	4.970	4.877
3	25.802	10.146	6.257	6.236	-	-
6	28.799	13.513	16.856	13.273	-	-
9	27.594	24.518	20.827	21.652	-	-
12	30.115	27.100	33.646	45.322	-	-

Table 14. Thermal conductivity (mW/(mK)) versus time for the additional B2 panels (that were started up when the B2_2 and B2_3 panels failed) in the heating cabinet at 70°C.

Time aged (weeks)	B2_4	B2_5
0	4.649	4.501
1	5.083	4.966
2	5.519	5.522
3	6.048	5.959

The following Figure 15, Figure 16 and Figure 17, Figure 18, Figure 19, Figure 20 and Figure 21 visualises the various relationships in the tables above.

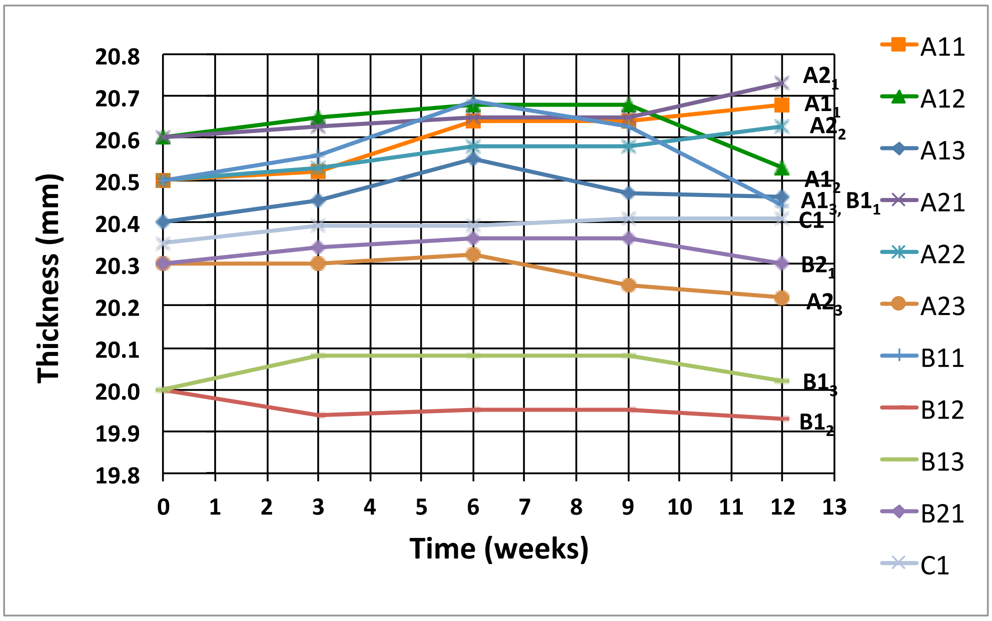

Figure 15. Thickness (mm) versus time for the VIP experiments exposed to various degradation procedures.

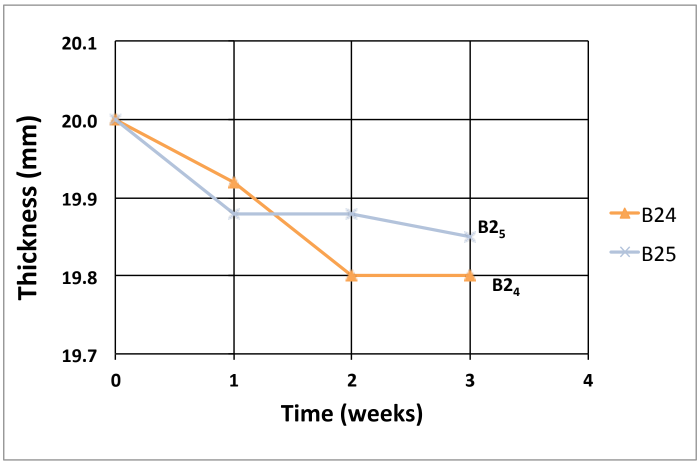

Figure 16. Thickness (mm) versus time for the additional B2 (that were started up when the B2$_2$ and B2$_3$ panels failed) panels in the heating cabinet at 70°C.

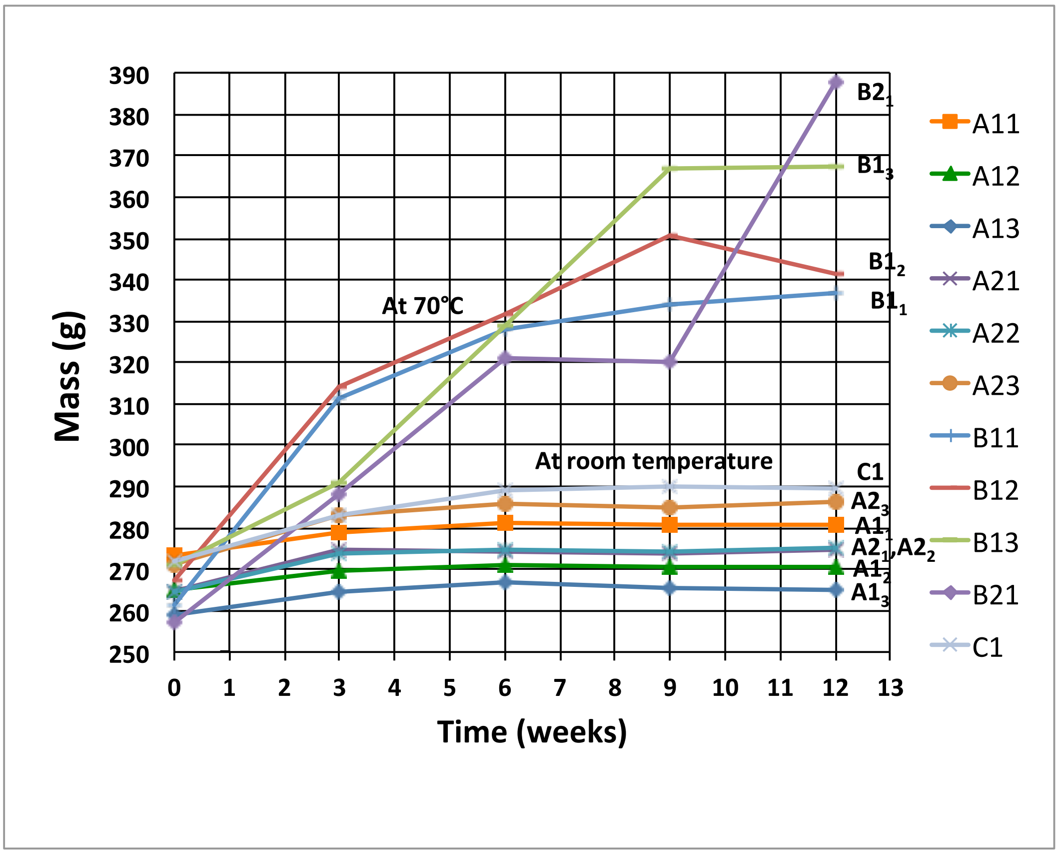

Figure 17. Mass (g) versus time for the VIP experiments exposed to various degradation procedures. There is an obvious distinction between the A and C experiments at room temperature and the B experiments in the heating cabinet at 70°C.

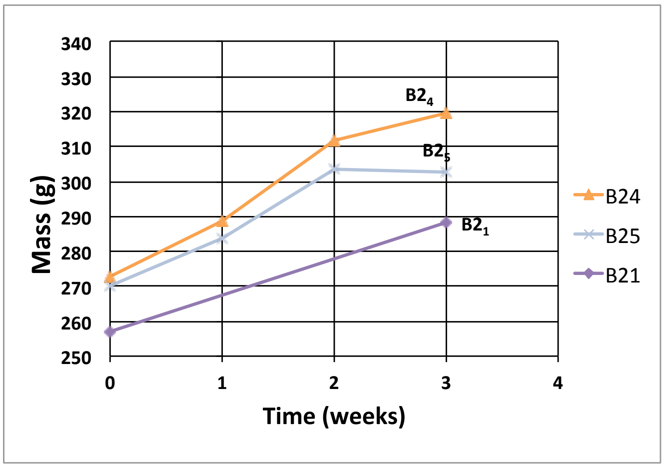

Figure 18. Mass (g) versus time for the additional B2 panels (that were started up when the B2$_2$ and B2$_3$ panels failed) in the heating cabinet at 70°C, including B2$_1$ for comparison.

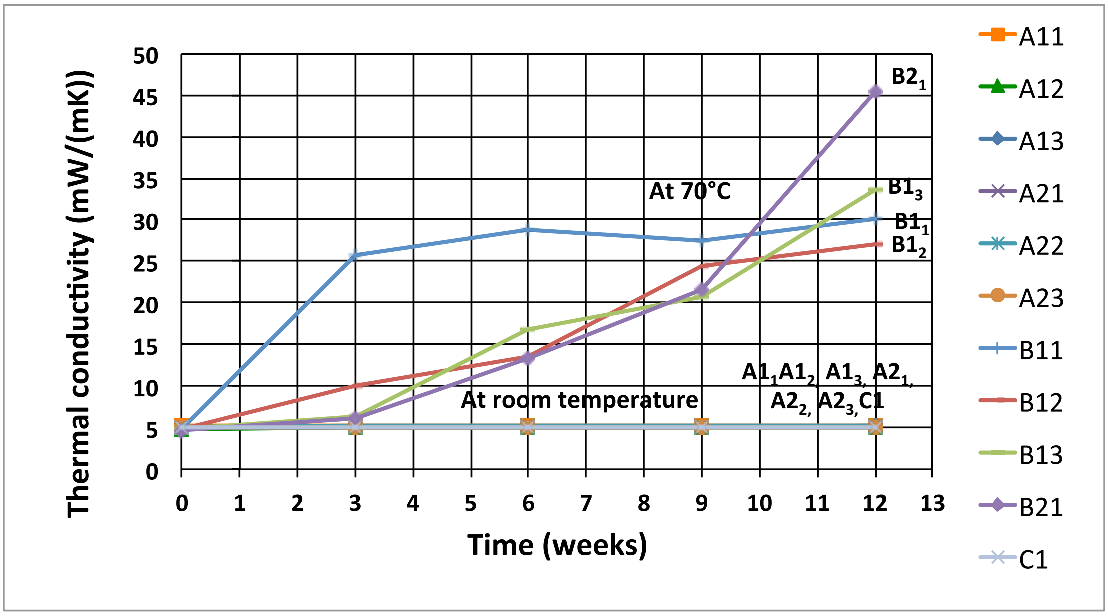

Figure 19. Thermal conductivity (mW/(mK)) versus time for the VIP experiments exposed to various degradation procedures. There is an obvious distinction between the A and C experiments at room temperature and the B experiments in the heating cabinet at 70°C.

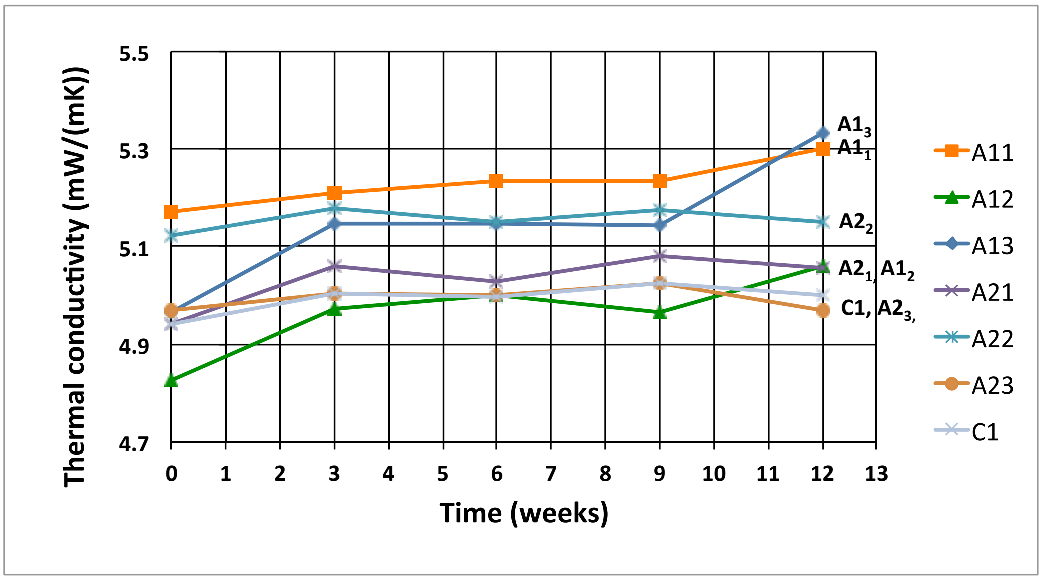

Figure 20. Thermal conductivity (mW/(mK)) versus time for the A experiments at room temperature and the VIP reference experiment, C1.

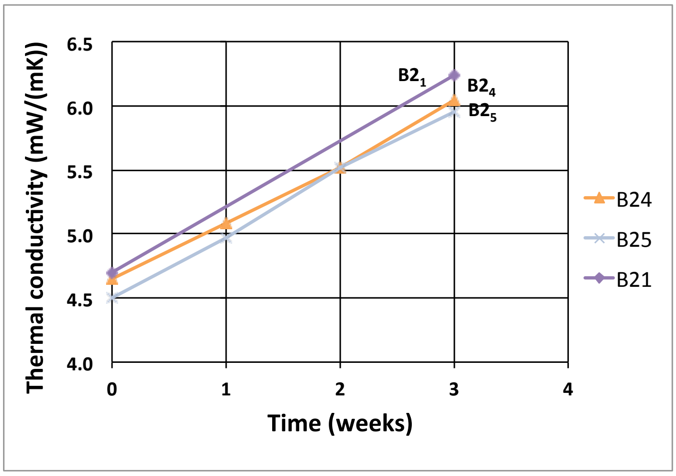

Figure 21. Thermal conductivity (mW/(mK)) versus time for the additional B2 panels (that were started up when the $B2_2$ and $B2_3$ panels failed) in the heating cabinet at 70°C, including $B2_1$ for comparison.

Aluminium is not stable in any alkaline environment, but it was however assumed that the alkaline solution, (e.g. $Ca(OH)_2$ aqueous solution) with a pH-value of about 12.6 would degrade the VIPs to a greater extent than what the natural concrete water with a pH-value of 10.6 would do. Nevertheless, comparing the two different A experiments (A1 with pH = 10.6 and A2 with pH = 12.6) and the two different B experiment (B1 with pH = 10.6 and B2 with pH = 12.6) with each other, this proved not to happen after 12 weeks of testing, see Figure 19.

It is obvious that there is a distinction between the results from the panels aged at room temperature (A experiments) and the panels aged in the heating cabinet at 70°C (B experiments). The increase in thermal conductivity measured for the A experiments was relatively low compared to B experiments, and the overall performance of the A experiments was better than first expected. Thus, it may seem that the temperature have the greatest influence on the degradation of the thermal conductivity performance of the

VIPs, not the pH-value of the solution alone. This is an indication that the different alkaline solutions may not be an as hard exposure to the VIPs as first assumed.

It should be noted that exposing the VIPs to 70°C represents an extreme condition, and is regarded as a worst-case scenario. This scenario was investigated identify how sensitive the VIPs were outside ideal conditions. The VIPs may however reach such a high temperature in especially unfavourable cases, e.g. on dark coloured building façades heated through solar radiation, where e.g. the drainage channels are blocked.

3.3 Degradation of VIP Envelope Specimen in Alkaline Environment

The following collages of photos (Figure 22 and Figure 23) show the degradation of a VIP envelope specimen throughout a testing period of nine weeks. According to previous reports (Brunner et al. 2006), there are three polyurethane (PU) layers sandwiched between the polyethylene terephthalate (PET) and Al layers. However, the (PU) layers were not visible during the SEM analysis, see Figure 22. Moreover, there is a PET layer that is the outmost layer to protect the nearby fire retarder layer. This layer was neither observed during the SEM analysis. A closer look at the energy dispersive spectrum (EDS) mapping (not shown here) and the cross section photos indicates that there is possible delamination between the VIP envelope layers, as shown with arrows in Figure 24 (VIP envelope specimen that were aged for 6 weeks).

As depicted in Figure 1(b) and Figure 25 the VIP envelope is built up of three Al layers in between the organic material layers (such as PET, PU and PE). The metallic Al layers may act as main barriers for N_2 and O_2, and H_2O (Brunner and Simmler 2008) and are critical for the durability of the VIP envelope. The use of Al in the VIP envelope is due to its easy preparation and low cost as well as the highly developed area of application, such as with food packaging. However, the stability of these metal layers is questionable during the very long service life that is requested for some building applications such as concrete and lightweight concrete elements, especially when exposed to alkaline environment, as shown in Figure 22 and Figure 23. Figure 22 indicates an obvious shrinkage of the total envelope thickness, from 138 µm for the non-aged sample to 107 µm after 3 weeks ageing. It was reduced further to 87 µm after nine weeks ageing (see Table 15). The fire retarder layer (Figure 22 (a) and Figure 23(a)) had totally disappeared after 3 weeks ageing in the alkaline solution. This indicates that, firstly, the fire retarder layer due to its reactivity in both acidic and alkaline environment (see Equation (1) and (2)) may not be the most suitable material for VIP envelopes in acidic and alkaline environment. Secondly, the outmost PET layer, which protects the fire retarder layer, should be improved to protect the metallic layers better.

$$Al(OH)_3 \rightarrow Al^{3+} + 3OH^- \qquad (1)$$

$$Al(OH)_3 \rightarrow Al(OH)_4^- + H^+ \qquad (2)$$

As shown in Table 15, the distance between the three Al layers changed dramatically during the nine weeks testing period, clearly indicating degradation of the organic material layers in the VIP envelope. A comparison between the initial sample and the 9 weeks aged sample suggests that the organic layers were not very stable. The distance between the three Al layers was clearly reduced from 46 µm for the non-aged sample to 31 µm for the 9 weeks aged sample. Thus, the degrading of the organic material layers had a great influence on the reduction of the total thickness of the VIP envelope.

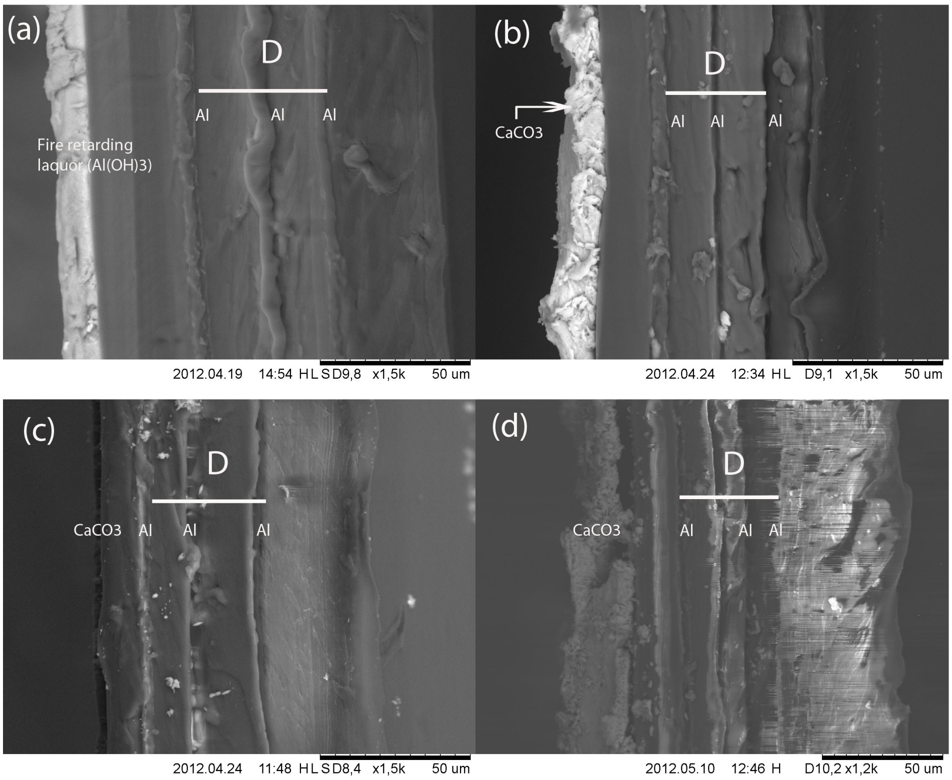

Figure 22. SEM cross-sections of all the VIP envelope specimens (a) non-aged, (b) 3 weeks, (c) 6 weeks, (d) 9 weeks. D is the distance between the three Al layers of the VIP envelope. The thickness of the Al layers is 0.1 µm, and they are therefore difficult to spot. Note: the artificial area on (d) is due to the surface charging of the organic layer.

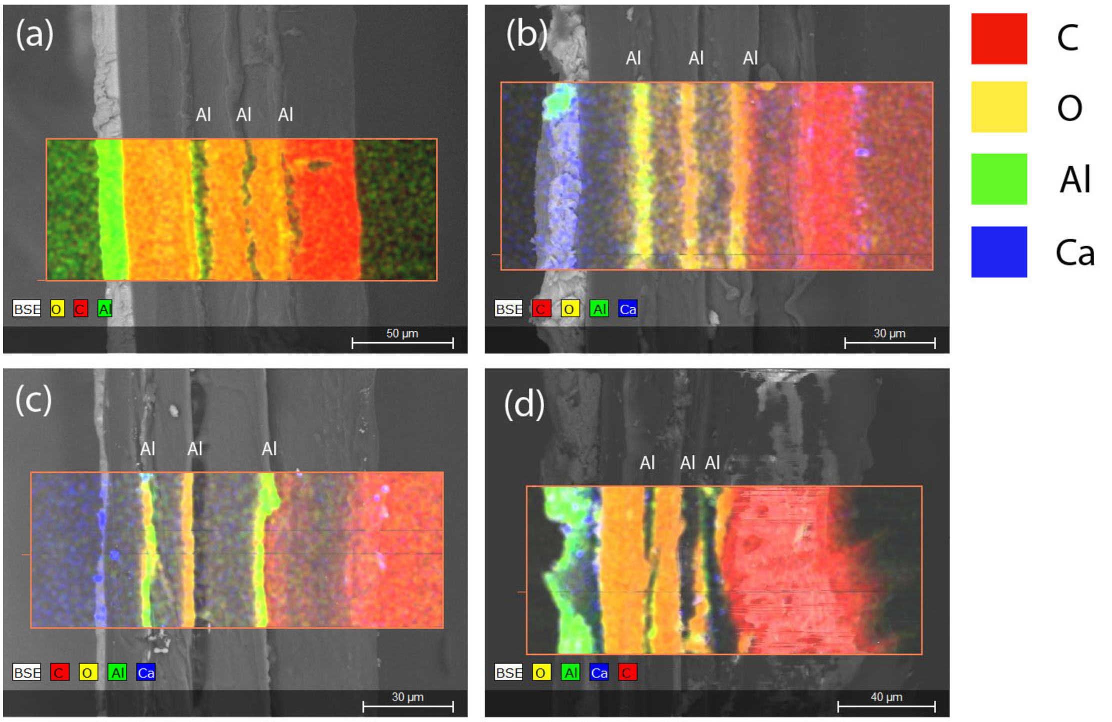

Figure 23. Area mapping for all the VIP envelope specimens exposed to alkaline solution, $Ca(OH)_2$ with pH = 12.5. (a) non-aged, (b) 3 weeks, (c) 6 weeks, (d) 9 weeks. Note: Different scale for the different photos (50 µm, 30 µm and 40 µm). The outmost 'Al-layer' in photo (d) is attributed to Al oxide/hydroxide formed during the experiment. In both (c) and (d) the second Al layer is hidden in the delamination between the metallised layers and the organic layers.

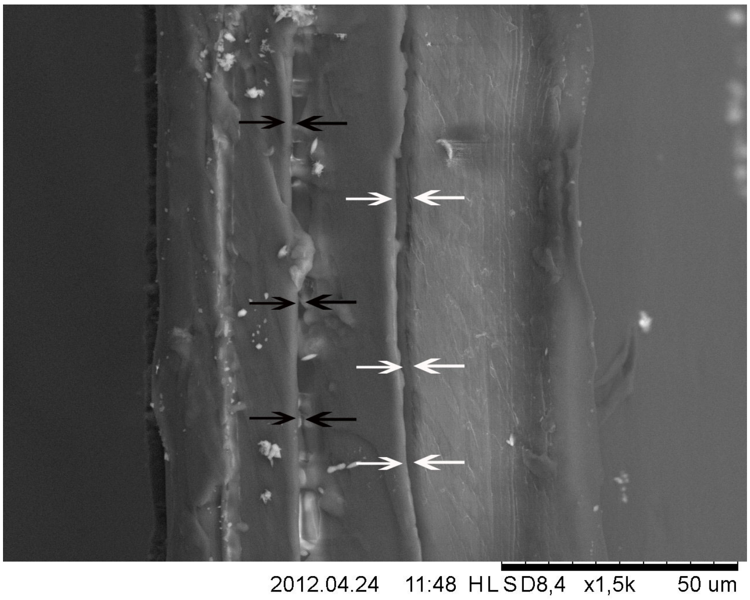

Figure 24. SEM cross-section of the 6 weeks aged sample, showing possible delamination between the organic material layers.

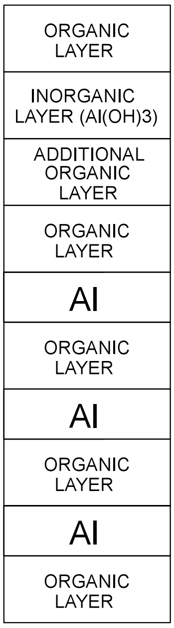

Figure 25. An overall configuration of the VIP laminate used in this work.

Table 15. Visualising the reduction of the total thickness and the distance between the three Al layers of the VIP laminate.

Weeks in alkaline environment	Total thickness of laminate (µm)	Distance between the three Al layers (µm)
0	138	46
3	107	36
6	92	33
9	87	31

3.4 Transport of Hydroxide

In order to evaluate the degradation process of VIPs, an experiment concentrating on a possible transport of OH^- through air was carried out. After one week distilling on 96°C, the results showed that the pH-value of the distilled $Ca(OH)_2$ was 8.5 ± 0.1. Compared with the pH-value of distilled water, which was also measured at the same time; pH = 8.5 ± 0.1, it seems obvious that no OH^- was transported through air. The only thing that may defy this theory is that the *possible* OH^- that was *possibly* transported through air was neutralised by the CO_2 in the air. Even though the distillation system was closed, there was still some CO_2 in the pipe. Thus, the neutralisation process may possibly have occurred during the distillation process.

4 Conclusions

A laboratory study on the degradation of vacuum insulation panels (VIPs) in different alkaline solutions at different temperatures has been performed throughout a testing period of 12 weeks. Various experiments have been carried out to represent the situation of applying VIPs in concrete and lightweight concrete elements. A laboratory study of the degradation of the VIP envelope in alkaline environment in particular have also been performed in order to obtain a better understanding of the ageing process of the whole VIP panel. The testing period of the VIP envelope specimen experiment lasted for nine weeks.

Results and measurements from the VIP experiments showed that the VIPs at room temperature performed well, and did not show any significant signs of degradation during 12 weeks of exposure. However, the degradation effect was evident for the VIPs in the heating cabinet at 70°C. These panels showed a rapid increase in thermal conductivity, and they were all punctured after nine weeks. Also visible signs of degradation were evident on the VIPs in the heating cabinet. The top polyethylene terephthalate (PET) layer was delaminated from the VIP surface after only three weeks, and throughout the end of the 12-week testing period only small spots of aluminium was still visible.

The pH-value of the solutions that the VIPs were subjected to proved not to be the hardest strain for the VIPs, as first assumed, when the two experiments at room temperature, subjected to different alkaline solutions (pH = 10.6 and pH = 12.6) performed almost the same. Thus, it may seem that the elevated temperature has the greatest influence on the durability of the VIPs, and that the VIPs degrade slowly in in combination with alkaline solutions with pH-values less than 12.6 at temperature around 20°C.

However, interesting results were also obtained from the VIP envelope specimen experiment where the outmost fire retarder layer with aluminium in the form of aluminium hydroxide, exposed to an alkaline environment, dissolved after only a short time. This behaviour of the fire retarder layer in alkaline environment may indicate that this material may not be the most suitable material for VIP envelopes in acidic and alkaline environment. From the SEM analysis it was evident that the total thickness of the VIP envelope was strongly reduced after nine weeks of testing, where firstly the top fire retarder layer dissolved, and secondly the organic material layers were degraded. The thickness of the organic material layers had, in particular, been clearly reduced.

Acknowledgements

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APPENDIX A – Heat Flow Meter Measurements

The heat flow meter (HFM) apparatus was used to measure the thermal conductivity of the panels during the testing period. The HFM apparatus is produced by SINTEF Building and Infrastructure, and is depicted in Figure A1. The measurements were performed in accordance with NS-EN 12667 (2001) and ISO 8301 (1991).

Figure A1. The heat flow meter apparatus.

The principle of the HFM apparatus is to establish a unidirectional, constant and uniform density of heat flow rate through the VIP by using heating and cooling plates, above and below the specimen respectively (NS-EN 12667, 2001). A constant distance between the heating and cooling plates was used throughout the 12-week testing period, when the change of the thickness of the VIP panels proved to be insignificant.

When the heat flow rate, q (W), and the metering area, A (m^2), is measured, the density of the heat flow rate, ϕ (W/m^2) may be calculated. And further, when the temperature difference, ΔT (K) is known, the thermal resistance, R (m^2K/W) may be calculated. When the thickness of the VIP specimens is measured, the *equivalent* thermal conductivity, λ_{eq} (W/(mK)), can finally be calculated. The equivalent thermal conductivity allows for inhomogeneity of the specimen.

Apparatus and Setup

There are several possible configurations of the HFM apparatus, and the one used in this study is a single-specimen symmetrical configuration, according ISO 8301 (1991). Heat flow meters were used to reach a desirable temperature on each side of the test specimen. Due to the limited size of the VIPs used in the experiment (250 mm x 250 mm), a customised setup for smaller test pieces was used. The HFMs used were of the type PU43T 0168 and 0169, made of 1 mm thick, flexible, circular plate of polyurethane, with an outer diameter of 100 mm and a circular measuring area of 50 mm on each side of the specimen. The setup is depicted in Figure A2.

Figure A2. The customised setup for smaller test pieces. The heat flow meter is the black, circular plate in the middle of the frame.

APPENDIX B – Distillation Apparatus Experiments

To evaluate a possible transport of hydroxide (OH^-) through air, a distillation experiment was carried out. A distillation apparatus was used to evaporate the alkaline water, as shown in Figure B1. The bottle with alkaline water was placed in a bowl of silicone oil and heated up to a temperature of 96°C. From the bottle of alkaline water, the solution was evaporated through a pipeline, which was cooled by running water. In the other end of the distillation apparatus (see Figure B2), the solution was investigated by using a pH-meter.

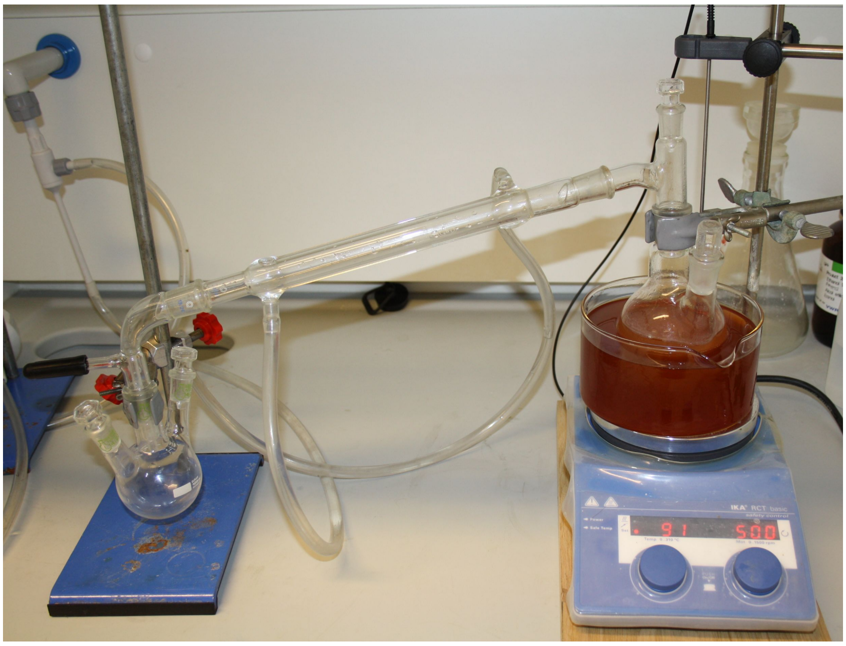


Figure B1. Distillation apparatus used in the transport of OH^-experiment.

Figure B2. A close-up photo of the condensation.

APPENDIX C – Scanning Electron Microscope

Area mapping

The following Figure C1, Figure C2, Figure C3 and Figure C4 shows the area mapping of the VIP envelope specimen after respectively 0 weeks, 3 weeks, 6 weeks and 9 weeks in alkaline environment, 0.03M $Ca(OH)_2$, with pH = 12.6.

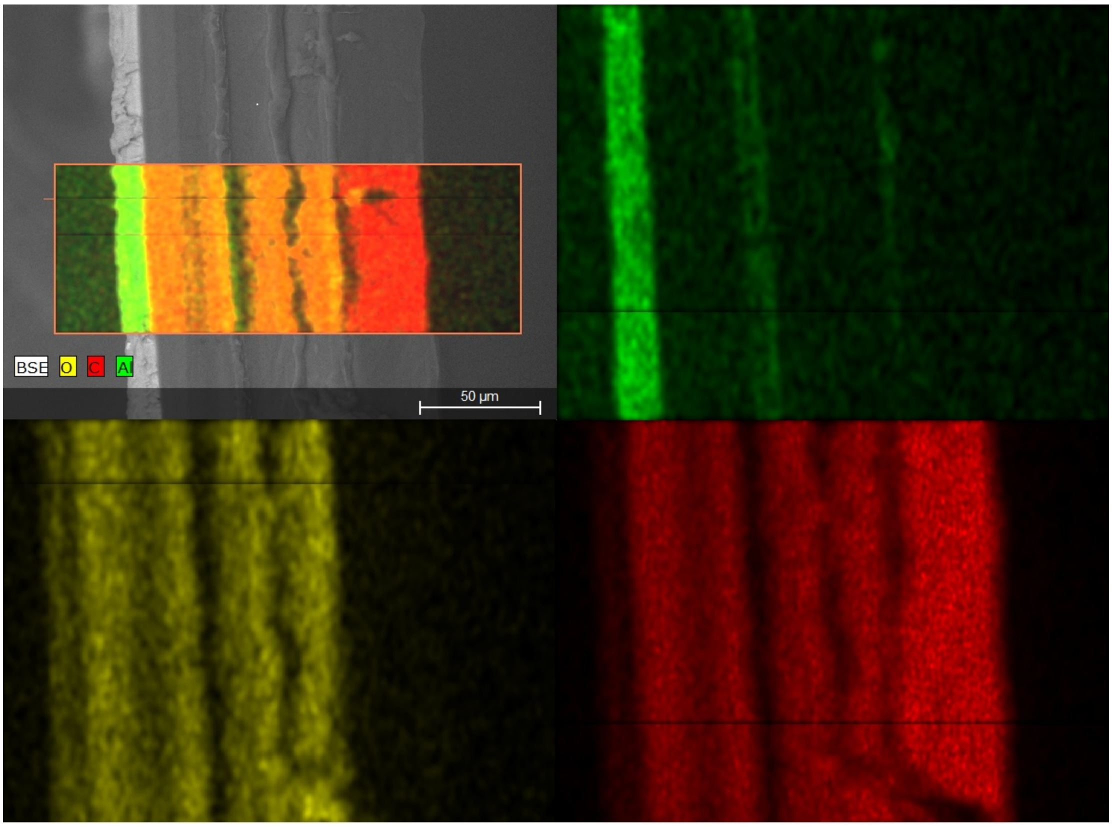


Figure C1. EDS mapping of the non-aged VIP envelope specimen shows three layers of Al. The outer layer on the area mapping of Al is the fire retarder layer that consists of aluminium hydroxide ($Al(OH)_3$). The third Al layer was not visible on the area mapping of Al.

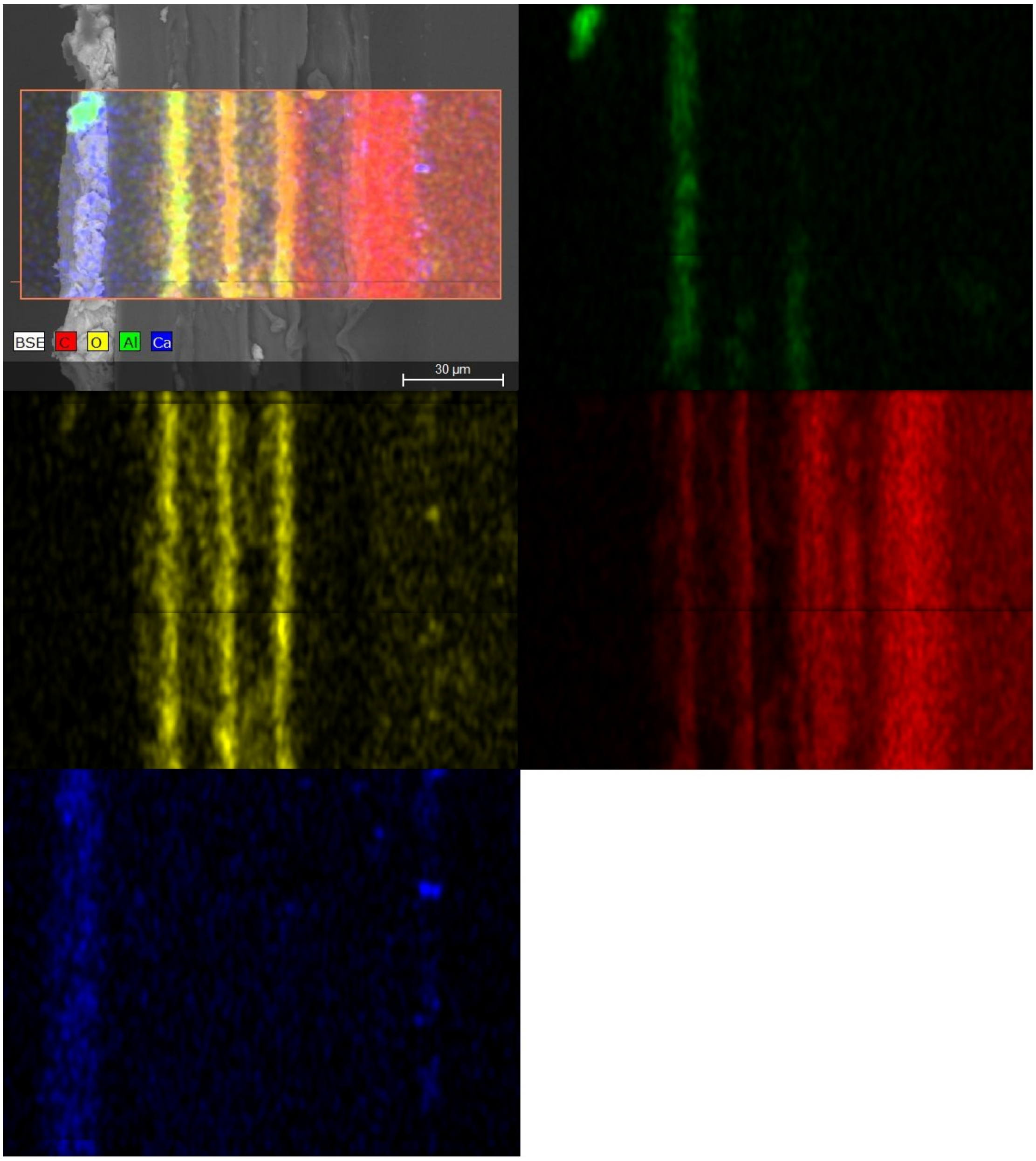


Figure C2. EDS mapping of the 3 weeks aged VIP envelope sample shows that the outer fire retarder layer has dissolved. The three Al layers are visible. However, the middle Al layer appears very faintly. A layer of calcium carbonate has emerged from the $Ca(OH)_2$ solution. The total thickness of the VIP envelope has also strongly been reduced during three weeks in alkaline environment (from 138 µm to 107 µm). The calcium carbonate has followed the razor cut, when cutting the sample, and contaminated the cross-section.

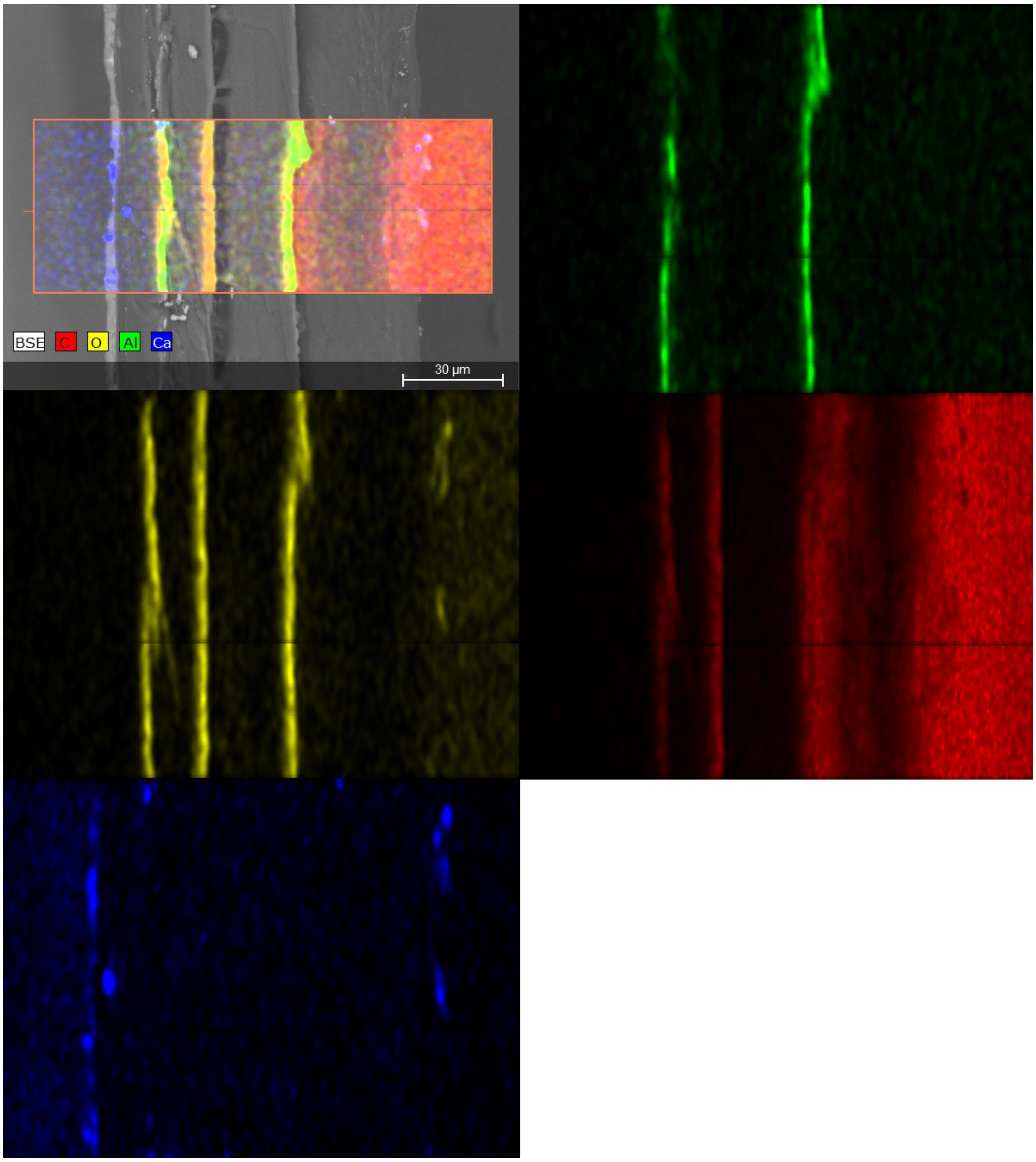


Figure C3. EDS mapping of the 6 weeks aged VIP envelope specimen sample. Three Al layers are still evident, but the middle Al layer still appears very faintly. The total thickness of the laminate has been reduced further from the 3 weeks aged sample (from 107 µm to 92 µm).

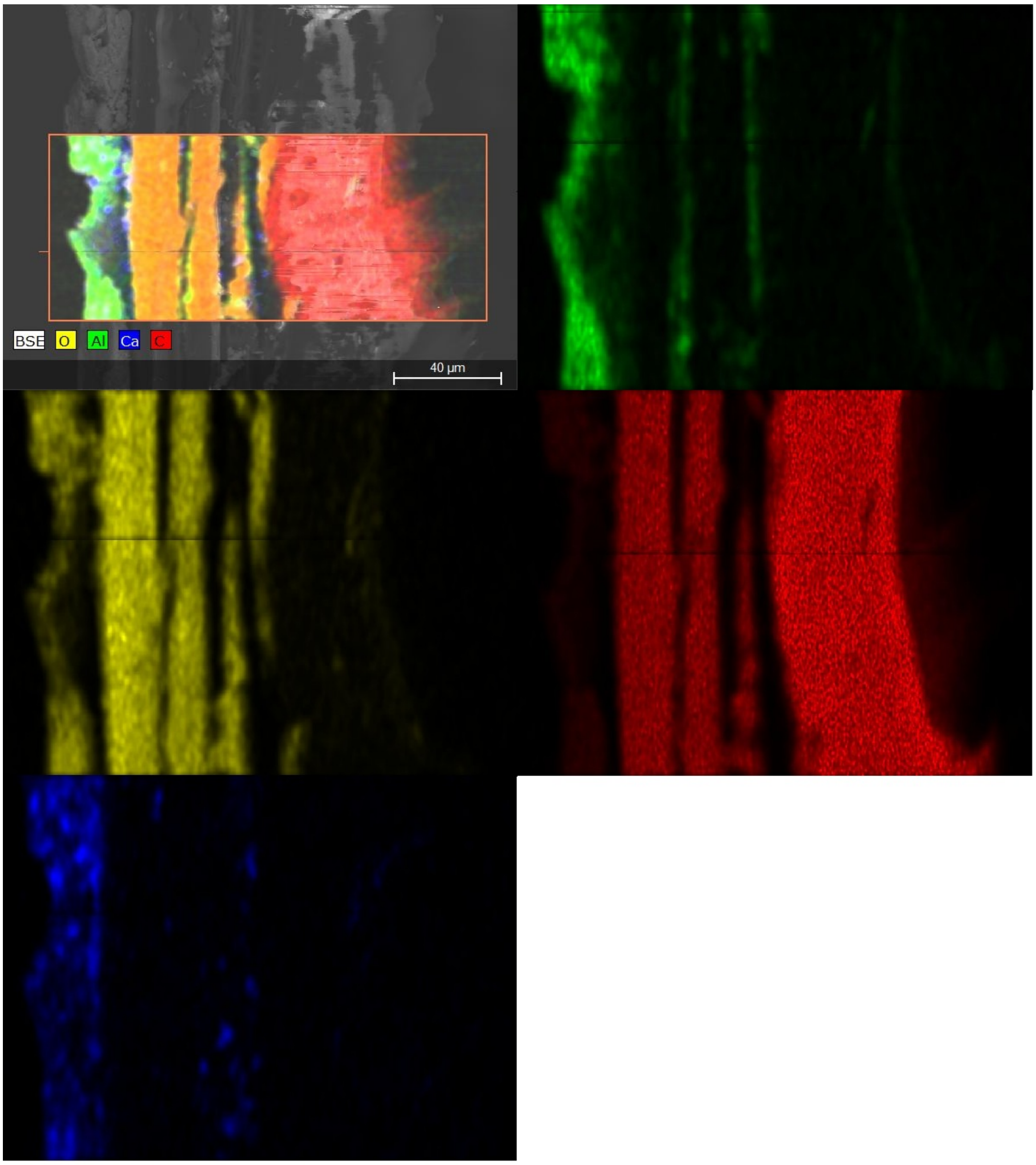


Figure C4. EDS mapping of the 9 weeks aged VIP envelope specimen sample. The outmost, thick layer on the area mapping of Al is attributed to Al oxide/hydroxide formed during the experiment. Two thin Al layers are still evident. The second Al layer is hidden in the delamination between the metallised layers and the organic layers. The total thickness of the laminate has been reduced further from the 6 weeks aged sample (from 92 µm to 87 µm).

Spectrum (EDS)

The following Figure C5 and Figure C6 show the energy dispersive spectrum EDS of the elements found on the initial VIP envelope specimen and the 6 weeks aged VIP envelope specimen respectively. Atoms with low number are less well detected. Atoms below Boron (B), such as Hydrogen (H), are not detected at all.

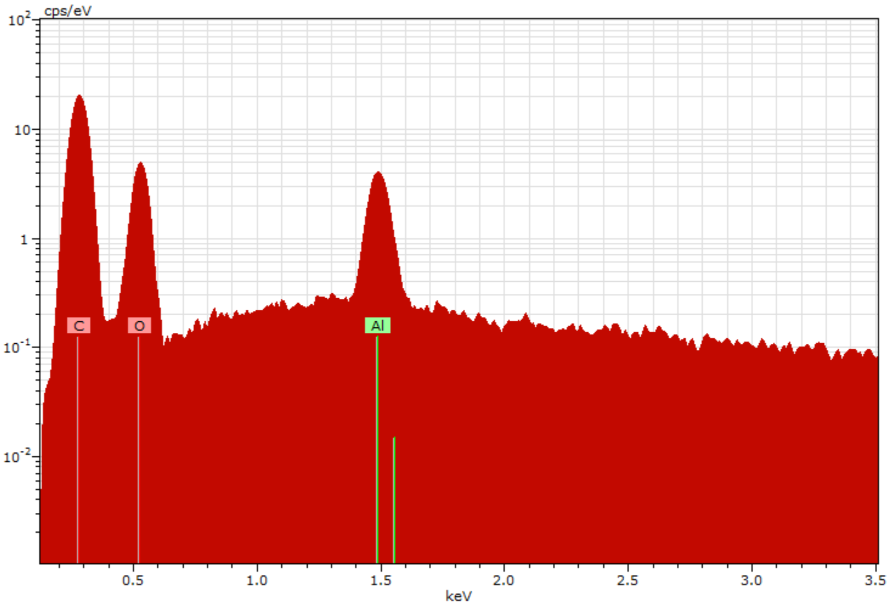


Figure C5. Elements found on the initial VIP envelope specimen.

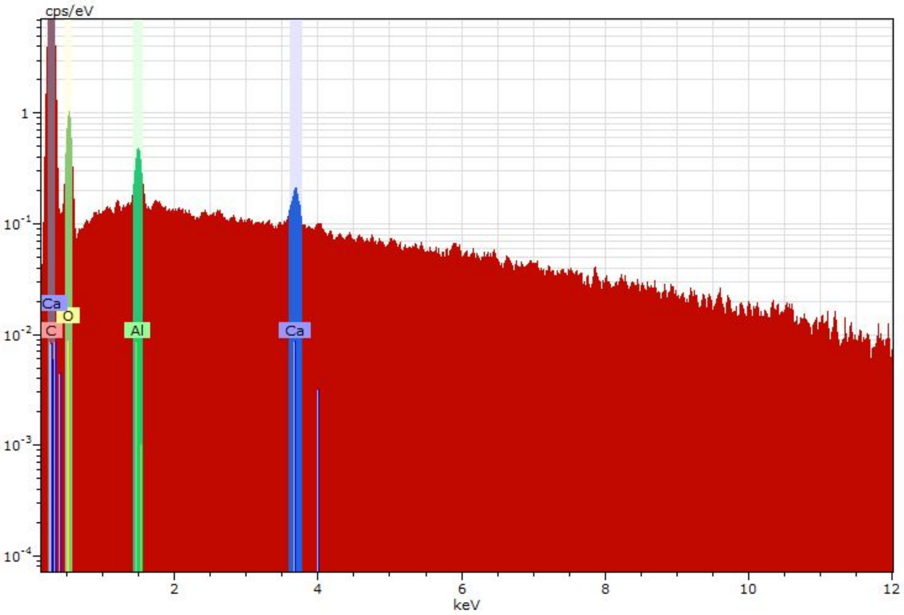


Figure C6. Elements found on the 6 weeks aged VIP envelope specimen. The Ca is from the thin film of $CaCO_3$ on the surface of the envelope.

APPENDIX D - Presentation

Lunch lecture at SINTEF Building and Infrastructure 15.05.12

(In Norwegian)

Alkalisk aldring av VIP

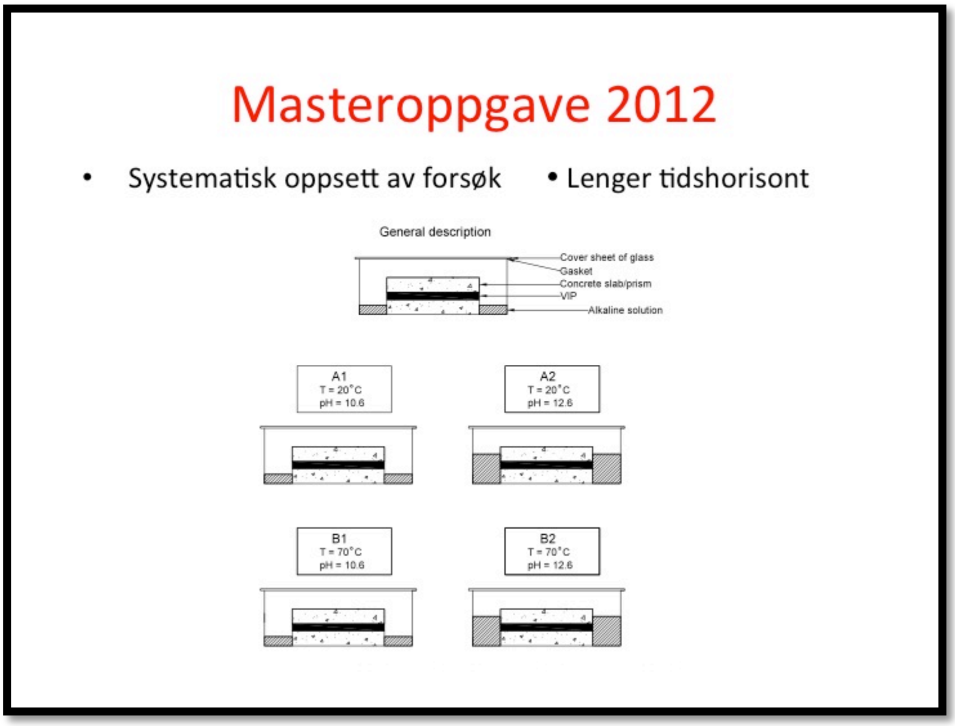
"Durability of Vacuum Insulation Panels in Alkaline Environment"

Inspirasjon til oppgaven

Prosjektoppgave

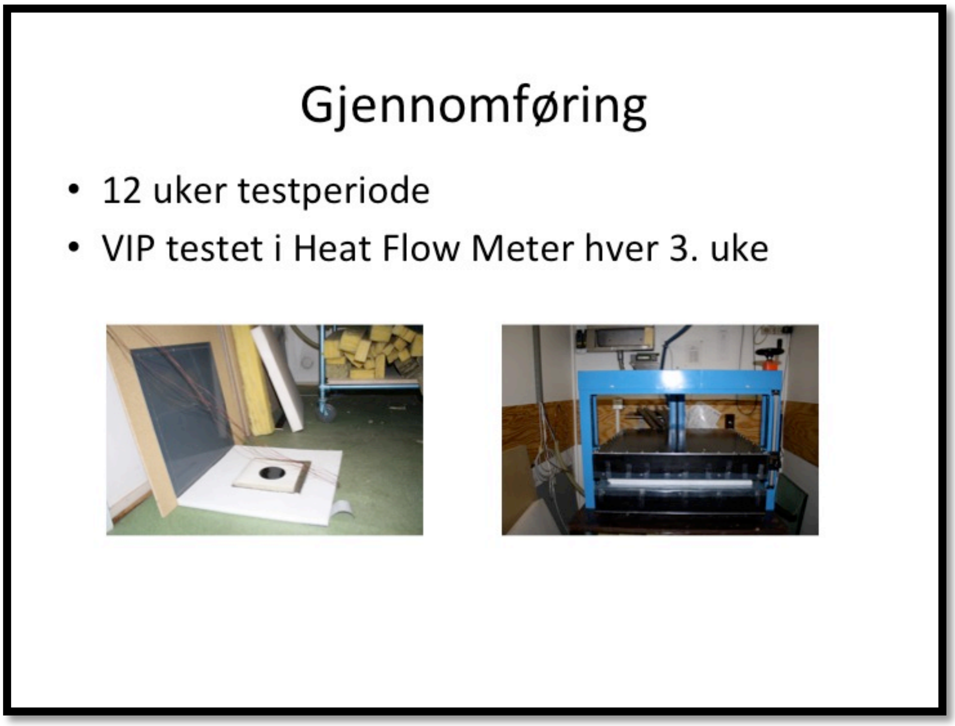
Før jul

- Antatt rask ødeleggelse av VIP i alkalisk løsning på bakgrunn av observasjoner og erfaringsutveksling som var gjort på en messe i Sveits. Kun noen dager i romtemperatur?
- Dette viste seg å være feil. Panelene vi satte i gang før jul viste seg å være relativt bestandige ift først antatt.



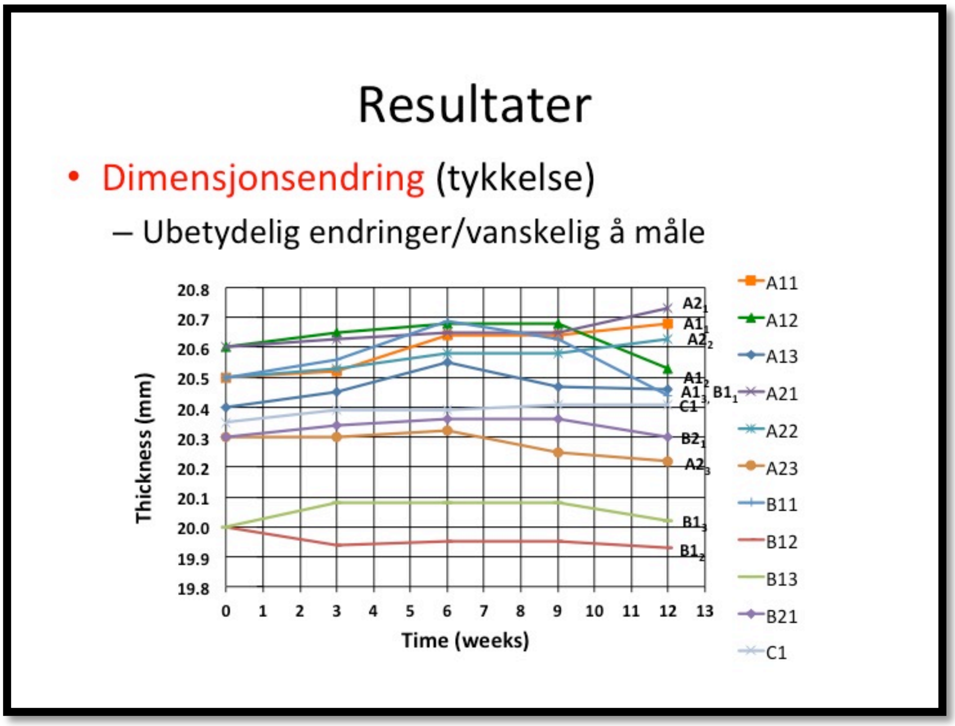
Masteroppgave 2012

- Systematisk oppsett av forsøk
- Lenger tidshorisont



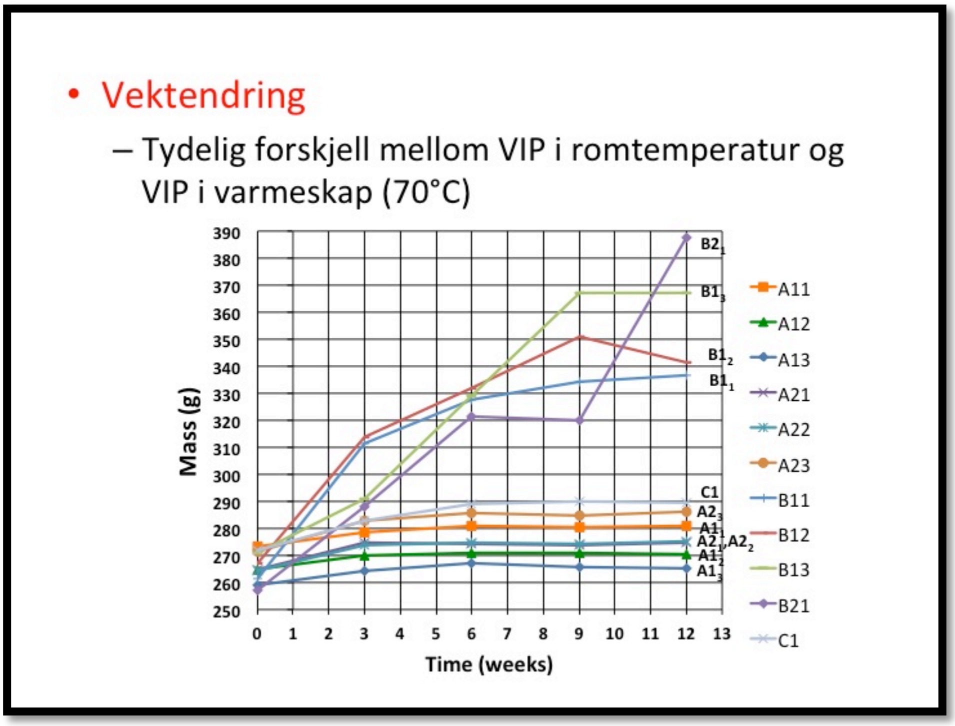
Gjennomføring

- 12 uker testperiode
- VIP testet i Heat Flow Meter hver 3. uke

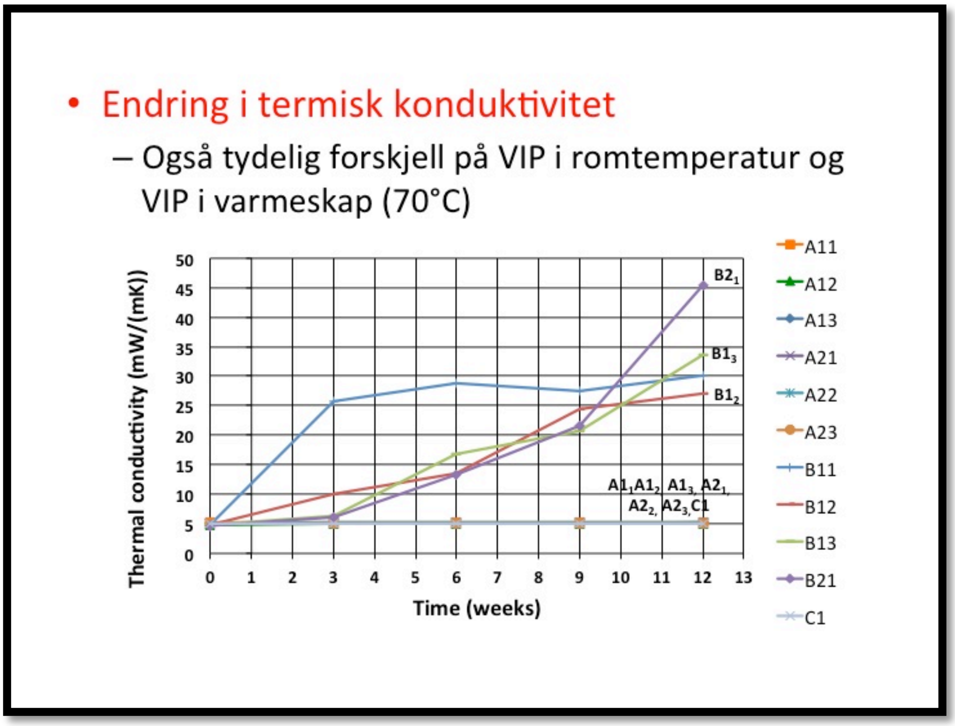


Resultater

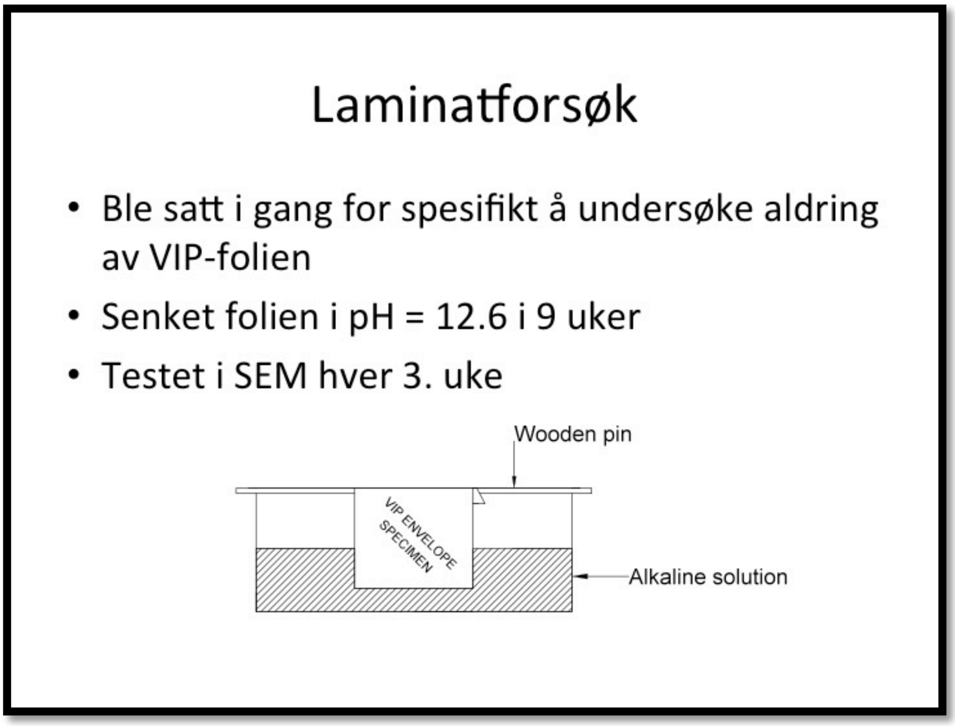
- Dimensjonsendring (tykkelse)
  - Ubetydelig endringer/vanskelig å måle



- Vektendring
  - Tydelig forskjell mellom VIP i romtemperatur og VIP i varmeskap (70°C)

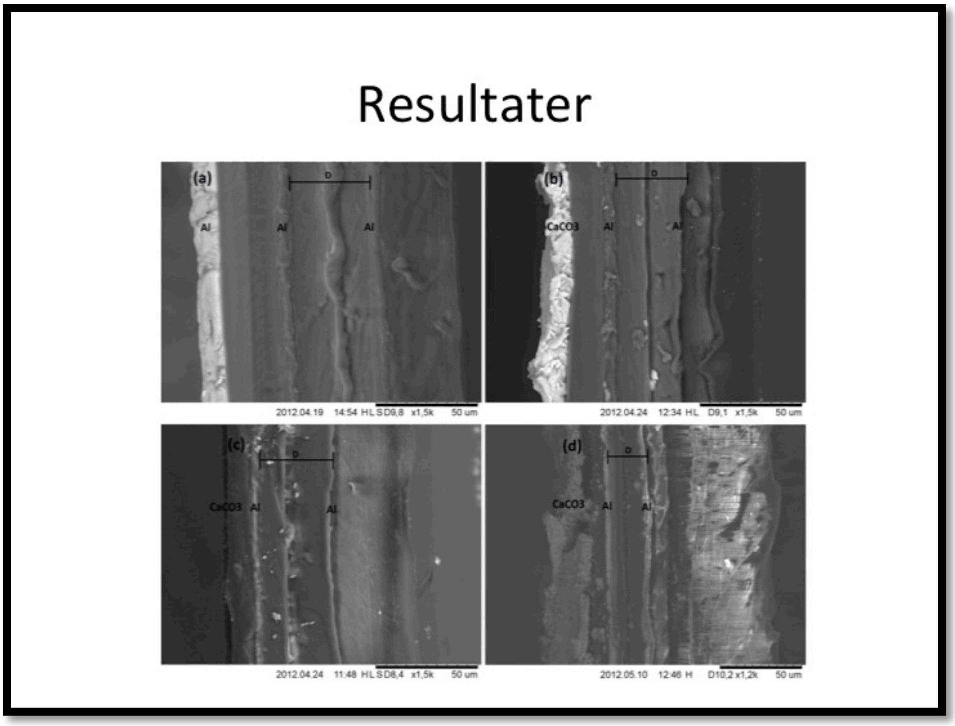


- Endring i termisk konduktivitet
  - Også tydelig forskjell på VIP i romtemperatur og VIP i varmeskap (70°C)

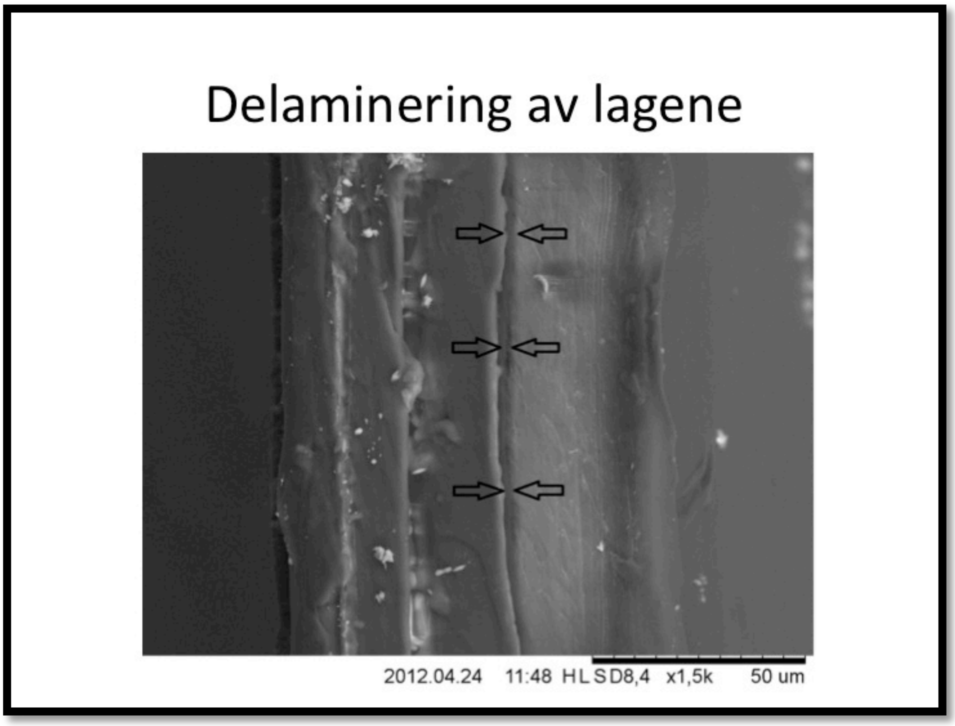


Laminatforsøk

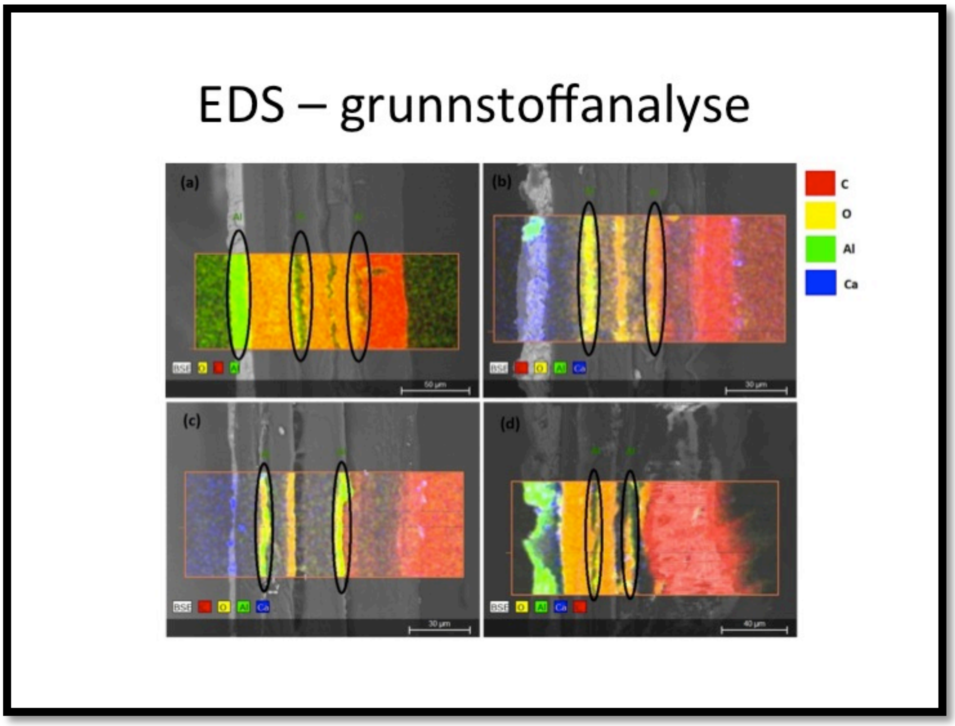
- Ble satt i gang for spesifikt å undersøke aldring av VIP-folien
- Senket folien i pH = 12.6 i 9 uker
- Testet i SEM hver 3. uke



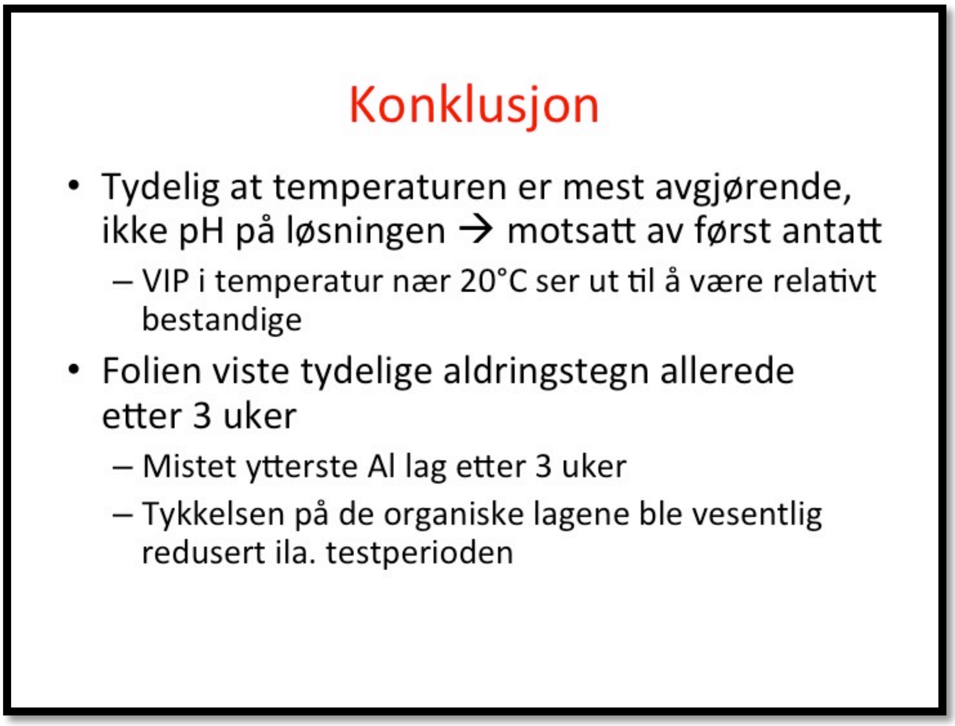
Resultater



Delaminering av lagene



EDS – grunnstoffanalyse



Konklusjon

- Tydelig at temperaturen er mest avgjørende, ikke pH på løsningen → motsatt av først antatt
  - VIP i temperatur nær 20°C ser ut til å være relativt bestandige
- Folien viste tydelige aldringstegn allerede etter 3 uker
  - Mistet ytterste Al lag etter 3 uker
  - Tykkelsen på de organiske lagene ble vesentlig redusert ila. testperioden