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RESEARCH REPORT

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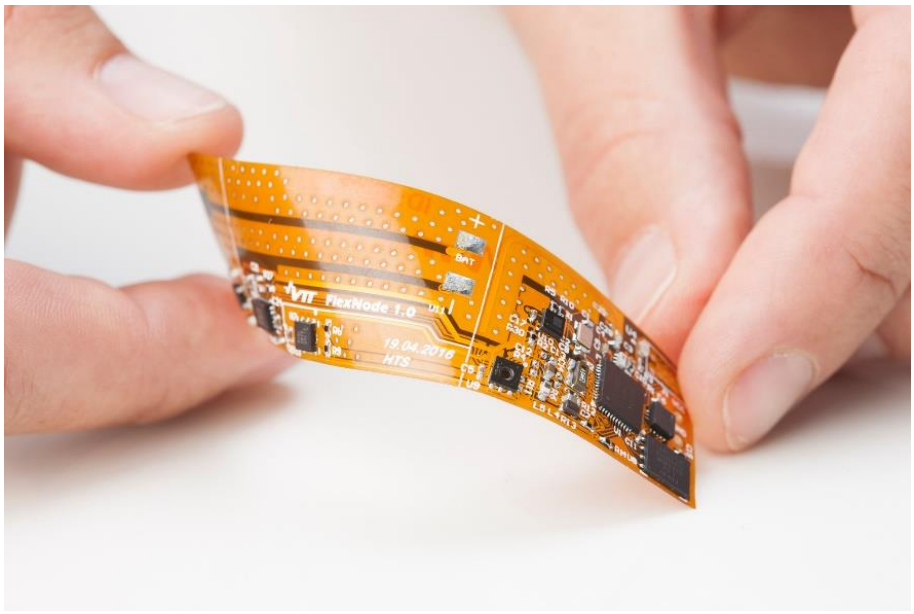

TGA and DSC test results of XLPE virgin and fast-aged samples

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Summary

This work covers micro-scale experiments of XLPE sheets. The XLPE sheets were made by VTT, and the Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) experiments were done also at VTT. Both non-aged (virgin) and thermally aged samples were studied. The thermally aged samples were "fast-aged" using the TGA apparatus and relatively high temperatures at around 200°C - 230°C and the aging time was some tens of minutes. The fast-aging scenario is based on the literature findings that were summarized in the SAFIR2022 programme project URAN that formed the base of the present project.

The aim of the present study is to provide experimental data for computational models for aged and virgin cables that can be built to carry out small scale Fire Dynamics Simulator (FDS) based simulations concerning TGA and Cone Calorimeter. The current work is continued later in the FASAANI project and Cone Calorimeter experiments are done for the non-aged (virgin) XLPE sheets. The computational material models is expected to be further developed to carry out full scale cable fire simulations representing a real cable fire. It was noted in the previous study (URAN project) that a thorough micro-scale experimental information is needed in order to generate realistic material models for XLPE pyrolysis which can be further used in the CFD based fire simulations. Thus, the presented TGA/DSC experiments use different heating rates, different atmospheres (O_2/N_2), and a range of fast-aging samples to estimate the O_2 reactions during the aging process giving a wider picture of possibilities of thermal degradation of XLPE materials.

The virgin XLPE results are in line with the previous URAN project results and findings in the literature. The fast-aging results are qualitatively in line with the few related findings from the literature, where some information of fast-aging type experiments for PE (not XLPE) were found.

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Preface

This work has been carried out as a part of work package 2 of FASAANI project which is one of the projects in the SAFER2028. The work covers the task 2.2 of the project. Thanks are extended to the State Nuclear Waste Management Fund (VYR) and as well as other key organisations operating in the field of nuclear energy in Finland for funding the project work.

18.12.2024

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1. Introduction

The experimental research results reported in this document are part of the Finnish SAFER2028 research programme project FASAANI, specifically concerning its Task 2.2. The findings made in the previous project, the Finnish SAFIR2022 research programme project URAN, particularly its Task 2.2, motivated the present work. The goal of this research is to increase the safety of the Finnish nuclear infrastructure, especially the fire safety of nuclear power plants (NPPs). One of the major fire loads present in NPPs is the various kinds of cables in the plants. A special type of cable in the fire risk analysis of nuclear facilities is the power cable. There are many different types of power cables in plants, but cables using cross-linked polyethylene (XLPE) as the electrical insulation material are quite common. The task of the FASAANI project presented here addresses the fire safety of these cables. There is some research knowledge on the fire behaviour of such cables when they are “virgin,” i.e., not very old, but there is much less knowledge on the properties of such cables after they have been installed and used in NPPs for decades. NPPs typically operate for at least five decades, at least in Finland. The Loviisa plants were erected in the late 1970s, and their lifetime (the licence) might be extended by 20 years, enabling the plants to operate for a total of 70 years. Polymeric materials like XLPE undergo ageing due to the environmental conditions at the plants during this interval. The cables are exposed to O_2 (air) and might also be exposed to humidity, radiation (both particle and electromagnetic), above ambient temperatures, mechanical stresses, etc.

The work reported in this report is not aimed to answer all the question relating to the aging of power cables in NPPs. The work is targeted on the aging of the XLPE insulation material itself. Thus, preliminary work done for the aging of XLPE material is reported in this report. There have been some aging experiments done for XLPE in the earlier SAFIR2022 projects (Korhonen et al., 2023) which are used as background information in the present project. The availability of experimental information of aging of XLPE is a requirement when making material models suitable for cable fire simulations using computational fluid dynamics (CFD) based methods, especially using Fire Dynamics Simulator (FDS) software (McGrattan et al., 2023). FDS is a CFD-based tool developed by NIST (USA) and VTT (Finland) to study the transport of heat and smoke from a fire. It also provides options to carry out research in fire and combustion. It enables to do complex pyrolysis modelling which falls in line with the objective of this work.

The aim of the current task is not to draw comprehensive conclusions, but to increase the amount of the experimental information on XLPE and its artificially aged counterpart. In the SAFIR2022-URAN project it was found that more information was needed that could be obtained from the SAFIR2022-SAMPO data and this data was tried to be found using a quick internet search. The aim was not to do a comprehensive literature survey, the aim was just to find some information that might be helpful when performing the task. The main problem that was noted was the fact that no comprehensive experimental data was found on the same XLPE sample material covering many scales from microscale to full scale experiments including information on the O_2 reactions that shows up, e.g., in analysis of virgin and aged samples using TGA runs made in both N_2 and O_2/air atmospheres. The current work is aimed to answer (partly) to this need. Microscale and intermediate scale experiments will be done on the same XLPE material during this present project. The experiments will be done under different atmospheres so that the O_2 reactions can be analysed better.

The outcome of the Task 2.2. of the FASAANI project will be a set of experimental results of XLPE. These experiments are described, and their results are presented on “as is” basis. This report contains the results of the virgin XLPE experiments carried on by the simultaneous TGA/DSC equipment at the VTT location in Tampere. The results are compared to literature data somewhat to see that the results are credible, but a more thorough analysis of the results is not done, because it is not included in this project’s scope.

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2. TGA

In order to understand the behaviour of a material in a fire, it is crucial to have comprehensive knowledge about the thermal decomposition processes. These processes are often greatly influenced by the conditions, especially the heating rate and atmosphere. Additionally, sensitivity to these parameters provides essential clues to interpret the material's behaviour in fire. Thermogravimetric Analysis (TGA) is one of the most common methods to study such thermal decomposition processes of polymers. Materials science uses it widely to characterize materials' thermal stability, composition, and decomposition properties.

TGA measures a sample's mass change as it is heated, cooled, or held at a constant temperature over time. The material is expected to change its mass under a constant, elevated temperature or prescribed heating rate. The graph having the plots of change in the mass percentage along with differential mass loss aids in the understanding of the decomposition of a material at various temperatures (Figure 1). For example, it can be noted from the Figure 1 that the material starts to decompose at around 100 K and continues to lose its mass after that. Moreover, peak mass loss happens at around 150 K. Furthermore, along with such data and further analysis of the concerning graphs, the following are also found/determined:

1. Oxidative stability
2. Thermal stability
3. Kinetic data
4. Content of low volatiles
5. Content of fillers
6. Identification of polymers or other materials present in a composite material.

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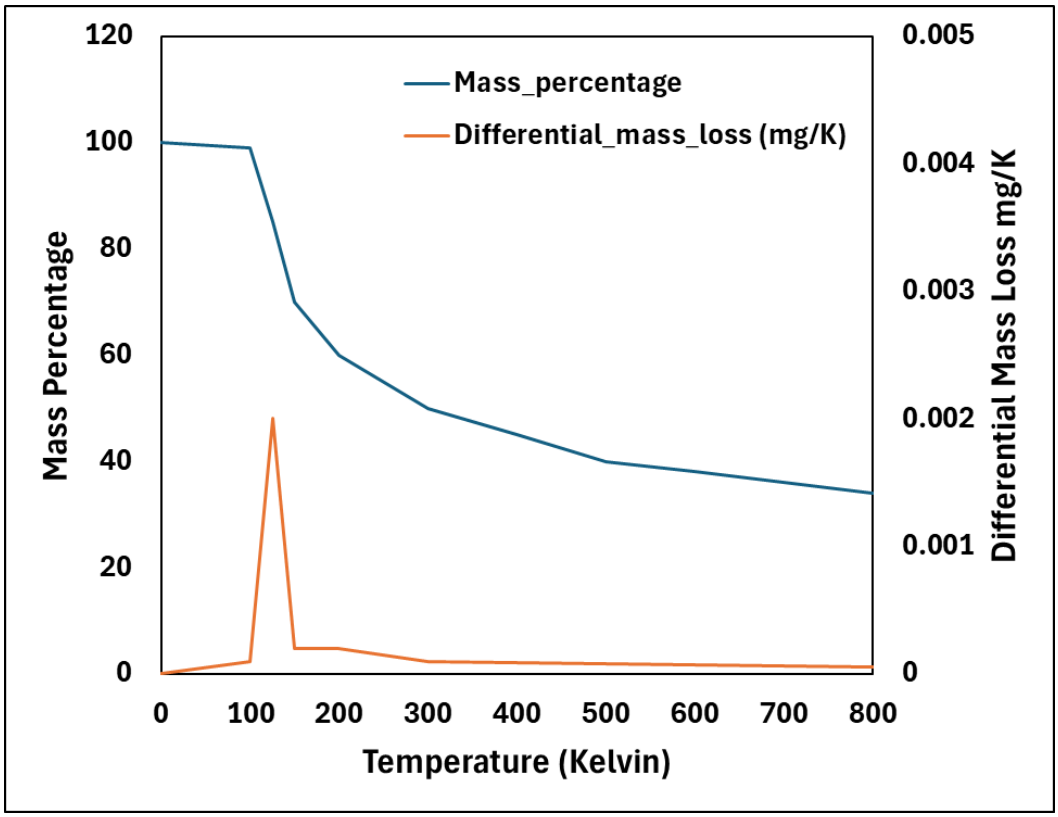

Figure 1. Sample plot of mass percentage and differential mass loss about from TGA

Kinetic data is very useful in making computational material models for TGA simulations. As the kinetic data concerns the degradation, its correct estimation aids in replicating such thermal degradation in such simulations. Some other missing parameters are found with data on the burning behaviour of the same material from the cone-calorimeter, and the overall material model is optimised. Once the material model is ready, that model can be used for full-scale fire simulation under different ignition and environmental conditions.

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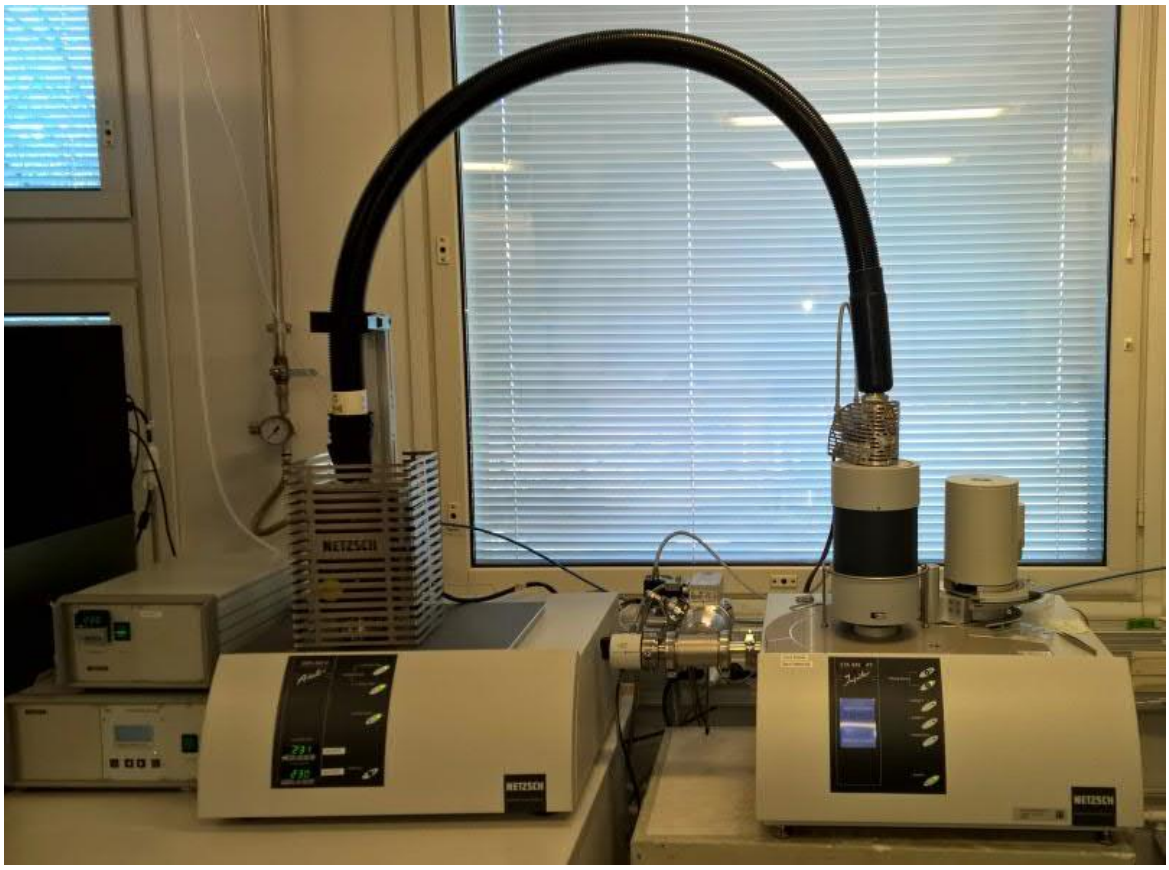

Figure 2. TGA at Tampere. Left: Mass spectrometer (MS), Right: TG-DSC (STA simultaneous thermal analysis)

Components

TGA uses a very sensitive scale to measure even a very small changes in the weight of a sample. The scale must be accurate and should work well at different temperatures. The small furnace in TGA carefully heats the sample. It's important for the furnace to heat the sample uniformly. The sample is put in a special container (sample holder) made of strong materials that can handle high heat without reacting with the sample. Sensors are used to measure and control the temperature. The system makes sure the sample is heated at the right speed and stays at a steady temperature. Different gases can be added to the furnace to create specific conditions for the analysis. This helps us understand how different conditions affect the sample. Modern TGA machines have software to control the experiment, save data, and do analysis in real-time. The software can create graphs showing how the sample changes over time or temperature and provide detailed analysis.

Working Principle

Thermogravimetric analysis (TGA) works by monitoring how the mass of a sample changes as it is heated or cooled over time. The process starts with preparation, where a small sample is weighed and placed in a sample holder. The furnace is then closed, and a controlled atmosphere is set up, using either an inert or reactive gas to suit the material being tested. After that heating or cooling phase as required is started. The sample is subjected to a specific temperature program, either increasing or decreasing the temperature at a steady rate. Throughout this process, the instrument keeps a close eye on the sample's

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mass, measuring any changes as the temperature shifts. The instrument continuously records the sample's mass alongside the temperature. This produces a graph which shows how the mass changes over time as the temperature varies. The graph is examined to understand the sample's behaviour under heat. It reveals critical information about the sample’s thermal stability, the temperatures at which it decomposes, and its overall composition. The derivative of this data, known as the DTG curve, helps pinpoint specific events like decomposition temperatures and mass loss more precisely.

Advantages and Limitations

One of TGA's main advantages is that it provides a precise measurement of mass change with respect to temperature change. It is almost a non-destructive exercise and facilitates further characterization of the sample left as a residue. However, the sample size is very small and typically ranges in milligrams. Moreover, some unwanted volatile decomposition can affect the mass measurement if not managed properly.

Overall, the thermogravimetric analysis apparatus is a powerful tool for studying the thermal properties of materials. With its precise control and measurement capabilities, the TGA apparatus enables detailed analysis of material behaviour under various thermal conditions, aiding in the study of fire behaviour.

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3. Fabrication of the XLPE sheets

The process of preparing thin crosslinked XLPE sample sheets was carried out using a hot press technique to induce crosslinking in the XLPE granules and produce solid, crosslinked sheets for further testing. A small amount of granules (~30 g) was carefully measured and poured onto the surface of a pre-heated hot pressing plate, which was heated to 160°C, slightly above the materials melting point. At this temperature, the polymer granules were melted uniformly over the span of 10 minutes. After the initial melting phase, pressure was applied to the molten XLPE to ensure uniform thickness and distribution of the sheet. Following this the temperature of the hot press plate was raised to 210°C to induce crosslinking. At this elevated temperature, the polymer chains began to form crosslinked bonds. This crosslinking phase was brief (~10 min) to prevent overexposure to heat, which could affect the sample's integrity. Once crosslinking was achieved, the sample was slowly cooled to allow for controlled solidification.

The degree of crosslinking in the hot pressed XLPE sample sheets was assessed using the Soxhlet extraction method. This technique allows for the separation of the non-crosslinked polymer fraction from the crosslinked gel by utilizing a solvent, such as xylene, which selectively dissolves the non-crosslinked polymer chains. The residual gel, which remains unaffected by the solvent, represents the crosslinked portion of the polymer. The Soxhlet extraction process is therefore an effective tool for quantifying the degree of crosslinking in crosslinked polyethylene materials.

In this work, xylene was chosen as the solvent for its ability to dissolve the non-crosslinked portions of the polymer while leaving the crosslinked gel intact. The entire procedure was performed inside a fume cupboard to prevent exposure to xylene vapours. The Soxhlet apparatus used for the extraction consisted of a boiling flask, a condenser, and an extraction chamber. A metal mesh was placed in the extraction chamber to hold the XLPE sample in place (Figure 3). The sample was carefully weighed before and after the extraction to calculate the mass of the remaining gel and determine the degree of crosslinking.

Figure 3. Metal mesh placed in the extraction chamber to hold the XLPE sample in place

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The process began by preparing a small sample of XLPE, which was placed in the metal mesh inside the Soxhlet extraction chamber, and an extraction sock. Xylene was then poured into the boiling flask of the Soxhlet apparatus. The xylene was heated to its boiling point (~138.5°C), causing it to vaporize and rise into the condenser. The vapor then condensed and dripped down onto the XLPE sample inside the extraction sock. This reflux cycle allowed the xylene to continuously wash over the sample, selectively dissolving the non-crosslinked polymer chains. This extraction was carried out for 8 hours to ensure thorough dissolution of the soluble polymer fraction. After the extraction period the remaining sample was removed from the Soxhlet apparatus, left to dry and then its final mass was measured.

To determine the degree of crosslinking in the XLPE sample, the initial and final masses of the sample were compared. The initial mass represented the total polymer content, while the final mass displays the crosslinked portion of the polymer, which had not dissolved in the xylene. The difference between the two masses indicates the amount of non-crosslinked material that had been dissolved and removed during the Soxhlet extraction.

The degree of crosslinking was calculated using the following equation:

$$Gel\ fraction(\%) = \frac{Mass\ of\ residual\ gel}{Initial\ mass\ of\ sample} \cdot 100$$

For the XLPE sample to be considered adequately crosslinked, the gel fraction needed to be greater than 50%. The results of the analysis indicated that all XLPE sheets tested were well crosslinked, as shown in Table 1.

Table 1. XLPE sheets fabricated. The prepared sheets were cut into 11x11 cm^2

Sample	Pieces	Piece height average (mm)	Piece width average (mm)	Piece thickness average (mm)	Degree of cross linkage average(%)
Sheet 1	9	115	113,4	1,61	89,15
Sheet 2	11	116,3	114,3	1,68	93,02
Sheet 3	6	115,6	113,2	1,55	73,67
Sheet 4	7	115,9	114,1	1,63	85,23

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4. Experimental data of the XLPE material

The pyrolysis of the XLPE material produced as described in Section 3 was studied using combined TGA/DSC device. The pyrolysis processes were studied for both non-aged (virgin) material and aged material. Three different sets of experimental campaign were carried out:

1. Virgin XLPE TGA/DSC runs:
    a. Four heating rates, N_2 and O_2 atmospheres used.
    b. Information of the virgin XLPE pyrolysis processes sought.
    c. Results were also used to “guess” aging temperatures for fast aging tests (step 2 below).
2. Fast-aging tests:
    a. One heating rate, only N_2 atmosphere, 3 different aging temperatures and times used.
    b. Samples are heated up to aging temperature inside the TGA apparatus and kept there for the specified time period, cooled down and then a standard TGA/DSC analysis was run.
    c. Objective was to find reasonable aging temperatures and times to be used in the actual fast-aging experiments (step 3 below), i.e., to see how high temperature can be used in the fast-aging experiments and how long should the aging phase last in order to see some effects in the TGA curves.
3. Fast-aging experiments:
    a. TGA/DSC: Two heating rates, N_2 and O_2 atmospheres used.
    b. Fast aging: Two temperatures and two aging times used.
    c. Fast aging done similarly as described above in step 2. The heating was done till aging temperature. Then it was cooled back to ambient temperature before the TGA/DSC analysis, which was always done in N_2, i.e., the fast-aging O_2 reactions happened only during the actual aging time slot.

A few micrograms sized samples were taken from the thin XLPE sheets made as mentioned in section 3. These sheets are to be used in the cone calorimeter experiments to be performed later during the FASAANI project. Together with the cone calorimeter results, the current TGA/DSC results can be used to generate material reaction computational models to be used in FDS modelling of cables containing XLPE insulation material. The experimental results are not just aimed for FDS usage, but the results are quite general and can be used whenever a detailed information on the XLPE thermal degradation process is required for studies and modelling. It should also be noted that the present results apply for the current XLPE material. There are many different brands and grades of XLPE, and, for example, the amount of the antioxidant additivities varies both in amount and type, so the present aging results and virgin material TGA in O_2 atmosphere results might not correspond to many other XLPE materials out there. The analysis of the aging and O_2 atmosphere results will not be straightforward due to the very limited oxygen diffusion (low speed) into XLPE. Only the surfaces of the samples are exposed to O_2 in the present experiments which does not fully replicate the real situation in which O_2 penetrates inside the material.

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4.1 Experimental results of the virgin XLPE material

The list of the TGA/DSC experiments done for the non-aged, virgin, XLPE material is presented in Table 2. Note that the case “1C” was terminated abruptly at 445 °C temperature and its data is not shown in the figures below. Just the successful TGA/DSC run results are reported.

Table 2. The experimental TGA/DSC campaign for the virgin XLPE. Repetition test 1C did not complete, data exists up to 445 °C and its results are not shown in the figures below, i.e., its data is discarded.

ID	Atmosphere	Heating Rate (K/min)	Repetition
1A	N_2	5	
1B	N_2	5	x
1C	N_2	5	x (445 °C)
2A	N_2	10	
2B	N_2	10	x
3A	N_2	20	
3B	N_2	20	x
4A	N_2	30	
4B	N_2	30	x
4C	N_2	30	x
5A	O_2	5	
5B	O_2	5	x
5C	O_2	5	x
6A	O_2	10	
6B	O_2	10	x
7A	O_2	20	
7B	O_2	20	x
8A	O_2	30	
8B	O_2	30	x
8C	O_2	30	x

The results of the TGA measurement of virgin XLPE are shown in Figure 4 (TGA was run from 40 °C to 800 °C, shown just 200 °C –600 °C). A close-up is shown in Figure 5. The mass loss rates (MLR) are shown similarly in Figure 6 and in Figure 7.

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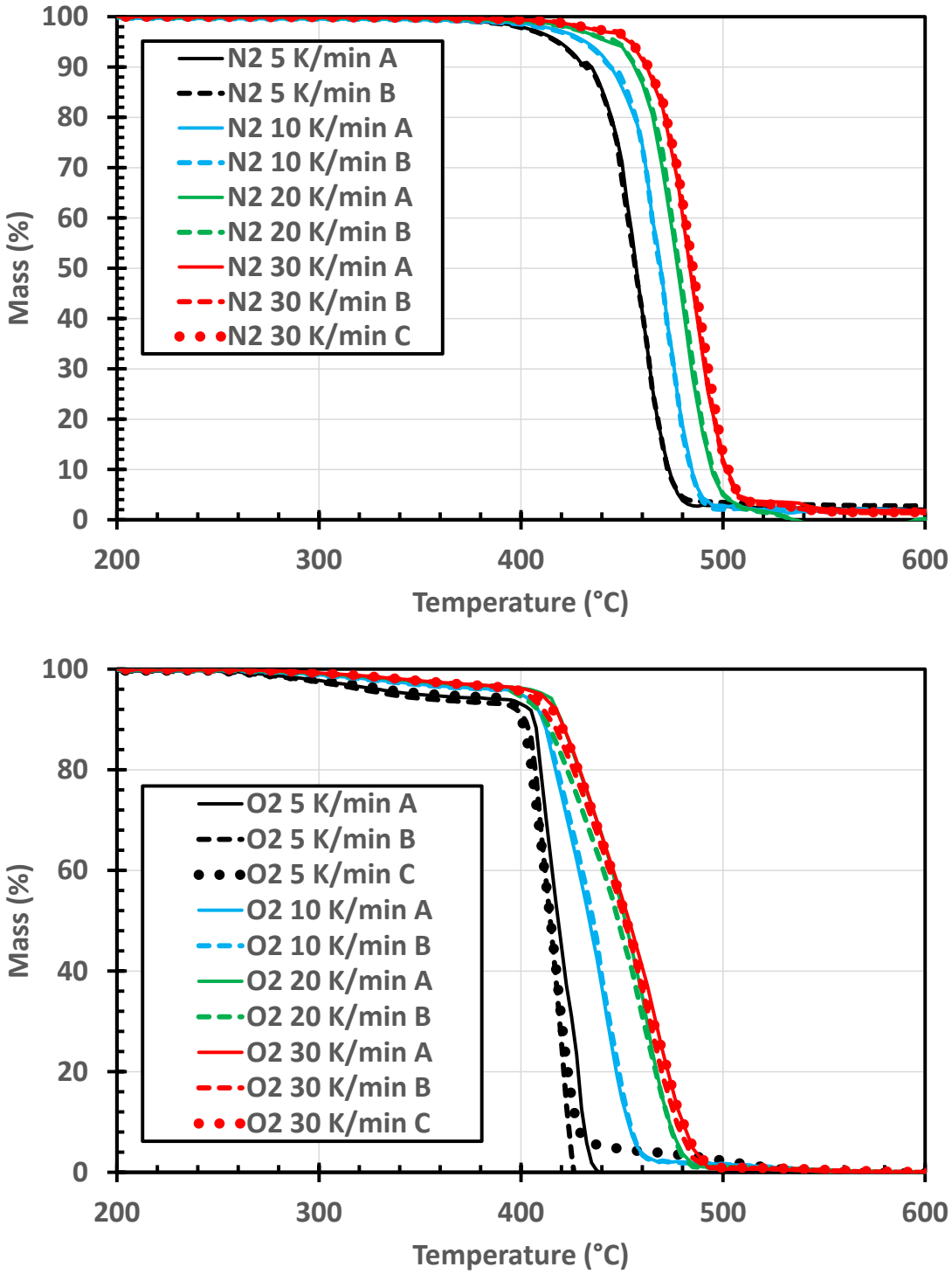

Figure 4. The TGA results of the non-aged (virgin) XLPE. Shown are the normalized mass curves measured in N_2 atmosphere (top) and in O_2 atmosphere (bottom). The legend shows the atmosphere (N_2/O_2), the heating rate, and the repetition index (A/B/C).

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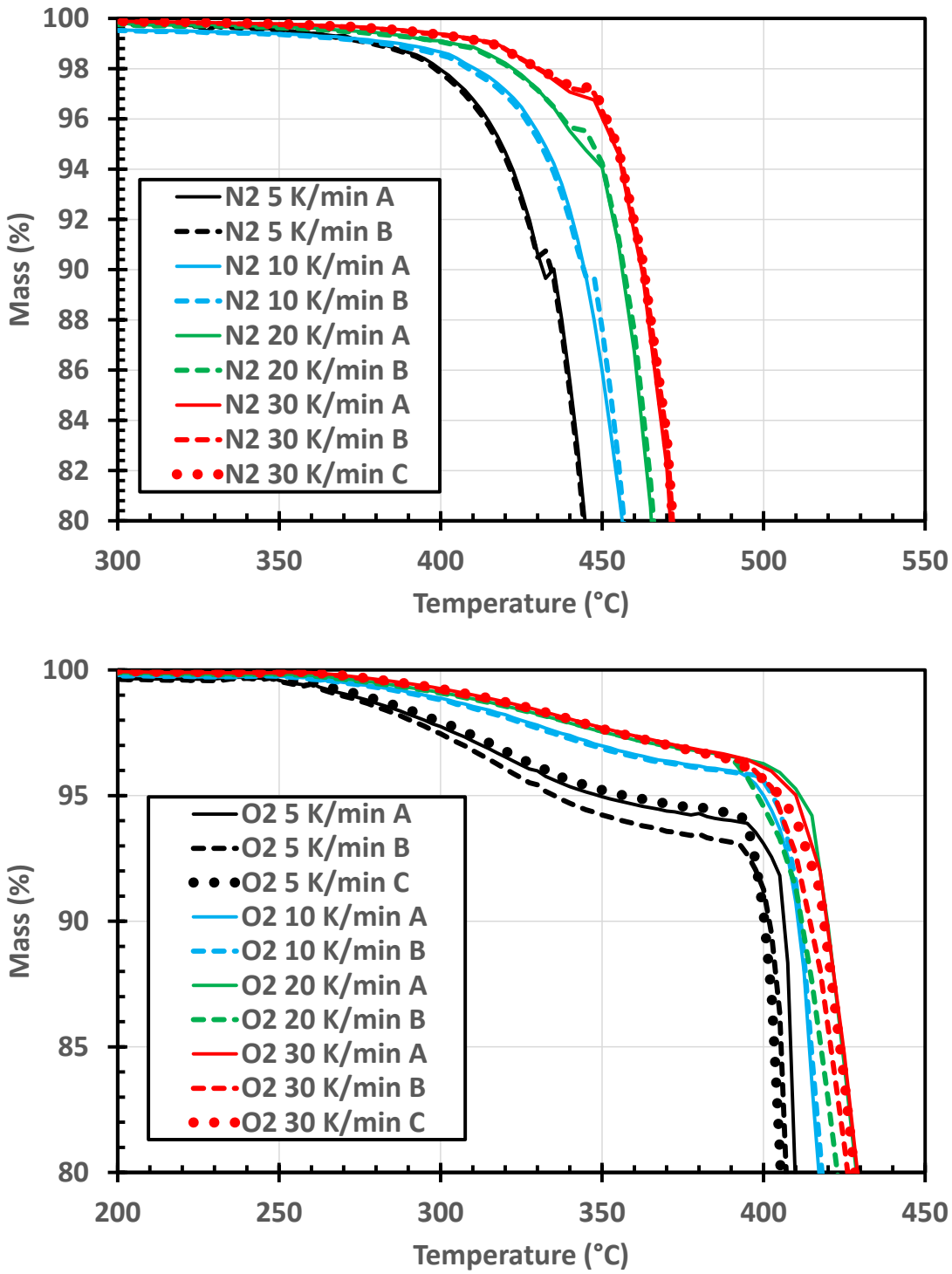

Figure 5. Closeup of the TGA results of the non-aged (virgin) XLPE. Shown are the normalized mass curves measured in N_2 atmosphere (top) and in O_2 atmosphere (bottom). The legend shows the atmosphere (N_2/O_2), the heating rate, and the repetition index (A/B/C).

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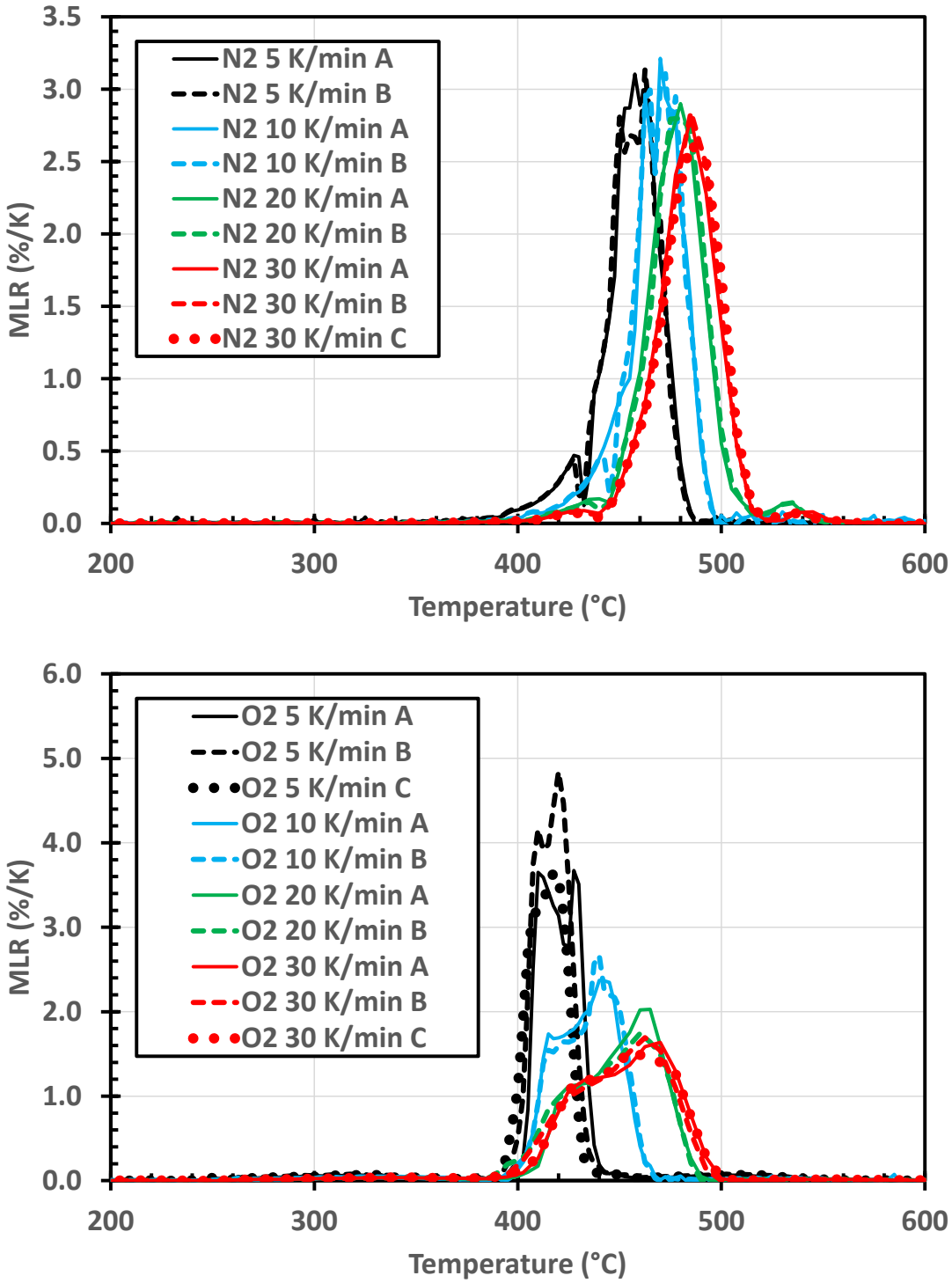

Figure 6. The TGA results of the non-aged (virgin) XLPE. Shown are the mass loss curves measured in N_2 atmosphere (top) and in O_2 atmosphere (bottom). The legend shows the atmosphere (N_2/O_2), the heating rate, and the repetition index (A/B/C).

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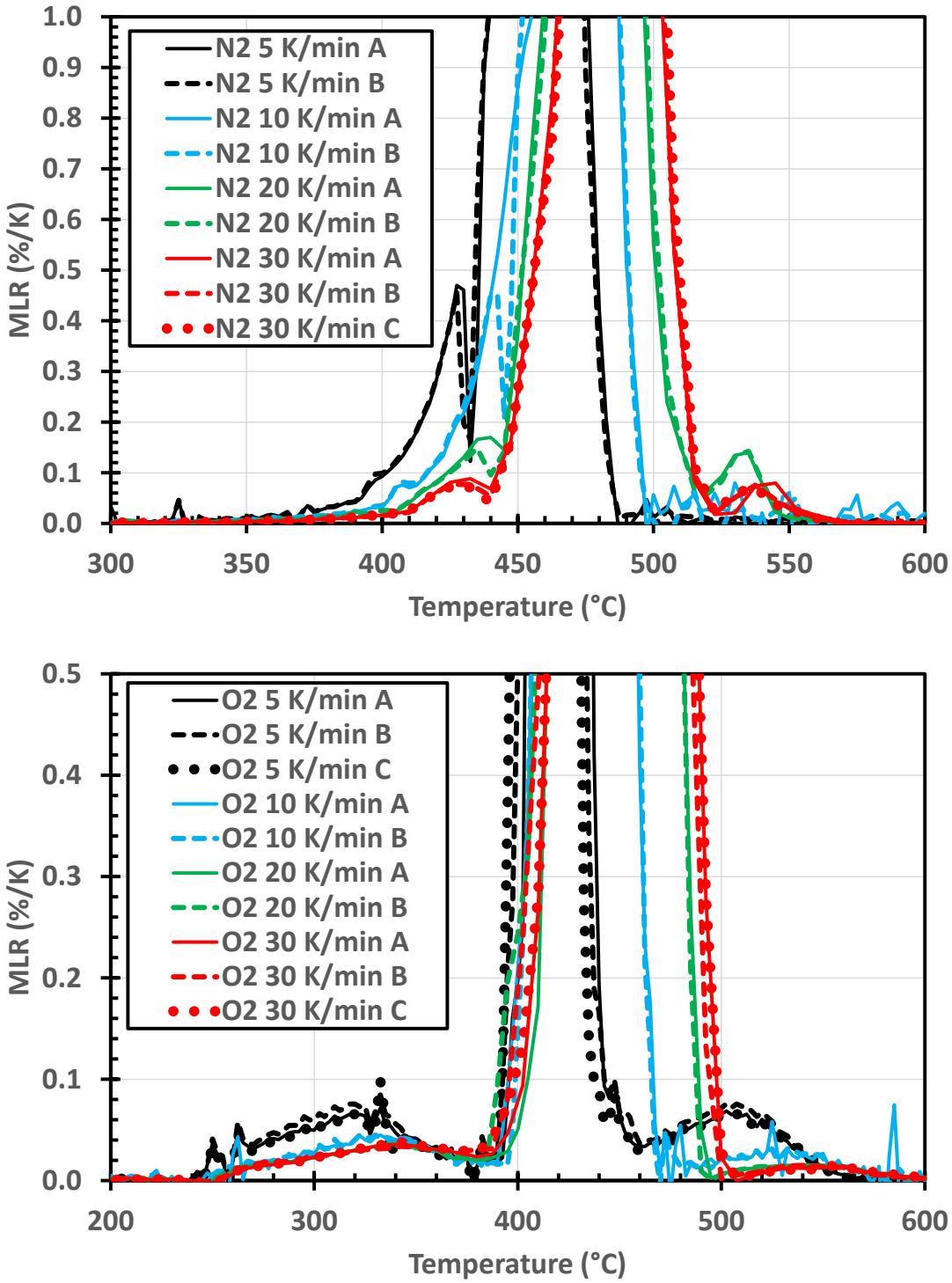

Figure 7. Closeup of the TGA results of the non-aged (virgin) XLPE. Shown are the mass loss curves measured in N_2 atmosphere (top) and in O_2 atmosphere (bottom). The legend shows the atmosphere (N_2/O_2), the heating rate, and the repetition index (A/B/C).

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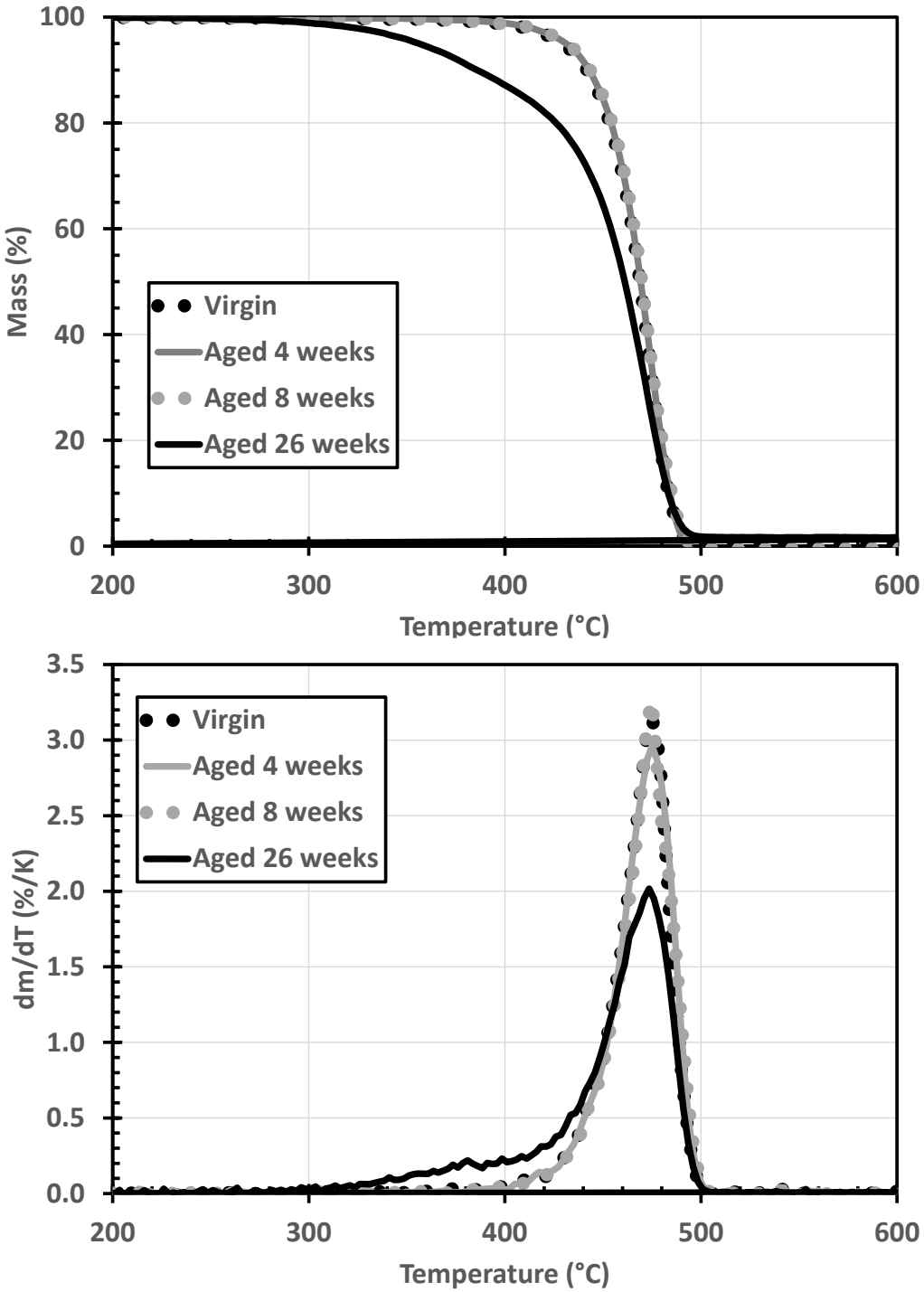

Figure 8. SAMPO TGA (in N_2) results for the virgin and aged (at 130 °C air) XLPE samples taken from Korhonen et al., 2023. Normalized mass curves (top) and temperature derivatives of the normalized mass curves, dm/dT (bottom), are shown.

In Figure 8 the results of the previous SAMPO project are shown (Figures 1 and 2 in Korhonen et al., 2023). The virgin sample (and 4-weeks and 8-weeks aged samples) TGA curve practically coincides the present 10 K/min TGA curve measured in N_2 atmosphere. The 26 weeks aged SAMPO sample shows noticeable reaction speeds starting about from 300 °C. The present results done in O_2 atmosphere show that some noticeable reactions start about at 240 °C. This can also be seen in the virgin XLPE DSC

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measurement results shown in Figure 9. The N_2 measurement results are shown as close-up to temperature range of 300 °C – 600 °C.

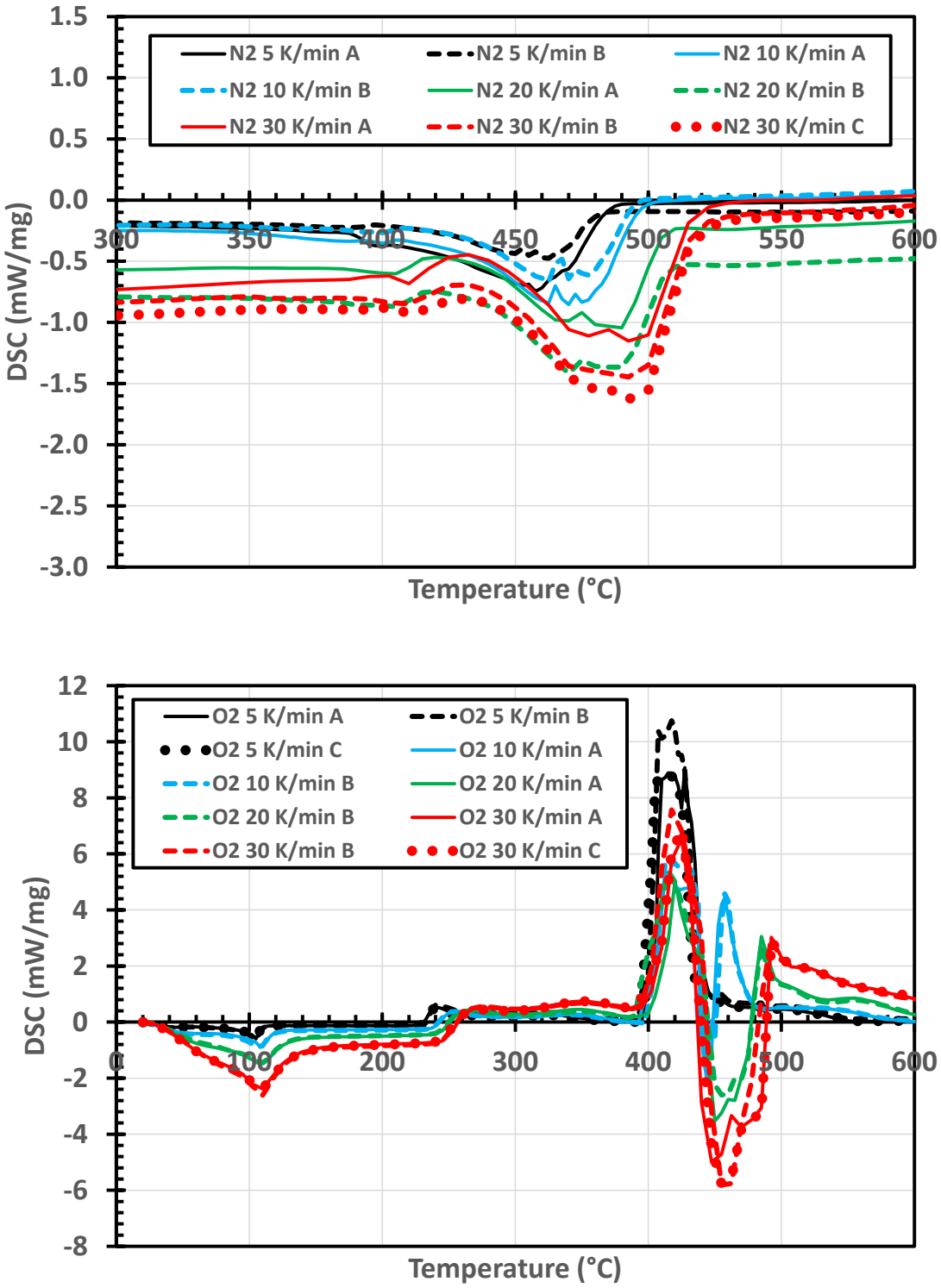

Figure 9. The DSC results of the non-aged (virgin) XLPE. Shown are the curves measured in N_2 atmosphere (top) and in O_2 atmosphere (bottom). The legend shows the atmosphere (N_2/O_2), the heating rate, and the repetition index (A/B/C).

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4.1.1 Analysis of the virgin XLPE TGA experiments

The above presented results for virgin XLPE are not much analysed in this report nor within this project. Below, the virgin TGA results measured in N_2 atmosphere are analysed up to some extent. The aim of this simple analysis is to see, if the present XLPE sample is similar to earlier samples and to some literature data. In Figure 10, the present results are compared to the SAFIR2022-SAMPO project result that were also used in the SAFIR2022 URAN project (Korhonen et al., 2023) to address the aging of XLPE. It is seen that the present XLPE material is very similar to the SAMPO project XLPE material. The FDS material models defined for the XLPE material used in the SAMPO project (Korhonen et al., 2023) were used to model the present results. Just the two parallel reaction scheme model was used to fit the FDS-based material model parameters in the present study. The present fits are shown in Figure 11. The SAMPO project fit is practically same as the present fit for the 10 K/min result, only very slight changes in the parameters occur. Thus, the present XLPE material seems to be very similar to the previously used material. It should be stated that the fits are “loosely tuned and optimized”, so not all parameters are changed (as the fit is good enough for present purposes, advanced optimisation is not required).

The TGA measurements done in O_2 atmosphere are not modelled in this work. That would need a reaction model for the O_2 reaction and a better analysis than done for the results of the TGA in N_2 done above. This kind of analysis is not in the scope of the current project. The experimental results are just given in this report to be later used, hopefully, by some other projects and scientific studies.

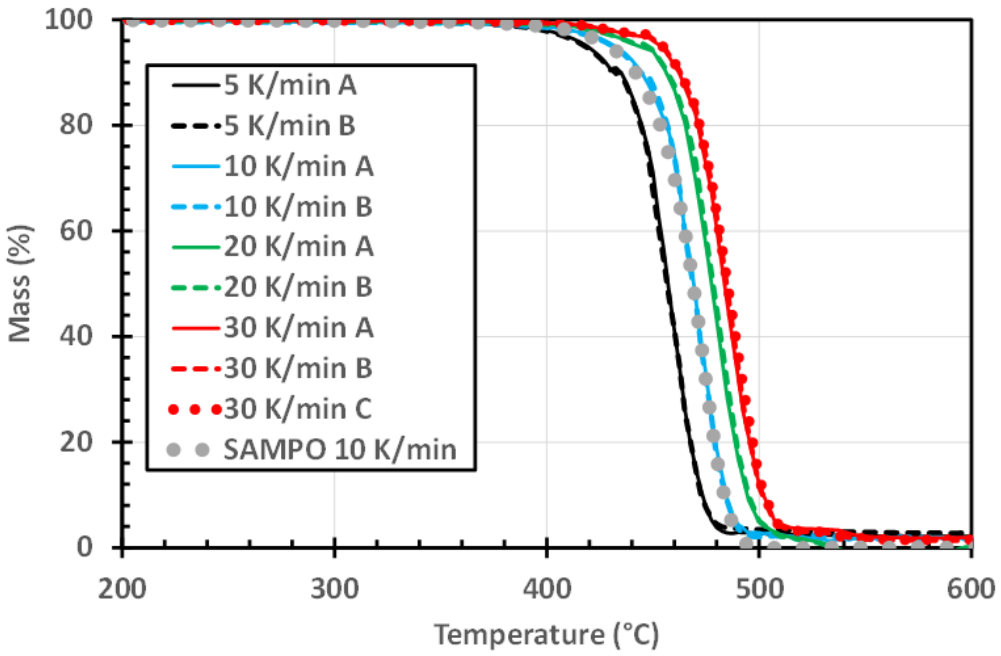

Figure 10. The TGA results of the non-aged (virgin) XLPE compared to the previous SAMPO project measurement. Shown are the normalized mass curves measured in N_2 atmosphere. The legend shows the heating rate, and the repetition index (A/B/C).

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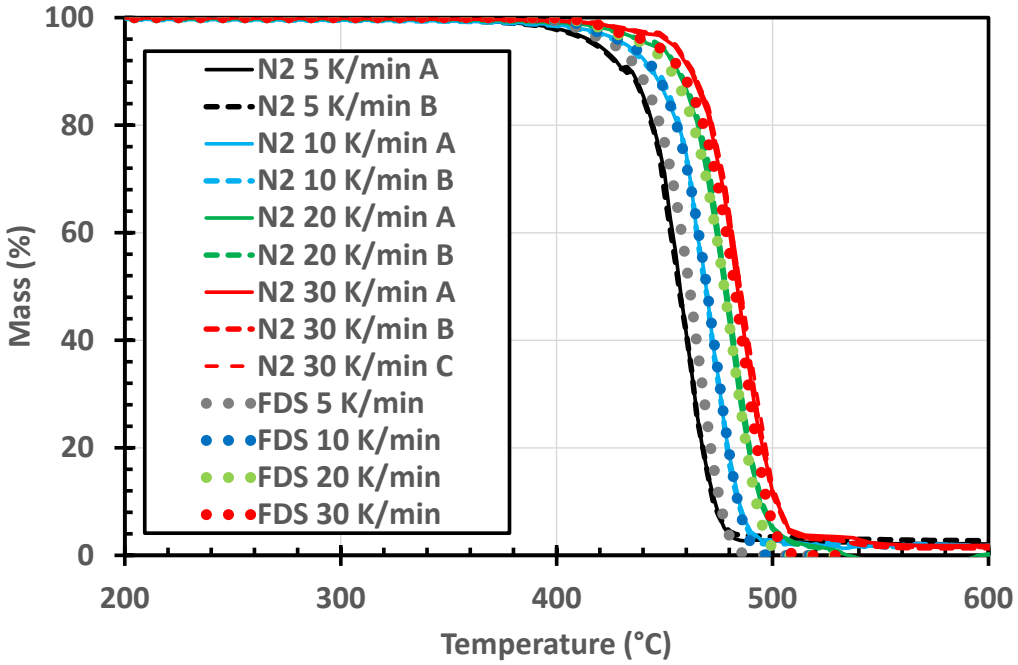

Figure 11. The FDS material model fits to the non-aged (virgin) XLPE TGA data. Shown are the normalized mass curves in N_2 atmosphere. The legend shows the heating rate.

The present TGA results conducted in O_2 can be qualitatively compared to the literature findings listed in the URAN project report (Korhonen et al., 2023). The effect of the O_2 reactions is clearly seen in the present TGA results (Figure 6), but the effect is not as large than in some cases in the literature (Peterson et al., 2001, Cho et al., 1998). The O_2 reaction of the present material seem to be also seen in the literature findings. But the magnitude of the O_2 reaction, i.e., how much of the sample mass is lost, is less for the present XLPE material than, for example, in Peterson et al., 2001. The difference might be due to the additivities of XLPE materials that vary between the manufacturers and the grade of the material. The present XLPE was not “pure” XLPE, it was a normal grade used in manufacturing of cables. So, it contains “typical” additivities and most typical ones are also the antioxidants that are needed to slow down the aging of the cables. Thus, it is not surprising that the O_2 reaction does not show up as strongly as in some literature findings, where “purer” XLPE might have been used.

4.2 Experimental results of the aged XLPE material

4.2.1 Results of the fast-aging tests

The virgin material TGA results showed that the present XLPE material seems to be quite in line with the literature data and also with the SAFIR2022-SAMPO data. Thus, it was assumed that the present XLPE sample material does not undergo any noticeable reactions with mass loss below 200 °C (based on various graphs). There is a melting-like reaction about 110-120 °C as seen in the DSC data (latent heat), but otherwise the material seems to be quite stable up to 200 °C. So, it is assumed that just the aging reactions (O_2 effects) occur at these temperatures and colder ones. The O_2 reaction is occurring fast at about 250 °C or so. Thus, a suitable fast-aging temperature should be determined in the temperature range 190 °C – 240 °C. For this reason, a set of seven “fast-aging test runs” were performed by the TGA/DSC apparatus. The fast aging was done in O_2 atmosphere (of course, O_2 reactions were required), but the TGA/DSC run was made only in N_2 atmosphere. The aim was just to see, how long and at which temperature the sample should be aged in order to see some (large enough) changes in the TGA curve. This information was then

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used to define the fast-aging temperatures and times for the final aged XLPE TGA/DSC test series, where TGA/DSC was to be done both in O_2 and N_2 atmospheres.

The list of the fast aging test is presented in Table 3 and the results are shown in Figure 12 and Figure 13 for TGA and DSC, respectively. It is seen that the present XLPE material does not react with O_2 strongly under 240 °C temperatures (degradation starts after 240 °C). The aging effects on the TGA and DSC curves are quite minor, if the fast-aging temperatures 200 °C and 215 °C are used. So, the fast-aging temperature to be used in the main high temperature fast-aging experiments should be 230 °C (neither low: 215°C nor high: 240°C). Some experiments should be conducted at 220 °C also. The aging times should be in the order of few hours in order to see remarkable aging effects in the TGA data.

Table 3. The experimental TGA/DSC campaign for the fast-aging tests of XLPE.

ID	Atmosphere	Heating rate (K/min)	Aging temperature (°C)	Aging time (min)
1	N_2	10	200	30
2	N_2	10	200	60
3	N_2	10	215	15
4	N_2	10	215	30
5	N_2	10	215	60
6	N_2	10	230	15
7	N_2	10	230	30

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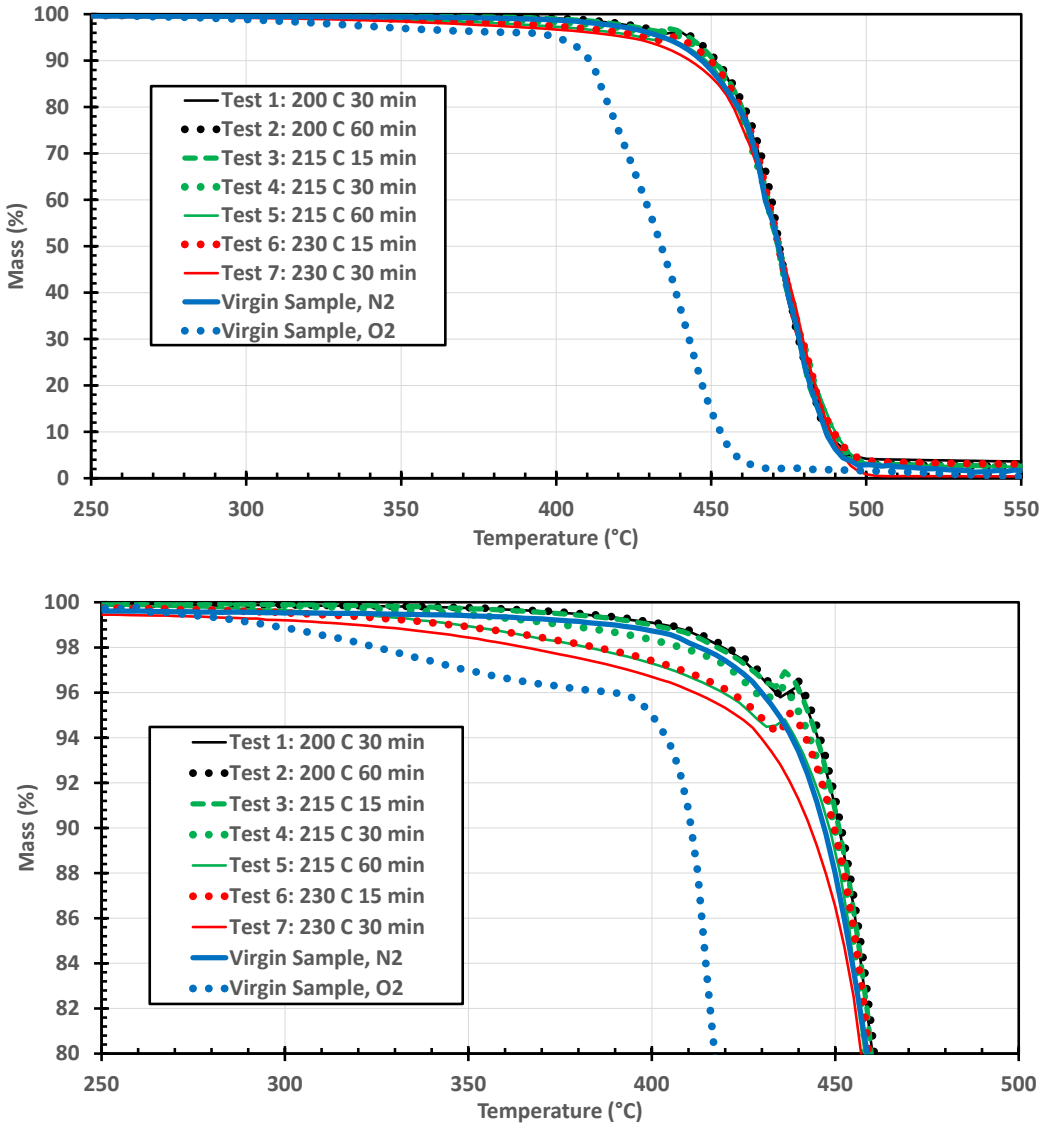

Figure 12. The experimental TGA results of XLPE in the fast-aging tests 1-7 compared to virgin XLPE. All measurements used 10 K/min heating rate and N_2 atmosphere for the TGA measurement, virgin sample was also measured in O_2 atmosphere. Bottom figure presents a closeup view.

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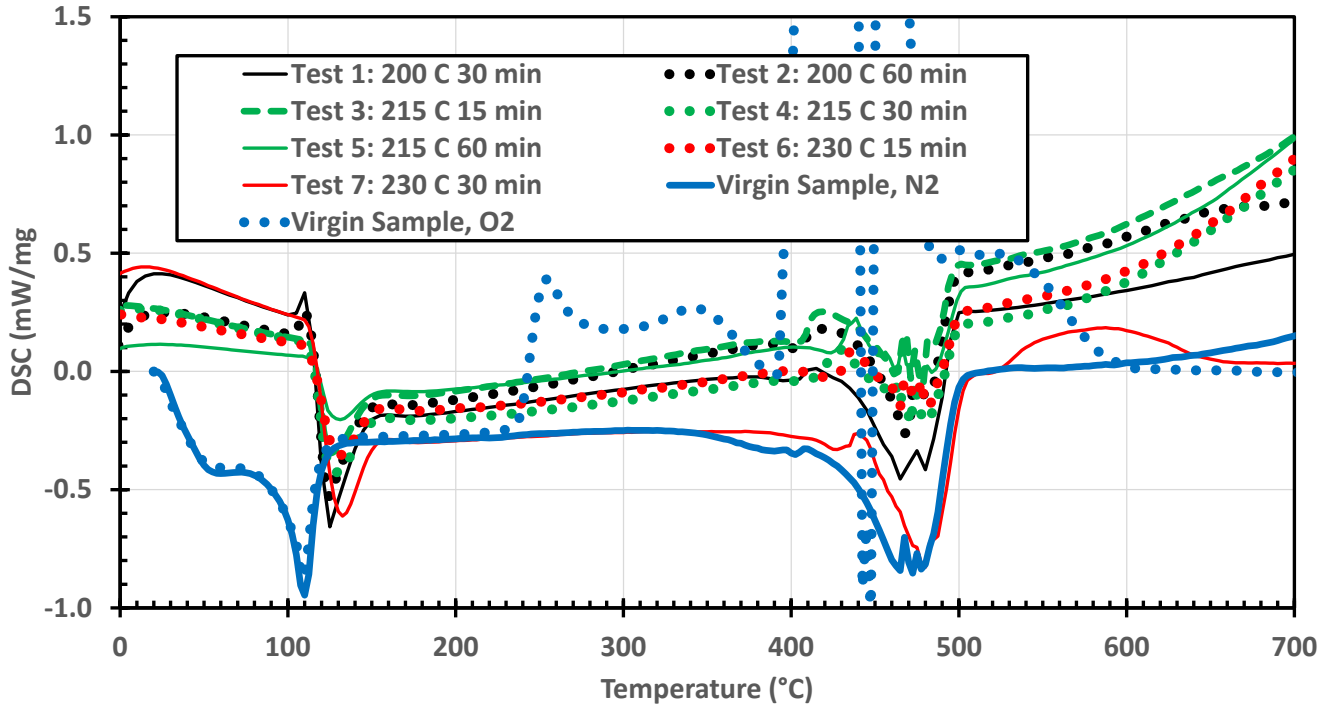

Figure 13. The experimental DSC results of XLPE in the fast-aging tests 1-7 compared to virgin XLPE. All measurements used 10 K/min heating rate and N_2 atmosphere for the DSC measurement, virgin sample was also measured in O_2 atmosphere.

4.2.2 Results of the aged XLPE material

The fast-aging temperatures chosen according to the above shown results of fast-aging test runs were 220°C and 230 °C, and fast-aging times were 60 and 120 minutes. The list of the final aging experiments is presented in Table 4 and the results shown in Figure 14 and Figure 15. It is seen that there are some aging effects, but they are not very pronounced. The results are in line with the fast-aging test run results shown above. The longer aging time increases the aging effects on mass loss. The TGA/DSC data measured in O_2 atmosphere does not show as large aging effects as the N_2 data. The O_2 reactions start at about 300 °C and the aging effects are seen at a little bit higher temperatures in the N_2 measured TGA curves. Thus, aging effects are not as easily seen in the measurement data. One should note that the O_2 atmosphere aged samples have lost some of their initial mass during the aging and their mass is not exactly 100 % as the TGA run starts after the aging. The TGA data in Figure 15 has O_2 aged sample data scaled to be 100 % at the start of the TGA run. The virgin XLPE TGA data (in O_2) is now quite well within the O_2 aged data.

Table 4. The experimental TGA/DSC campaign for the fast-aged XLPE.

ID	Atmosphere	Heating rate (K/min)	Aging temperature (°C)	Aging time (min)	Repetition
1	N_2	10	230	60	
2	N_2	10	220	60	x
3	N_2	10	230	120	
4	N_2	10	220	120	x
5	O_2	10	230	120	

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ID	Atmosphere	Heating rate (K/min)	Aging temperature (°C)	Aging time (min)	Repetition
6	O_2	10	230	60	x
7	O_2	10	220	120	
8	O_2	10	220	60	x

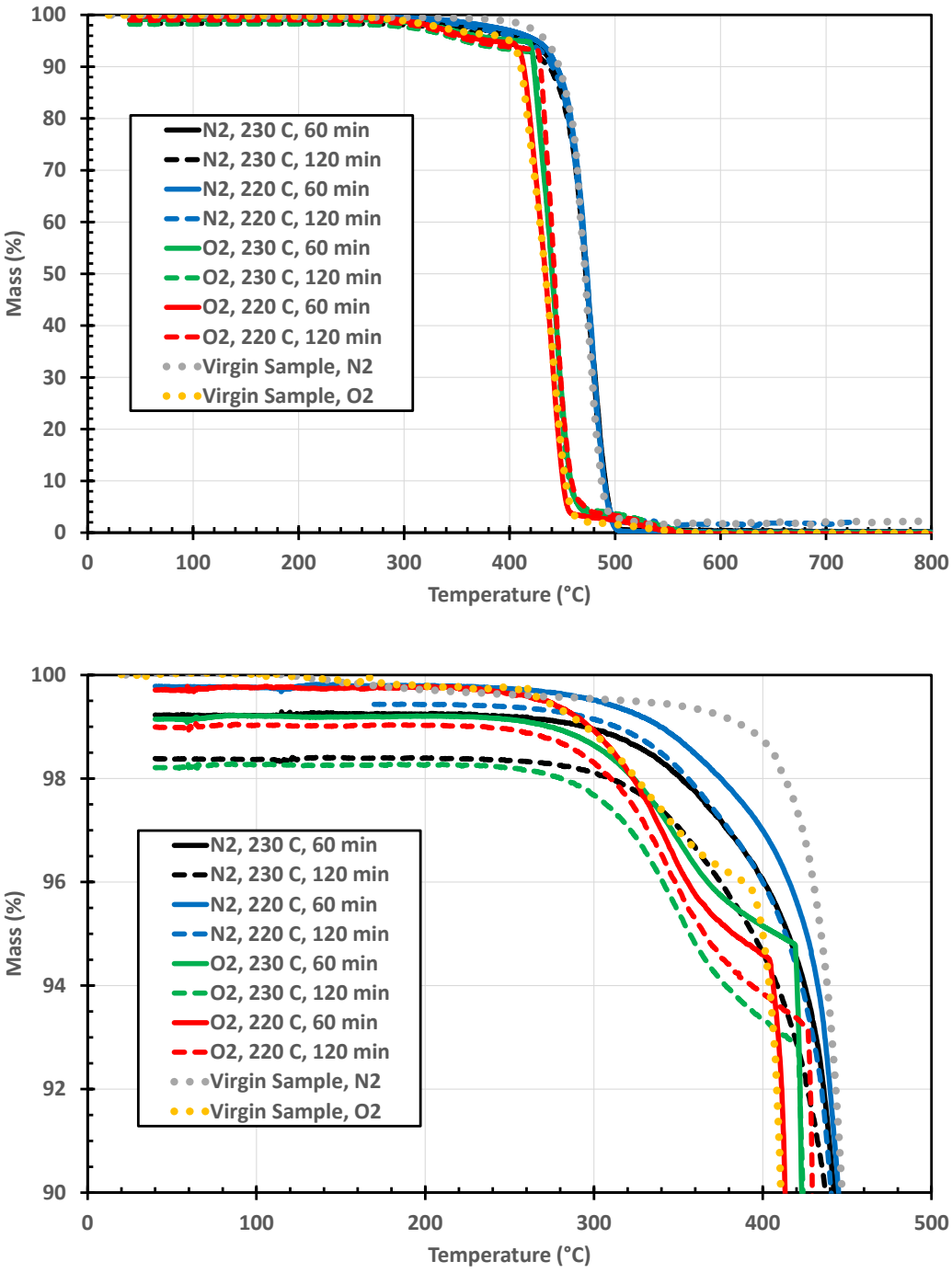

Figure 14. The experimental TGA results of XLPE in the fast-aging experiments 1-8 compared to virgin XLPE. All measurements used 10 K/min heating rate and the combined TGA/DSC runs after fast-aging were made both in O_2 and N_2 atmospheres.

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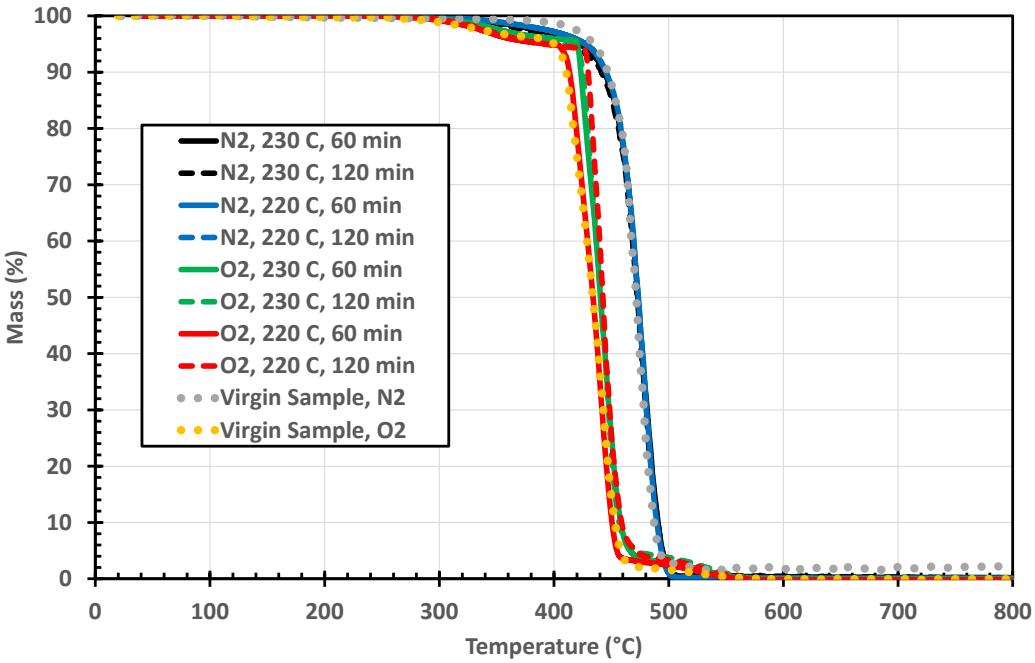

Figure 15. The experimental TGA results of XLPE in the fast-aging experiments 1-8 compared to virgin XLPE. All measurements used 10 K/min heating rate and the combined TGA/DSC runs after fast-aging were made both in O_2 and N_2 atmospheres. The O_2 aged data is scaled so that its initial mass is 100 % at the start of the TGA run after the aging period.

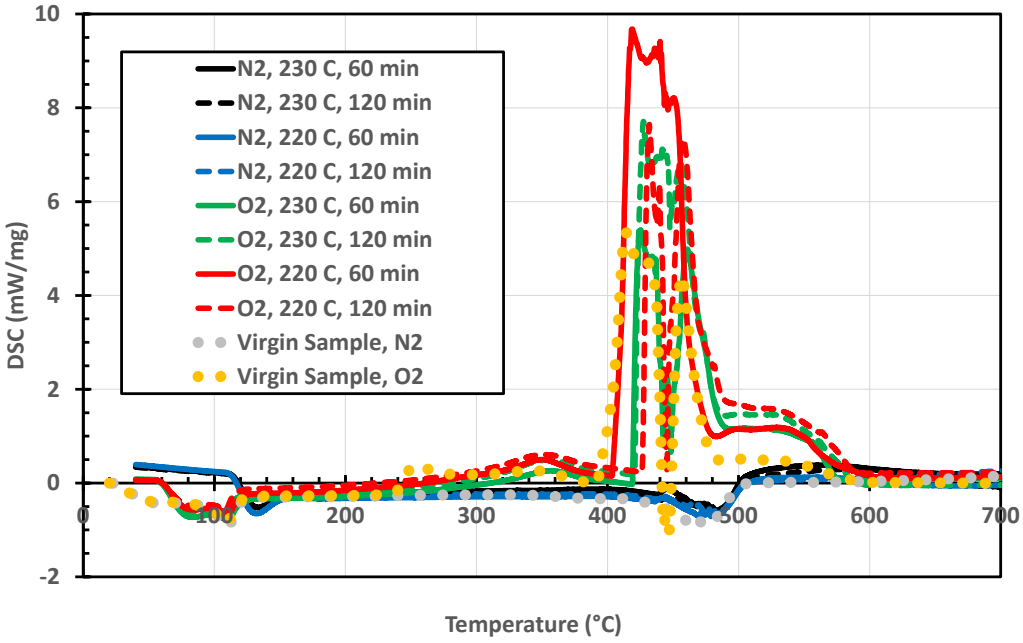

Figure 16. The experimental DSC results of XLPE in the fast-aging experiments 1-8 compared to virgin XLPE. All measurements used 10 K/min heating rate and the combined TGA/DSC runs after fast-aging were made both in O_2 and N_2 atmospheres.

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5. Conclusions

This report summarizes Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) experiments of samples taken from XLPE sheets. Both non-aged (virgin) and thermally aged samples were studied.

The virgin XLPE results are in line with the previous URAN project results and findings in the literature. The fast-aging results are qualitatively in line with the few related findings from the literature, where some information of fast-aging type experiments for PE (not XLPE) were found.

The aim of the work was to provide experimental data for cable models that can be built to carry out small scale FDS simulations concerning TGA, and later cone calorimeter. The current work is continued later in the FASAANI project and cone calorimeter experiments are to be done for the non-aged (virgin) XLPE sheets. The overall results from such tests will facilitate the development of complete FDS-based material models to carry out full scale cable fire simulations representing a real cable fire. It was noted in the previous study (URAN project) that a thorough micro-scale experimental information is needed in order to generate realistic material models for XLPE pyrolysis to be used in the CFD based fire simulations. Thus, the presented TGA/DSC experiments use different heating rates, different atmospheres (O_2/N_2), and a range of fast-aging samples to estimate the O_2 reactions during the aging process giving a wider coverage of possibilities of thermal degradation of XLPE materials which can be used for building computational material models for further work in future.

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