The Effect of Partial Discharge Aging on the Dielectric Response of Polymers

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Abstract

This work investigated the effect of partial discharges (PD) on the complex permittivity of PVC, PC, PTFE, PE and PA6. The material samples were subjected to prolonged PD activity and the complex permittivity of the material was measured by dielectric spectroscopy (DS) before and after the sample was aged. The results showed a change in the loss factor for the tested materials, some were more affected than others and showed different behaviors when subjected to the PD aging. Hence it was not possible to find any general trend in the complex permittivity of the tested materials. Several of the materials exhibited a loss factor with -1/2 slope at low frequencies corresponding to diffusion. The -1/2 slope implies that the loss factor and the dynamic component of the real permittivity should be equal, which could not be seen. Therefore it is uncertain whether what was observed is a diffusion process or not. The results could suggest that PD aging causes a change in the trapping characteristics of the material. Furthermore, space charges deposited on the cavity wall from the PD activity could diffuse into the bulk of the material where some of the charges are trapped. If a DS measurement is performed before the trapped charges have had time to recombine or be conducted away, it could affect the complex permittivity. This was demonstrated by performing another DS measurement on an aged PC sample which had been left to rest for 35 days. The measurement showed that the complex permittivity had returned to almost the same state as before aging. Measurement problems were encountered which affected the measurements in this work. The problems are believed to be caused by the measurement electrodes used for the DS measurements not being heavy enough to eliminate small unwanted air-gaps between the electrodes and sample. Furthermore, incomplete results were obtained for some measurements which was believed to be due to the capacitance of the sample being at the limit of what the measurement instrument was able to measure.
Sammanfattning

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Chapter 1

Introduction

1.1 Background

Most people are familiar with the proverb “A chain is not stronger than its weakest link” which says that if one link in the chain is weak, then the whole chain is weak. This saying is also true for electrical systems and installations. Here the weakest link is commonly the electrical insulating, or dielectric, material [1]. Add to this that in power engineering applications the insulation should be designed to have a service lifetime of at least 40 years [1]. The choice of dielectric material must therefore be made after careful consideration based on the intended use and what stresses it will be subjected to. Stresses put on the dielectric causes a gradual change of the material, this is known as aging. Aging of the material generally makes it less able to withstand the stresses to which it is subjected [2]. Hence, it’s important to know the basic properties of the dielectric material during design so it is able to withstand the stresses from normal operation and the surroundings for its entire service time. If the insulation fails it could have dire consequences, leading to the failure of that component or the entire system. A popular tool to use during design is the TEAM acronym [2], which stands for

- **Thermal**, the material must be able to conduct away heat generated by resistive losses in the conductors and in the dielectric material. For that reason the thermal properties of the material, e.g. thermal conductivity and maximum operating temperature must be known.

- **Electrical**, electrical properties like dielectric constant, dielectric losses and dielectric breakdown strength. Also the material must be strong enough to withstand faults in the system.
• **Ambient**, the environmental impact on the dielectric material is important to know. This can range from exposure to rain and salt in outdoor installations to water penetration in underground cables.

• **Mechanical**, mechanical properties of the material. Often the insulation acts as a mechanical support structure that separates high potential from low potential or ground so the mechanical strength of the material must in that case considered. It must also be able to withstand the forces generated during a fault.

The TEAM acronym is used to weigh different design considerations against each other to find the right balance between them. E.g. materials with very good electrical insulation properties also tend to be very poor thermal conductors [1]. If the temperature increase the aging of the material will speed up, e.g. the dielectric breakdown strength of the material will decay faster. However, this thesis will only deal with the electrical properties of insulation materials as it ages. More precisely it will deal with how the complex permittivity changes when it is subjected to accelerated aging caused by partial discharges (PD) in voids embedded in the dielectric. In previous work [3] the author investigated how the dielectric response in oil-impregnated paper insulation change when it was subjected to high voltage impulses and prolonged PD activity in enclosed voids. The study concluded that an aged zone is created as a result of the PD activity around the cavity and it will grow with time of PD application due to PD by-products deposited on the cavity walls penetrating into the bulk of the material. Voids in insulation occurs due to cracks, delimitation, enclosed gas bubbles and other defects [4] and PD in the void causes erosion and degrades the dielectric. PD induced degradation, or aging, of the dielectric is roughly due to a combination of chemical reactions and charge carrier bombardment of the cavity surface [5]. This eventually leads to tree growth and which ultimately leads to failure of the insulation system or the specific component. The prevailing problem of dealing with PD aging of solids and part of the complexity is due to the fact that the dielectric is aged due to PD activity but at the same time the PD mechanism is affected by the aging dielectric [5].

### 1.2 Aim

The aim of this thesis is to investigate PD induced aging in polymers and determine if and how the dielectric properties change by means of dielectric spectroscopy (DS) measurements. And if there is a change to try to provide a possible explanation on the mechanisms behind it.
1.3 Method

The method used to age the material and measure it is as follows

- 5 different materials will be tested; PA6, PC, PE, PTFE and PVC.
- Create samples with enclosed cavities by cutting three discs and making a hole in the middle disc and then sandwiching it between the other two.
- Age the material using partial discharges for a certain time.
- If possible measure PRPD patterns during aging.
- Measure the dielectric response using dielectric spectroscopy before and after aging on the top and bottom disc separately.
- Process the data and analyze the results.

1.4 Thesis Outline

Chapter 1 provide a brief background, aim and method of the thesis. Chapter 2 describes the background theory about polarization and polarization mechanisms. The concept of dielectric response is introduced and explained. Then there is a description of partial discharges, gas discharges and degradation of dielectrics due to partial discharges and thermal runaway. Lastly the materials tested in this thesis is introduced. In Chapter 3 the measurement circuits used for the the measurements are presented. Chapter 4 presents the measurement results and analysis of them. Chapter 5 contains the conclusions and suggestions on future work.
Chapter 2

Theory

2.1 Polarization

If a conductor is placed in an electric field the free charges inside the material redistribute on the conductor surface until an equilibrium is reached where the electric field inside the conductor is zero [6]. In an ideal dielectric material on the other hand, the charges are not free to move. Instead they are bound to molecules and atoms and can only move a small distance [1, 6]. If the dielectric is placed in an external electric field a small displacement of the nucleus and the electron cloud will occur [1, 2, 6]. The material has become polarized. This small displacement of charge densities give rise to a microscopic dipole moment, \( p_i \), which is usually unknown and not of interest. Of larger interest is how the material behaves on a macroscopic level, this is done by averaging all the microscopic dipole moments to obtain a macroscopic polarization vector \( \mathbf{P} \) [7].

\[
\mathbf{P} = \lim_{\Delta V \to 0} \frac{1}{\Delta V} \sum_{i=1}^{N} p_i
\]  

(2.1)

Where the subscript \( i \) refers to the i:th atom or molecule and \( \Delta V \) refers to a small volume where the microscopic dipoles are contained.

2.2 Polarization Mechanisms

2.2.1 Electronic Polarization

If an atom is placed in a external electric field there will be a displacement between the positively charged nucleus and the negatively charges electron cloud [8]. If the electric
field is strong enough it will pull the atom apart and ionize it. Otherwise, the nucleus and electron cloud will become displaced until an equilibrium is reached where the force from the external field trying to pull the atom apart and the attracting force between nucleus and electron cloud are equal [9]. The atom is now polarized, see figure 2.1. This process is extremely fast and able to follow temporal variations in the electric field up to optical frequencies [10].

![Electronic polarization](image)

**Figure 2.1:** Electronic polarization. Left atom show when no electric field is present. The right atom show the displacement of the nucleus and electrons when the electric field is present. Source: [11].

### 2.2.2 Atomic and Ionic Polarization

Atomic polarization occurs when atoms in a molecule are displaced due to an external electric field. A similar process is ionic polarization where the electric field displaces positive and negative ions in an ionic lattice, e.g. in NaCl [8]. Because atoms and ions has greater mass than single atoms this process is slower than electronic polarization. Typical time constants for atomic/ionic polarization are between $10^{-15}$ s and $10^{-13}$ s [8].

### 2.2.3 Rotational Polarization

Rotational polarization occurs when molecules with a permanent or induced dipole moment are present. An example is the water molecule shown in figure 2.2 which consist of one oxygen atom and two hydrogen atoms which do not lie on diametrically opposite sides of the atoms, this results in a net polarization that is non-zero [6, 9]. Normally the molecules are randomly oriented due to thermal motion but in the presence of an external electric field they align with the field to some extent [10]. This process is also quite fast with time constants around $10^{-9}$ s [1].

### 2.2.4 Interfacial Polarization

Interfacial polarization occurs in materials containing different dielectric materials such as oil-impregnated paper. The difference in the permittivities between the dielectrics
forces movable charges to become attached to the boundaries between the materials [10]. This is typically a slow process with time constant of seconds or longer [8].

2.2.5 Polarization by hopping charge carriers

This is mostly found in solids in which local hopping sites are present in which the charge carriers, electrons and ions, spend most of their time. Due to thermal excitation there is a finite probability that a charge carrier jump over or tunnel through the potential barrier surrounding the hopping site into a nearby site [7]. If there is a network of connected sites so the charge carriers are able to traverse the entire dielectric it will contribute to a DC conduction current [7, 10]. In the presence of an electric field, the probability of hopping transitions taking place will change but the transitions themselves are still due to thermal excitation [7].

2.3 Dielectric Response

The dielectric response of a material is its ability to follow temporal variations in the driving electric field [1]. Depending on the frequency of the electric field not all dipoles will have the ability to follow the electric field. When the frequency increases the slower dipoles will not be able to keep up with the time-varying electric field and as the frequency continues to increase, more and more dipoles is unable to follow the electric field. When the electric field reaches optical frequencies and above not even the induced electronic dipoles will be able to follow the variations in the electric field and the permittivity settles to unity [1].

In free space the electric displacement vector, or electric flux density, is defined as

\[
D = \varepsilon_0 E
\]  

(2.2)
Where \( \varepsilon_0 \) is the permittivity of free space and \( \mathbf{E} \) the electric field strength. The significance of the displacement field, \( \mathbf{D} \), is that it accounts for the presence of free charges which are deposited at the surface of the electrodes \([2, 10]\). If a material is present \( \mathbf{D} \) increases due to polarization and equation (2.2) is re-written to account for this by adding the macroscopic polarization vector from equation (2.1) to equation 2.2.

\[
\mathbf{D} = \varepsilon_0 \mathbf{E} + \mathbf{P}
\]  

(2.3)

As discussed in section 2.2 the different polarization mechanisms have different time constants. Some are very fast, like electronic polarization, while others are very slow, like interfacial polarization. Therefore it is common to separate the polarization into two parts. One which accounts for polarization processes which, for a specific application, is considered instantaneous and one slower part due to processes which is considered non-instantaneous.

\[
\mathbf{P}(t) = (\varepsilon_\infty \mathbf{E}(t) + \int_0^\infty f(\tau)\mathbf{E}(t - \tau)d\tau)\varepsilon_0
\]  

(2.4)

Where \( \varepsilon_\infty \) is the high-frequency permittivity signifying all polarization processes which can be considered infinitely fast. \( f(t) \) is called the dielectric response function and it describes the dynamic response of the material. The description of the dielectric response has up until this point been in the time domain. But if the excitation is of sinusoidal type it might be convenient to treat the response in the frequency domain instead. A new function is then introduced, called the frequency dependent, or complex permittivity, which is related to the dielectric response function through the fourier transform.

\[
\tilde{\varepsilon}(\omega) = \varepsilon_r(\omega)\varepsilon_0 = (\varepsilon'_r - j\varepsilon''_r)\varepsilon_0 = (\varepsilon_\infty + \Delta \varepsilon'_r - j\varepsilon''_r)\varepsilon_0
\]  

\[
= (\varepsilon_\infty + \int_0^\infty f(t)e^{-j\omega t}dt)\varepsilon_0
\]  

(2.5)

Where \( \Delta \varepsilon'_r \) represent the dynamic component of the real permittivity. The real part of the relative permittivity, \( \varepsilon'_r \), is simply called the dielectric constant and the imaginary part, \( \varepsilon''_r \), is called the dielectric loss factor. The introduction of the complex permittivity is done in order to account for dielectric losses caused by the non-momentary polarization processes. It’s now possible to write equation (2.3) as
If an electric field, \( E \), is applied to a dielectric it will give rise to a current density in the dielectric material which can be expressed as a sum of a conduction current and a displacement current

\[
J(t) = \sigma_{DC}E(t) + \frac{\partial D(t)}{\partial t}
\]  

(2.7)

\( \sigma_{DC}E(t) \) represent a conduction current density due to free charges. This is a simple model of the conduction current which assume a linear relationship between the current density and the electric field \([1, 10]\). \( \frac{\partial D(t)}{\partial t} \) is a displacement current density due to the motion of induced and permanent dipoles by the polarization \([1]\). Transformed to the frequency domain (2.7) becomes

\[
J(\omega) = \sigma_{DC}E(\omega) + j\omega D(\omega)
\]  

(2.8)

Using (2.6) and the relations in (2.5) and separating the real and imaginary part yields

\[
J(\omega) = (\sigma_{DC} + \omega \varepsilon'' \varepsilon_0)E(\omega) + j\omega \varepsilon_0 \varepsilon'_r E(\omega)
\]  

(2.9)

The first parenthesis represents the loss current density in the dielectric material and the imaginary part represent a pure displacement current density \([1]\). Figure 2.3 show the current density components in a phasor diagram.

---

**Figure 2.3:** Phasor diagram of the current density in the dielectric and the driving electric field.
It is seen that the total current density is shifted an angle $\delta$ from being completely capacitive. This angle, $\delta$, is called the loss angle and the tangent of this angle, $\tan(\delta)$, is called the dissipation factor.

$$\tan(\delta) = \frac{J_{\text{Loss}}}{J_{\text{Displacement}}} = \frac{\varepsilon''(\omega) + \frac{\sigma_{\text{DC}}}{\omega \varepsilon_0}}{\varepsilon'_r(\omega)}$$ (2.10)

In (2.9) it is seen that there exist a conductivity term in the dielectric losses due to DC conduction. The measurement instrument is unable to differentiate the conduction losses from the polarization losses at a single frequency. Therefore an apparent loss factor is commonly introduced [1].

$$\varepsilon''_{r,\text{App}} = \frac{\sigma_{\text{DC}}}{\omega \varepsilon_0} + \varepsilon''_r$$ (2.11)

As seen from (2.11) the conductivity will start to dominate when the frequency gets sufficiently low and in the logarithmic scale it is a straight line with a slope of -1 which is superposed to $\varepsilon''_r$. Another commonly used notation is the complex capacitance defined as

$$\tilde{C}(\omega) = C'(\omega) - jC''(\omega) = C_0 \tilde{\varepsilon}(\omega)$$ (2.12)

Where $C_0$ is called the geometric capacitance. This is the capacitance between the electrodes if there would have been free space in between [2, 10]. In this thesis the geometrical capacitance of the electrode arrangements is that of a parallel plate capacitor

$$C_0 = \varepsilon_0 \frac{A}{d}$$ (2.13)

Where $A$ is the area and $d$ the distance between the electrodes.

### 2.4 Partial Discharges

In the manufacture of polymeric materials it is very difficult to completely prevent any voids from forming [12, 13]. The type of void considered in this thesis is air-filled cavities completely enclosed by insulation. The permittivity of the cavity is lower than the surrounding dielectric so the electric field is enhanced in the cavity. For the cavity geometry used in this thesis the enhanced field, $E_{\text{cavity}}$, inside the cavity increases with a factor of the permittivity of the surrounding dielectric
\[ E_{\text{cavity}} = \varepsilon_r \text{dielectric} E_{\text{dielectric}} \]  

Due to the field enhancement and the fact that the air-filled cavity has a lower breakdown strength than the surrounding dielectric the cavity will generally breakdown before the dielectric as the voltage is raised. This discharge is called a partial discharge (PD) since it only partially bridges the insulation between the electrodes. IEC standard 60270 defines a partial discharge as [14]:

"localized electrical discharge that only partially bridges the insulation between conductors and which can or can not occur adjacent to a conductor."

The charges liberated by a discharge is deposited on the cavity walls where they slowly dissipate. The electric field from this surface charge density is superposed to the external electric field and depending on the polarity of the alternating external field it might come to a constructive or destructive superposition [13]. Consecutive discharges follow the total field within the void and not the external field. The next discharge occurs when the electric field is again above the breakdown limit. The consequence of this is that a discharge can occur even if the external voltage is zero because the field in the cavity from the surface charges may exceed the inception field [15]. The sequence of breakdowns in a cavity is shown in figure 2.4.

Figure 2.4: Breakdown of a cavity under AC voltage. \( V_c \) shows the cavity voltage waveform if no breakdown occurs, \( V_{cd} \) show the actual cavity voltage and \( V_a \) show the external applied voltage waveform. Source [3].
2.4.1 Gas Discharges

An electrical breakdown is a change from an insulating state of the gas to a conducting state where the voltage across the cavity become close to zero. This conductive state is reached through ionization of the gas. The ionization process starts with a free electron [10] that is accelerated in the electric field. The generation rate of seed electrons to start an avalanche, which are provided by thermal and field-supported emission out of the insulation surface, depend on the material and on the surface characteristics. The pd inception voltage and the complete pd process is thus determined by the surrounding dielectric [13]. If the electron obtain a high enough kinetic energy to overcome the ionization energy of the gas molecules it can upon collision with a molecule cause ionization of that molecule, thereby freeing another electron which is accelerated in the field. This is the beginning of an electron avalanche. The kinetic energy of the electron depends on the electric field and the mean free path, which is the mean path between collisions [10, 12].

\[ \Delta W_e = eE \lambda_e \propto \frac{E}{p} \]  \hspace{1cm} (2.15)

2.4.1.1 Townsend Discharge

The Townsend model of a discharge is that of a self-sustaining avalanche [12]. The generation rate of free electrons from impact ionization is given by Townsend’s first ionization coefficient, \( \alpha \), which is defined as the number of electrons produced by an electron per unit length of path in the direction of the electric field [10]. The number of electrons reaching the anode can be written as

\[ n = n_0 \epsilon^\alpha d \]  \hspace{1cm} (2.16)

Where \( n_0 \) is the number of electrons generated at the cathode. \( \alpha \) is commonly replaced with an effective ionization coefficient \( \overline{\alpha} \) [2]

\[ \overline{\alpha} = \alpha - \eta \]  \hspace{1cm} (2.17)

Where \( \eta \) is an attachment coefficient that describe the reduction in number of electrons per unit distance due to recombination with positive ions or attachment to electronegative gases, if such are present. In order for the discharge process to become self-sustained new electron avalanches must be able to start after the electrons of the
first avalanche has drifted across the cavity. Thus, new seed electrons must be emitted from the cathode. Causes of secondary emissions include positive ion bombardment of the cathode, photoelectric emission at the cathode and photoelectric ionization in the gas itself [10, 12]. Townsend’s secondary ionization coefficient, $\gamma$, is defined as the mean number of electrons generated by all secondary mechanisms [2]. The Townsend criterion for a self-sustained discharge can be shown to be [2, 10]

$$\frac{\alpha \gamma}{\alpha} (e^{\sigma d} - 1) = 1$$

(2.18)

During the course of the self-sustained discharge charge is transferred across the cavity and deposited on the surfaces where it slowly dissipate. The deposited charges will set up an opposing field in the cavity and as more charge is deposited the cavity voltage will decrease until the discharge can no longer be maintained [12].

### 2.4.1.2 Streamer Discharge

The Townsend mechanism is valid as long as the electric field of the space charges of electrons and ions can be neglected in relation to the external electric field [10]. A streamer head is a concentration of electrons which set up an electric field which is comparable in strength to the external electric field [2]. For this to happen the required concentration of charges in the streamer head is about $10^8$ [2]. For the accumulation of this charge concentration to be possible the streamer criterion needs to be fulfilled

$$\pi \sigma \gamma \geq 18$$

(2.19)

When the charge concentration exceeds this value the electric field in the cavity is modified. The field before the streamer head is enhanced and electrons in front of the streamer head therefore forms strong avalanches which neutralize the region that was the streamer head and charge a region further along. The streamer head thus moves forward faster than the actual electrons can travel, as a wave of ionization [2]. Secondary electrons are generated through a photoionization process from recombinations of electrons and ions generated in front of the streamer head. The secondary electrons are accelerated in the field and develop into secondary avalanches. Since photons travel at the speed of light this process leads to a rapid development of a conduction channel across the cavity [10].
2.5 Partial Discharge Degradation

PD in cavities in solid insulation can be detrimental to the total lifetime of that component because PD activity lead to an accelerated aging which deteriorates the insulation, and ultimately lead to insulation failure. The aging mechanisms of PD can be said to fall into two categories, those initiated by ion bombardment or by chemical reactions [5, 12]. Early signs of aging is an increase of the conductivity of the cavity surface due to reaction processes of humidity and the dissociation products of air caused by the pd inside the cavity [5]. If the pd activity is continued the cavity surface will become rougher due to the particle bombardment and deposition of chemical by-products on the surface. Eventually solid by-products will start to form in the shape of localized crystals. This leads to field enhancements and an increased PD activity at these locations, now the formation of pits and craters can usually be observed [5]. This is a beginning of electrical treeing which will propagate through the material and eventually lead to breakdown of the insulation. In [3] the degradation due to PD and high voltage impulses was investigated for oil-impregnated paper. The author proposes the division of the material into non-aged and aged zones. The material around the cavity ages with time of PD application due to PD by-products penetrating into the bulk, this has also been a working hypothesis for this thesis to see if polymeric materials also form aged and non-aged zones.

2.6 Thermal Runaway

Conduction of free charges and the polarization of dipoles in the dielectric causes heating of the dielectric material which will cause a temperature rise in the material. The dielectric must be able to conduct away the generated heat, otherwise it will lead to a thermal runaway. The balancing equation can be written as [10]

\[ \sigma E^2 = C_v \frac{dT}{dt} - \nabla \cdot (K \nabla T) \] (2.20)

Where \( C_v \) is the thermal capacity of the dielectric, \( K \) is the heat conductivity and \( \sigma \) the electrical conductivity. The electrical conductivity contains one part representing the DC-conductivity and one part representing polarization losses. The term to the left of the equal sign represent the heat generated in the material, the first term on the right of the equal sign represent heat absorbed by the dielectric and the second term represent the heat lost to the surrounding by thermal conduction. The conductivity of the dielectric is usually exponentially dependent on the temperature [12] so when the
temperature increase the conductivity will increase exponentially. Which means more heat is dissipated in the dielectric as the current density increase. This serves to further increase the temperature and hence the conductivity. This process is called thermal runaway and will lead to breakdown of the dielectric material [12].

2.7 Materials

PA6 and PTFE were chosen because of their respective placement in the Triboelectric series. The other materials were chosen based on what was available in the lab.

2.7.1 Polyamide 6, PA6

PA6 is a material belonging to the polymer group of materials called polyamides or Nylons. The material is made up of repeating amide, -CO-NH-, links. There is a large variety of Polyamides with tailored properties to fit a wide range of applications. It is used in a large number of applications from everything from car parts to electrical insulation. PA6 has the chemical formula (C_{10}H_{11}NO)_{n}, and the repeating unit is shown in figure 2.5.

![Figure 2.5: Repeating unit of PA6. Source: [16]](image)

Some of the properties that make it so popular is its high mechanical strength, good high temperature performance, good flammability properties, good chemical resistance and good electrical properties ($\varepsilon'_r = 4.2$, $\tan(\delta) = 0.03$ at 100 Hz) [17].

2.7.2 Polycarbonate, PC

PC is a transparent thermoplastic material. The repeating unit, see figure 2.6, has the chemical formula (C_{16}H_{14}O_{3})_{n}

PC has very good mechanical properties. Its impact strength is very high compared to other materials. It only has moderate thermal properties with a limit temperature of 120 °C [1]. It has good dielectric properties with $\tan \delta = 0.01$ and $\varepsilon'_r = 2.9$ at 1 MHz [19]. Due to its good dielectric properties, heat-resistant and flame-retardant properties
it is often used in electronic and communication products. PC has the property that it can be colored and still be transparent, it is therefore extensively used in power tools and other applications where its good mechanical strength comes into good use [1].

### 2.7.3 Polytetrafluoroethylene, PTFE

PTFE which is more commonly known as Teflon is a fluorocarbon polymer, which means that it only consist of fluor and carbon atoms in long chains. Its molecular formula is \((C_2F_4)_n\) and its repeating unit is shown in figure 2.7. PTFE is an extremely linear polymer with a crystallinity up to 95 %, which is the reason for its very good thermal properties [1]. Its high melting point of 327°C and its very low coefficient of friction makes it well suited for its probably most well known application, namely as a non-stick coating in pans and cookware. It also has very good dielectric properties with \(\varepsilon'_r = 2.1\) over a wide frequency range and a very low dissipation factor of 0.0004 or below over a frequency range up to \(10^8\) Hz [20]. Due to the high degree of crystallinity and that the needed thermal treatment and processing is difficult and requires special methods it’s not very common for PTFE to be used as electrical insulation in large scale [1].

![Figure 2.7: Repeating unit of PTFE. Source: [21]](image)

### 2.7.4 Polyethylene, PE

PE is the most common plastic with its primary use as plastic bags, bottles and other sorts of packaging. PE is also very important when it comes to electrical insulation and is one of the most commonly used insulation materials. Its chemical formula is \((C_2H_4)_n\) and the repeating unit is shown in figure 2.8. PE comes in different forms: Low Density
PE (LDPE), High Density PE (HDPE) and cross-linked PE (XLPE) which today is the most commonly used insulation material in high-voltage cables with rated voltages up to 500 kV AC and 150 kV DC [1]. The difference between LDPE and HDPE can be said to be the density of the material and the degree of crystallinity [1]. PE has good dielectric properties with \( \varepsilon' = 2.3 \) over a wide frequency range and \( \varepsilon'' = 0.0003 - 0.0007 \) [1].

![Figure 2.8: Repeating unit of PE. Source: [22]](image)

### 2.7.5 Polyvinyl chloride, PVC

PVC is one of the most common plastics after polyethylene, PE, and polypropylene, PP. Its chemical formula is \((C_2H_3Cl)_n\) and its repeating unit is shown in figure 2.9. Due to its low flammability it’s commonly used in indoor cords and as jacketing material for cables laid in ducts. The low flammability is due to the high chlorine content in PVC, but this also has the negative effect that when it does burn it decomposes into HCl which is highly toxic [1]. PVC has very limited use as high voltage electrical insulation because of its high \( \tan(\delta) \) that can vary between 0.08 to 0.15 [23] and the dielectric constant is 3.2 at 60 Hz. Therefore it’s rarely used as insulation for voltages above 1 kV-10 kV [1, 23].

![Figure 2.9: Repeating unit of PVC. Source: [24]](image)
Chapter 3

Measurement Setups

3.1 Sample Preparation

In this thesis aging due to PD activity in insulation enclosed cavities is investigated and for this reason the samples were made by cutting three separate discs. A top, bottom and spacer disc. The cavity was then produced by making a hole in the spacer disc and sandwiching it between the top and bottom disc, which are shown as the purple discs in figures 3.2 and 3.3B. In the short aging tests the same material was used for all discs, e.g. when testing PA6 all three discs were cut from sheets of PA6. In the long aging tests the spacer disc was made from 0.25 mm thick PC discs and the top and bottom discs were made from the material under investigation. The choice of a different spacer disc for the long aging tests was made in order to get a thinner air gap between the top and bottom discs and in that way decreasing the inception voltage for PD onset.

3.2 Test Cells

There were no electrodes available that could be used for both aging and DS measurements so two different electrode arrangements was used for this. Furthermore, during DS measurements two different test cells with slightly different electrodes was used. This was done because unwanted noise appeared in the oscilloscope figure of the current during measurements with test cell 1, see figure 3.1A, which affected the results negatively. Therefore another test cell, manufactured by Keithley Instruments Inc. and shown in figure 3.1B, was used instead for all materials except one in the long time aging tests in chapter 4. The Keithley cell connects to the electrometer via coaxial cables instead, then the noise in the current waveform disappeared.
3.2.1 Electrodes Used For Dielectric Spectroscopy

Two different sets of electrodes was used during the DS measurements. The general setup is shown in figure 3.2 and the specific data of each set of electrodes is shown in table 3.1. When performing the DS measurements on the samples, the top and bottom discs were measured separately.

![Figure 3.1: Photos of the electrodes used for the DS measurements. (A) shows Test cell 1 and (B) the Keithley test cell.](image)

**Figure 3.2:** The electrode arrangements used during DS measurements. The purple disc is the sample to be tested and the grey blocks are the metal electrodes. The top and bottom discs were measured separately. The figure is not drawn to scale.

3.2.2 Electrode Used For Aging

The electrode arrangement used during aging is shown in figure 3.3.
Table 3.1: Electrode Specifications

<table>
<thead>
<tr>
<th>Test Cell 1 [mm]</th>
<th>Keithley Test Cell [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>$l_{HV}$</td>
<td>78</td>
</tr>
<tr>
<td>$l_g$</td>
<td>13</td>
</tr>
<tr>
<td>$l_m$</td>
<td>50</td>
</tr>
<tr>
<td>$l_{LV}$</td>
<td>78</td>
</tr>
</tbody>
</table>

An epoxy resin is molded onto the base of the High-voltage electrode, which is seen in figure 3.3A and in figure 3.3B as the orange blocks at the base of the top electrode.

3.3 Dielectric Spectroscopy Analyzers

Two insulation diagnostics analyzers, IDA200 and IDAX300, was used to measure the complex capacitance of the test objects. The schematic diagram of the DS instruments is shown in figure 3.4 and the instruments are shown in figure 3.5.

The feedback component, FB(i), consist of some combination of resistive and capacitive elements which can be varied in order to give a suitable output voltage from the electrometer. The type of measurement that was used was the Ungrounded Specimen Test (UST). When this measurement type is used only the current from the measurement electrode is measured by the electrometer while the guard electrode is connected to earth.
and any unwanted current is lead away. More information about the DS instruments and measurements can be found in [2, 25]

![Schematic drawing of the dielectric spectroscopy measurement system.](image1)

**Figure 3.4:** Schematic drawing of the dielectric spectroscopy measurement system.

![Photos of IDAX300 and IDA200.](image2)

**Figure 3.5:** Photos of (A) IDAX300 and (B) IDA200.

### 3.4 Partial Discharge Measurement

#### 3.4.1 Partial Discharge Model

Consider a cavity enclosed by solid insulation as shown in figure 3.6.

In figure 3.6 the insulation and cavity can be represented by an equivalent capacitance network. $C_c$ represent the capacitance of the cavity, $C_b'$ and $C_b''$ the capacitance of the insulation in series with the cavity and $C_a'$ and $C_a''$ represent the capacitance of the insulation of the bulk parallel to the cavity. The capacitance network is simplified by writing
Figure 3.6: Model of insulation cavity with equivalent capacitance representation. Source [26].

\[ C_b = \frac{C'_b C''_b}{C'_b + C''_b} \quad (3.1) \]

\[ C_a = C'_a + C''_a \quad (3.2) \]

The magnitudes of the capacitances is controlled by the inequality \( C_a \gg C_c \gg C_b \) [10]. Now it is possible to make a circuit representation of the PD event in the insulation. This model is commonly referred to as the ABC model, see figure 3.7.

Figure 3.7: ABC Model. Source [26].

When increasing the voltage over the terminals A and B in figure 3.7 the electric field in the cavity will build up until it discharges and a PD occurs. This is modeled in the ABC model as the switch \( S \) closing and \( C_c \) discharging through the resistance \( R \). The discharge time of \( C_c \) is very short and can be seen as a voltage drop of \( \Delta V_c \) in the circuit. The voltage drop that can be measured at the terminals A and B can be found by making the Thevenin equivalent of the discharge circuit. The voltage drop at the terminals will then be

\[ \Delta V_a = \frac{C_b}{C_a + C_b} \Delta V_c \quad (3.4) \]
When making PD measurements a coupling capacitor, $C_k$, is connected to the terminals A and B in figure 3.7 in parallel with the test sample. Then during the very fast discharge process it will act as a voltage source and try to cancel the voltage drop $\Delta V_a$ by releasing a current which is proportional to the charge transfer or discharge current from the PD. If $C_k$ is large in relation to the test sample the charge transfer provided by the discharge current is completely compensated by $C_k$ [10]. This charge then becomes

$$q_{App} = C_k \Delta V_c$$

(3.5)

Notice in (3.5) that this is called the apparent charge of the PD pulse. Because the charge measured at the terminals is not equal to the charge involved in the discharge event. IEC 60270 [14] gives the definition of apparent charge as:

"apparent charge q of a PD pulse is that charge which, if injected within a very short time between the terminals of the test object in a specified test circuit, would give the same reading on the measuring instrument as the PD current pulse itself. The apparent charge is usually expressed in picocoulombs (pC)"

"NOTE The apparent charge is not equal to the amount of charge locally involved at the site of the discharge, which cannot be measured directly."

3.4.2 Measurement Circuits

3.4.2.1 PRPD Measurement Circuit

The measurement circuit used for the Phase Resolved Partial Discharge (PRPD) measurements is shown in figure 3.8 and is based one of the measurement circuit given in IEC 60270 [14]. A detection impedance, $Z_{Detect}$, is connected in series with the coupling capacitor, $C_k$. The detection impedance consist of a parallel connection of a capacitance, inductance and resistance which can be set to different values. The voltage is measured over $Z_{Detect}$ and fed into a preamplifier before it reaches the Insulation Condition Monitoring (ICM) system from Power Diagnostix Systems GmbH where the signal is processed.
3.4.2.2 Circuit Used For Long Time Aging

When performing the long aging tests in this thesis it was not possible to use the circuit from figure 3.8 due to the simple reason that it is used by several persons, and it would therefore not be feasible to use it for weeks without end. Therefore, another setup was used for the long time measurements. Unfortunately this circuit did not have the ability to measure PRPD pattern or even show the occurrence of PD in the cavity. The circuit is shown in figure 3.9. Before using this circuit for aging, the samples were placed in the circuit shown in figure 3.8 in order to find an appropriate voltage level where a good amount of PD activity is seen. Then the samples was placed in the simpler circuit from figure 3.9 and aged at the determined voltage level.
Chapter 4

Measurement Results

4.1 PVC

4.1.1 Short Time Aging

A sample of PVC was aged for 65 hours and 55 min while recording the phase resolved partial discharge (PRPD) pattern during 20 seconds every 5 minutes. The applied voltage was 18 kV peak. The PVC discs was 1 mm thick each and the cavity diameter was 8 mm. The complex permittivity was measured before and after aging using IDA200 at the frequency range 10 mHz - 1 kHz and 200 V peak.

4.1.1.1 Dielectric Spectroscopy Measurements

The dielectric spectroscopy measurements before and after aging for the top disc are shown in figures 4.1 and 4.2.

In figure 4.1A it is seen that the real part of the complex permittivity, $\varepsilon'_{r,App}$, for the top disc is fairly constant during the whole observed frequency range with only a slight increase for very low frequencies. It is also seen that the increase at low frequencies is steeper for the aged sample. If attention is instead given to the loss factor, $\varepsilon''_{r,App}$, it can be seen for both the top and bottom disc that the dielectric losses increase with aging for low frequencies while remaining unchanged for high frequencies. The results also show a distinct minimum in $\varepsilon''_{r,App}$ around 1-10 Hz for the two measured curves. If there really is a minimum there or if this is some measurement artifact is difficult to tell. In figure 4.1B it can be seen at low frequencies that the imaginary parts increases, with a corresponding increase in the real part which could be an indication of a loss peak at lower frequencies. The corresponding behavior is seen at high frequencies, the
Figure 4.1: Permittivity of PVC before and after aging for the top disc. (A) shows the complex permittivity. In (B) $\varepsilon_\infty$ has been subtracted to display the dynamic part $\Delta\varepsilon'$ of the real part of the complex permittivity.

real parts decreases at the same time as the imaginary parts increases which suggests the beginning of a loss peak.

Figure 4.2: Permittivity of PVC before and after aging for the bottom disc. (A) shows the complex permittivity. In (B) $\varepsilon_\infty$ has been subtracted to display the dynamic part $\Delta\varepsilon'$ of the real part of the complex permittivity.

The behavior of the top and bottom disc is very similar if figure 4.1 is compared to figure 4.2. The same behaviors at low and high frequencies and the loss minimum between 1-10 Hz is observed in the bottom disc as well. The loss factor for the aged curve can in both figures 4.1B and 4.2B be seen to follow a power law with exponent $-1/2$.

$$k \cdot (j\omega)^{-\frac{1}{2}}$$

(4.1)
with some constant \( k \). In the logarithmic scale this becomes a straight line with slope -1/2 and is characteristic for diffusion [27]. (4.1) can be rewritten using the relation 
\[
 j = e^{j\frac{\pi}{2}} = \cos\left(\frac{\pi}{2}\right) + j\sin\left(\frac{\pi}{2}\right)
\]
as
\[
 k \cdot \omega^{-\frac{1}{2}}(\cos\left(\frac{\pi}{4}\right) - j\sin\left(\frac{\pi}{4}\right))
\]
(4.2)

By separating the real and imaginary part it is seen that they form parallel lines in the logarithmic scale. By choosing suitable values of \( k \) lines can be fitted to the loss factor, see figure 4.3.

\[
(A) \\
(B)
\]

**Figure 4.3:** The figure show a straight line (black) with slope \(-\frac{1}{2}\) fitted to the loss factor of PVC. (A) Show the top disc and (B) the bottom disc.

Figure 4.3A show the top disc and the fitted curve show a very good fit with the loss factor belonging to the aged sample for slightly above two decades. The bottom disc in figure 4.3B also shows a good fit for the aged sample but not as good as the top disc. The fitted line does not agree very well with \( \Delta\varepsilon'_{r,App} \) for either sample or aging since according to (4.2) \( \Delta\varepsilon'_{r,App} \) should be equal to \( \varepsilon''_{r,App} \). It is seen that the non-aged sample fits very badly to the line for both the top and bottom disc which implies that the charge transport from diffusion has increased in both samples during aging.

### 4.1.1.2 Partial Discharge Pattern

18 kV was applied to the sample and after 5 min the PRPD pattern has the shape shown in figure 4.4.

After this the PD activity and magnitudes are fairly constant for some time before it starts decreasing and after 6 hours the PD magnitude falls below the threshold level and no more PD activity is recorded by the ICM. The PD magnitude then stays below the
threshold for the rest of the recorded time with only sporadical measurements showing any PD activity.

4.1.2 Long Time Aging

A new sample was prepared and aged for approximately 188 hours to see how that would affect the complex permittivity of the material. The spacer disc that was used to make the cavity was made from a thin PC sheet with thickness 0.25 mm and the cavity itself was made much larger for this aging, approximately 3.8 cm in diameter. First the PD inception voltage was pin-pointed by placing the sample in the setup shown in figure 3.8 that was used during the short time aging and checking at what voltage PD activity started, because the setup used for long time aging could not measure any PD activity. The sample was then placed in the measurement setup shown in figure 3.9. The PD inception voltage was found to be approximately 6 kV peak. The voltage used for aging was then chosen to 8.5 kV peak.

4.1.2.1 Dielectric Spectroscopy Measurements

The complex permittivity was measured once a day except during the weekend using IDAX300 and the results are presented in figures 4.5 and 4.6.

If figures 4.5 and 4.6 are considered it can be seen that the aging of the material has little to no effect on the loss factor, it varies a bit between measurements but there does not
Figure 4.5: Dielectric constant of PVC top disc at different times during long aging. (A) shows the complex permittivity. In (B) $\varepsilon_{\infty}$ has been subtracted to display the dynamic part $\Delta\varepsilon'$ of the real part of the complex permittivity and lines (purple) with slope -1/2 has been added to the plot.

seem to be any visible decreasing or increasing trend in the material. On the other hand, visual inspections of the samples show heavy discoloration of the discs and after 143 h and 32 min a very sharp smelling greenish liquid which was believed to be hydrogen chloride (HCL) acid was found on the discs inside the cavity. This liquid by-product is a result of chemical degradation inside the cavity caused by the PD. A major effect of the creation of liquid by-products is a strong increase of surface conductivity [5] which could be one of the reasons that the recorded PD activity disappears as described in section 4.1.1.2. The liquid by-products and the discoloration of the cavity surface show that aging is definitely occurring but it is not showing in the DS measurements. Therefore,
Figure 4.6: Permittivity of PVC bottom disc at different times during long aging. (A) shows the complex permittivity. In (B) $\varepsilon_{\infty}$ has been subtracted to display the dynamic part $\Delta \varepsilon_r'$ of the real part of the complex permittivity and lines (purple) with slope -1/2 has been added to the plot.

It can be concluded that, for the aging time used here, the discs have only been aged on the surface, space charges does not seem to have penetrated into the bulk of the PVC discs to any large extent nor is there any sign of tree initiation. A straight line in the logarithmic scale is fitted to $\varepsilon''_{r, App}$ and $\Delta \varepsilon_r'$ using (4.2). It can be seen in figures 4.5B and 4.6B that neither $\varepsilon''_{r, App}$ or $\Delta \varepsilon_r'$ follow the purple lines as well as the aged samples in figure 4.3. It is difficult to say if the increase is due to diffusion of charges in the discs or if it is the beginning of a loss peak located at lower frequencies. On one hand the slope of the loss factor and dynamic component is not far away from -1/2 but
on the other hand the slope of the loss factor is seen to decrease, suggesting the presence of a loss peak just below 10 mHz.

The minimum in $\varepsilon''_{r,App}$ which was observed during short time aging in figures 4.1 and 4.2 is also present here and it is much sharper in these measurements. It was suspected that some kind of measurement artifact was behind this minimum and not a property of the material. Some kind of noise was picked up during the measurements and was visible in the oscilloscope picture of the current waveform. Therefore, another measurement was performed on the discs. For the remeasurement IDAX300 and a different pair of electrodes from Keithley, see figure 3.1B, was used instead. The difference between test cell 1 and the Keithley test cell is that the latter connects to the IDAX300 via coaxial cables instead. Also approximately 1.5-2 kg of extra weight was put on the top electrode to make sure the sample lies flat against the electrode surfaces. The results of the remeasurement is shown in figure 4.7. The results from the remeasurements in figure 4.7 show no loss minimum as was seen in the previous DS measurements. Then it can with some certainty be said that the sharp minimums seen in figures 4.5 and 4.6 are measurement artifacts. It can also be seen that the results above and below this minimum correspond well to the remeasured curve. The remeasured curve shows better correspondence at high frequencies than it does at low frequencies, but the general trend at low frequencies is the same so it should still be possible to analyze the sample in both frequency regions using the obtained results in figures 4.5 and 4.6.

![Figure 4.7: Remeasurement of the complex permittivity of PVC using other measurement electrodes and extra weights. (A) shows the result for the top disc and (B) the bottom disc.](image)
Chapter 4. Measurement Results

4.2 PC

4.2.1 Short Time Aging

The PC sample was aged in the same manner as PVC in section 4.1.1. The PC sample was made up out of three discs, two with 1 mm thickness and one with 0.8 mm thickness. A 10 mm hole was made in the 0.8 mm disc, this disc was then sandwiched between the other two discs during aging to create the enclosed cavity. The sample was aged for 65 hours and 25 min at 20 kV peak and the PRPD pattern was recorded during 20 seconds every 5 minutes. The complex permittivity was measured before and after aging using IDA200.

4.2.1.1 Dielectric Spectroscopy Measurements

![Figure 4.8: Permittivity of PC top disc before and after aging. (A) shows the permittivity before and after aging. In (B) $\varepsilon_\infty$ has been subtracted to display the dynamic part $\Delta\varepsilon'_r$ of the real part of the complex permittivity and lines (purple) with slope -1 has been added to show the presence of a conduction mechanism at low frequency.](image)

The most striking feature that can be observed from figures 4.8 and 4.9 is that the loss factor for the aged sample is almost parallel and only differs by some constant from the loss factor for the non-aged sample. In figures 4.8B and 4.9B the hints of a loss peak is seen in $\varepsilon''_{r,App}$ around 0.46 Hz with a corresponding decrease in $\Delta\varepsilon'_r,App$ which indicate that it really is a loss peak around this frequency. After this small loss peak the loss factor is seen to have the characteristic -1 slope shown by the purple lines in figures 4.8B and 4.9B. This is associated with a conduction mechanism in the material at low frequencies.
Chapter 4. Measurement Results

Figure 4.9: Permittivity of PC bottom disc before and after aging. (A) shows the permittivity before and after aging. In (B) $\varepsilon_\infty$ has been subtracted to display the dynamic part $\Delta \varepsilon'$ of the real part of the complex permittivity and lines (purple) with slope -1 has been added to show the presence of a conduction mechanism at low frequency.

After the sample had been aged it was left to rest for 35 days to let any trapped charges in the material relax. Then a new DS measurement was performed. This was done to see if the material had been permanently damaged by the PD activity. If it had suffered permanent damage during aging the complex permittivity would still be different from what was measured on the non-aged sample. The results of the measurements are shown in figure 4.10.

Figure 4.10: Permittivity remeasure of PC top and bottom after 35 days. (A) shows the measurement for the top disc and (B) the bottom disc.

As can be seen from figure 4.10 the complex permittivity has virtually returned to its initial value before it was aged, which would indicate that its only the surface of the sample which is aged and the bulk of the material has not suffered any permanent damages during the time the sample was aged. The results in figure 4.8 and 4.9 is
therefore likely caused by trapped charges in the aged material from the PD activity in the cavity or injected by the electrodes.

### 4.2.1.2 Partial Discharge Pattern

**Figure 4.11**: PRPD pattern of PC, (A) shows the PD pattern just after voltage application, and (B) shows the last recorded PD pattern after 65 h and 20 min.

The PD pattern in figure 4.11A which show the first recorded PD pattern just after voltage application, and figure 4.11B which show the PD pattern after 65 h and 20 min looks very similar. In fact, the PD pattern was found to be very consistent during aging. Just after voltage application there was 42.38 recorded discharges per cycle, and at 65 h and 20 min it has decreased a little to 39.4 discharges per cycle, so as can be seen the mean discharges per cycle is almost the same during aging. The mean PD magnitude was a bit less just after voltage application compared to after 65 h and 20 min.

### 4.2.2 Long Time Aging

A new PC sample was prepared for the long time aging test. The top and bottom disc had a thickness of 0.75 mm. The spacer disc was also made from PC but with a thickness of only 0.25 mm. A 3.9 - 4 cm hole was cut in the spacer disc to form the cavity. The discs were then sandwiched together and aged for 139 h and 30 min at 8.5 kV peak. The complex permittivity was measured once per day, except during the weekend, using IDAX300 between 1 kHz and 10 mHz at 200 V peak.
4.2.2.1 Dielectric Spectroscopy Measurements

Figures 4.12 and 4.13 show a large difference at high frequencies in the loss factor for different aging times, whereas at low frequencies they all tend towards a similar value. In other words the losses at low frequencies remains virtually the same despite being aged while the losses increase at high frequencies when it is aged.

![Figure 4.12: Permittivity of PC top disc at different times during long aging. (A) shows the complex permittivity. In (B) $\varepsilon_\infty$ has been subtracted to display the dynamic part $\Delta\varepsilon'_r$ of the real part of the complex permittivity.](image)

After 92 h and 40 min a small loss peak is seen around 20 Hz for both discs. For the bottom disc the loss peak is still faintly visible after 116 h and 20 min, while for the top disc it has disappeared again. After 139 h and 30 min the loss peak has disappeared in
Figure 4.13: Permittivity of PC bottom disc at different times during long aging. (A) shows the complex permittivity. In (B) $\varepsilon_{\infty}$ has been subtracted to display the dynamic part $\Delta\varepsilon'_r$ of the real part of the complex permittivity.

Both discs. It can be seen at low frequencies that the loss factor for all aging times follow a straight line with slope $-1/2$ according to (4.2) which is an indication of diffusion in the material, see figures 4.12B and 4.13B. The loss factors is seen to follow the purple line rather well for almost one decade. It can also be seen that the discs still follow this line when it is non-aged, and therefore not something incurred by the aging process. This result is different from the short time aging where the loss factor was found to exhibit a -1 slope behavior at low frequencies, see figures 4.8B and 4.9B. Perhaps the difference could have something to do with the different voltage levels used during aging. For the short time aging 20 kV peak was used and for the long time aging 8.5 kV peak was used.
instead. Or it could have something with that different electrodes was used for the short time measurement and the long time measurement.

4.3 PTFE

4.3.1 Short Time Aging

The discs was made from a 1 mm thick sheet of PTFE and the spacer disc had an 8 mm hole was drilled trough it to make up the cavity. The complex permittivity was measured using IDA200 before and after aging on the top and bottom discs. The sample was then aged for 72 hours at 15 kV peak and the PRPD pattern was recorded during 20 seconds every 5 minutes.

4.3.1.1 Dielectric Spectroscopy Measurements

The results from the spectroscopy measurements are shown in figures 4.14 and 4.15.

The blue line representing the aged sample has some frequencies where a negative loss was recorded by the IDA200 and since the plots are made using a logarithmic scale in Matlab all negative numbers are ignored. The recorded negative losses are likely due to limitations in the IDA200 where the measured value of the capacitance is right at the limit of what the equipment can measure. Therefore it is not possible to say anything about the behavior of the material between these frequencies. The results show that the
loss factor is the same at low frequencies for both the non-aged and aged sample, while there is a small increase in the loss factor at high frequencies. At low frequencies the loss factors of the non-aged and the aged discs exhibit a -1/2 behavior given by (4.2), see figures 4.14B and 4.15B. At high frequencies the loss factor appears to settle on a constant level in figures 4.14B and 4.15B, at least for frequencies just above 1 kHz.

4.3.1.2 Partial Discharge Pattern

Figure 4.16A shows the PD pattern just after voltage application. The recorded PD patterns exhibit a large distribution in phase for the whole aging time. Although the discharges are more concentrated in figure 4.16A than in figure 4.16B. It can be seen that the first PD in every half cycle tend to have larger magnitude than the rest of the PD’s and are concentrated to a narrow frequency band around the voltage zero-crossing, this is seen most clearly in figure 4.16A but is also visible in the other two. This suggests that these first large magnitude discharges are caused by the field inside the cavity from charges deposited on the surface from previous discharges, since the external voltage has not had sufficient time to increase enough for a discharge to occur.

4.3.2 Long Time Aging

Another PTFE sample was prepared for the long time aging of the material. The top and bottom discs of the sample was cut from a 1mm thick sheet of PTFE and the spacer disc was cut from a 0.25 mm thick sheet of PC. A hole with a diameter of 3.9-4 cm was
cut from the spacer disc. The sample was placed in the measurement setup shown in figure 3.8 and the PD inception voltage was pin-pointed. It was found to be around 10 kV peak for this PTFE sample. The sample was then placed in the measurement setup shown in figure 3.9 and aged at 13 kV peak. The complex permittivity of the top and bottom discs were measured before aging started using IDAX300 and then the sample was taken out of the aging setup once per day, except during the weekend, and a new spectroscopy measurement was performed.

### 4.3.2.1 Dielectric Spectroscopy Measurements

The IDAX300 was set at 200 V peak and the complex permittivity was measured between 10 mHz-10 kHz. The results for the top and bottom discs are shown in figures 4.17 and 4.18.

In both figures 4.17 and 4.18 sharp minimums are seen at 1 kHz and 100 Hz. No change in $\Delta\varepsilon'_r,App$ was observed in figures 4.17B and 4.18B to support the presence of such a loss minimum in the loss factor, in fact $\Delta\varepsilon'_r,App$ is more or less constant during the whole measured frequency range. Therefore, the minimum is more likely caused by switching of feedback components in the IDAX300 or some other measurement error. As can be seen in figures 4.17 and 4.18 no large change can be seen in $\varepsilon''_r,App$ until 107 h where there is an increase in the loss factor and the increase is larger for low frequencies. At low frequencies the loss factor is seen to have a -1/2 slope, this behavior is shown in figures 4.17B and 4.18B where (4.2) has been added to the plots as the purple straight lines which, as mentioned before, is an indication of a diffusion process in the material.
Figure 4.17: Permittivity of PTFE top disc at different times during long aging. (A) shows the complex permittivity. In (B) $\varepsilon_\infty$ has been subtracted to display the dynamic part $\Delta\varepsilon'_r$ of the real part of the complex permittivity and straight lines (purple) with -1/2 slope have been added.

4.4 PE

4.4.1 Short Time Aging

Three discs of PE was cut from a 1.3 mm thick sheet of PE. In one of the discs a 8 mm hole was drilled. It was then sandwiched between the two other discs to form the sample. Before and after aging the complex permittivity of the top and bottom discs were measured using IDA200. The sample was then aged for 66 hours and 55 minutes
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Figure 4.18: Permittivity of PTFE bottom disc at different times during long aging. (A) shows the complex permittivity. In (B) $\varepsilon_{\infty}$ has been subtracted to display the dynamic part $\Delta\varepsilon'$ of the real part of the complex permittivity and straight lines (purple) with -1/2 slope have been added.

at 16 kV peak in the measurement setup shown in figure 3.8. The PRPD pattern was recorded during 20 seconds every 5 minutes.

4.4.1.1 Dielectric Spectroscopy Measurements

The DS measurements was done using the internal 200 V peak source of the IDA200 between frequencies 10 mHz-1 kHz. The results of the measurements before and after are shown in figures 4.19 and 4.20.
As can be seen from figures 4.19 and 4.20 the loss factors for both the before and after measurements are incomplete. Likely causes to this problem are the fact that PE is a low loss material, the discs has a relatively large thickness and the measurement electrode has a relatively small diameter, see table 3.1. This gives a small geometrical capacitance according to (2.13), which is right at the border of what IDA200 can measure. Therefore it is hard to draw any conclusions about the effect how the PD induced aging affects the material because of the missing loss factor data. It is also difficult to draw any conclusions about $\Delta \varepsilon_{r,App}$ in this same interval just from looking at figures 4.19B and 4.20B. Although it is possible that the behavior of the loss factor is correct above 100
Hz and below 20 mHz. At low frequencies the same -1/2 slope trend of the loss factor is seen as has been seen in almost all other tested materials as well. Again (4.2) is plotted to more clearly show this relation as the purple lines in figures 4.19B and 4.20B.

### 4.4.1.2 Partial Discharge Pattern

![Figure 4.21: PRPD pattern of PE. (A) shows the PD pattern just after voltage application, (B) after 50 min and (C) is the last recorded PD pattern after 66 h and 55 min.](image)

Figure 4.21 shows the PD pattern of PE at some selected times. Figure 4.21A shows the PD pattern right after voltage application. The PD is spread over almost 90 degrees of the voltage phase with a larger concentration at around 45 degrees. The mean charge magnitude is a bit higher at the positive half-cycle of the voltage. Figure 4.21B shows the PD pattern after 50 min. It has a very different shape than at voltage application. Most PD are concentrated in a very narrow phase band and two PD clusters can be identified. The first cluster contains PD of rather small magnitude, the second cluster which is located at virtually the same phase angle but with larger PD magnitudes than the former. The PD pattern then stays like this for some time before it starts to decrease.
until the PD magnitude of the PD at the negative half-cycle of the voltage falls below the threshold level of the ICM and no more PD activity is detected. The last recorded PD pattern is shown in figure 4.21C which was recorded after 66 hours and 55 minutes of aging. It can be seen that there is no recorded PD activity during the negative half-cycle of the voltage. The recorded PD is around the same phase angles as in the other two subfigures, so the phase-angle of the PD activity does not seem to change particularly during aging, only the PD magnitudes.

4.4.2 Long Time Aging

For the long time aging two discs were cut from a 1.3 mm sheet of PE to form the top and bottom discs of the sample. The spacer disc was produced from a 0.25 mm thick sheet of PC. A 3.9-4 cm hole was cut in the spacer disc and it was then sandwiched between the two PE discs to form the sample. The complex permittivity was measured using IDAX300 before aging started and then once per day, except during the weekend. Before aging the PD inception voltage was determined using the circuit shown in figure 3.8. For this sample it was found to be approximately 11 kV peak. The sample was then placed in the circuit from figure 3.9 for aging. The voltage was set at 13 kV peak.

4.4.2.1 Dielectric Spectroscopy Measurements

During the DS measurements the IDAX300 internal 200 V source was used and the complex permittivity was measured between 10 mHz-10 kHz. The results are shown in figures 4.22 and 4.23.

The first thing that is noticed in figures 4.22 and 4.23 is the presence of some kind of measurement artifact of the same kind as was seen for PTFE in figures 4.17 and 4.18. This adds further to the suspicion that it is caused by the IDAX300 machine rather than being a property of the material. In figure 4.22 it can be seen that initially \( \varepsilon''_{r,App} \) decrease below the measured curve for the non-aged top disc. After 107 h an increase in the loss at low frequencies can be observed then at 129h and 30 min it has decreased again. Regarding the bottom disc in figure 4.23 it show the same behavior as the top disc at first, with a decrease of \( \varepsilon''_{r,App} \) at first and then after 107 h an increase is observed and a loss peak can be seen around 2 - 3 Hz. At 129 h and 30 min the loss factor has decreased again but it is still higher than the non-aged curve for the lowest frequencies. For high frequencies both the top and bottom disc show the same behavior, \( \varepsilon''_{r,App} \) remains virtually the same until 107 h where a slight increase is seen. At 129 h and 30 min it has decreased slightly again for the top disc while it remains more or less the same for the bottom disc. At low frequencies the loss factor is very
Figure 4.22: Permittivity of PE top disc at different times during long aging. (A) shows the complex permittivity. In (B) $\varepsilon_{\infty}$ has been subtracted to display the dynamic part $\Delta\varepsilon'$ of the real part of the complex permittivity and straight lines (purple) with -1/2 slope have been added.

close to a straight line with -1/2 slope in the logarithmic scale, this is feature appears when diffusion occurs in the material and is shown in figure 4.22B and 4.23B.
Figure 4.23: Permittivity of PE bottom disc at different times during long aging. (A) shows the complex permittivity. In (B) $\varepsilon_\infty$ has been subtracted to display the dynamic part $\Delta\varepsilon'_r$ of the real part of the complex permittivity and straight lines (purple) with -1/2 slope have been added.

4.5 PA6

4.5.1 Short Time Aging

Three discs were cut from a 1 mm thick sheet of PA6. In one of the discs an 8 mm hole was cut to form the cavity. DS measurements was performed before and after aging using IDA200 on the top and bottom discs separately. The sample was then aged for 72 hours and 5 minutes in the circuit shown in figure 3.8 at 10 kV peak. Every 5 min the PRPD pattern was recorded during 20 seconds.
4.5.1.1 Dielectric Spectroscopy Measurements

The DS measurements was performed using the internal 200 V peak source of the IDA200 between 10 mHz-1 kHz. The results of the DS measurements before and after aging for the top and bottom discs are shown in figures 4.24 and 4.25.

![Figure 4.24](image1.png)  ![Figure 4.25](image2.png)

**Figure 4.24:** Permittivity of PA6 top disc before and after aging. (A) shows the permittivity before and after aging. In (B) $\varepsilon_\infty$ has been subtracted to display the dynamic part $\Delta\varepsilon'_r$ of the real part of the complex permittivity and straight lines (purple) with -1/2 slope have been added.

**Figure 4.25:** Permittivity of PA6 bottom disc before and after aging. (A) shows the permittivity before and after aging. In (B) $\varepsilon_\infty$ has been subtracted to display the dynamic part $\Delta\varepsilon'_r$ of the real part of the complex permittivity and straight lines (purple) with -1/2 slope have been added.

It can be seen in figures 4.24 and 4.25 that $\varepsilon''_{r,App}$ is lower for the aged sample during the whole measured frequency range and the aged sample appear to be separated by a constant value from the non-aged sample. At low frequencies the loss factor increases and seem to tend toward a -1/2 slope of the curve which can be seen in figures 4.24B and...
4.25B where (4.2) is plotted to show this relation which is an indication of a diffusion process in the material. If the loss factor stays like this or if it is just the beginning of an up-slope that will become steeper at even lower frequencies which would be more consistent with a conduction mechanism or some kind of low frequency dispersion is unknown.

4.5.1.2 Partial Discharge Pattern

The PRPD pattern of the PA6 sample at different times during aging is shown in figure 4.26.

Figure 4.26: PRPD pattern of PA6, (A) shows the PD pattern just after voltage application, and (B) shows the last recorded PD pattern after 72 h and 5 min.

Figure 4.26A show the PD pattern just after voltage application. There is a large spread in phase of the PD, almost 90 degrees. The magnitude of the PD is quite low and evenly distributed, there are no sharp peaks in the pattern. As the aging continues the PD pattern decreases and eventually disappears altogether. Then it continues to be at the threshold of what is detected by the ICM, sometimes some PD activity is registered and sometimes not. Figure 4.26B show the last recorded PD pattern after 72 h and 5 min of aging. The decrease in PD activity could be an indication of that the surface conductivity inside the cavity and on the cavity walls are high enough so as to prevent the build up of a high enough field inside the cavity.

4.5.2 Long Time Aging

A top and a bottom disc was cut from a 1 mm thick sheet of PA6 and a spacer disc was cut from a 0.25 mm sheet of PC. A 3.9-4 cm hole was cut in the spacer disc for the
Chapter 4. Measurement Results

The cavity. The DS measurements was performed before and the once per day, except during the weekend, on the top and bottom discs using IDAX300. Before aging the sample was put in the circuit shown in figure 3.8 and the PD inception voltage was determined. It was found to be approximately 5 kV peak for this sample. The sample was then put in the circuit shown in figure 3.9 for aging.

4.5.2.1 Dielectric Spectroscopy Measurements

The results of the DS measurements are shown in figures 4.27 and 4.28. The measurements was done at 200 V peak using the internal source of the IDAX300 in the frequency range 10 mHz-10 kHz.

There is a large difference in the results between the top and bottom disc. If figures 4.27A and 4.28A are compared the loss factor behaves very differently at low frequencies. Both discs exhibit a very flat loss factor down to about 10 Hz, where the bottom disc start to show a different behavior after 86 h and 15 min. At low frequencies $\varepsilon''_{r,\text{App}}$ increase compared to the non-aged measurement. At 108h and 5 min a decrease of $\varepsilon''_{r,\text{App}}$ was observed to a level below the non-aged measurement. The bottom disc on the other hand show an increased loss for all measurements compared to the non-aged sample. After 86 h and 15 min a sharp increase in the loss factor can be seen at low frequencies and after 108 h and 5 min $\varepsilon''_{r,\text{App}}$ increase even more. The same variations in $\varepsilon''_{r,\text{App}}$ at high frequencies is not observed. For the top disc the loss factor remains more or less the same until 108 h and 5 min where there is a decrease of the loss factor. For the bottom disc, on the other hand, there is a larger increase in $\varepsilon''_{r,\text{App}}$. At low frequencies there is an increase in the loss factor which corresponds quite well to a -1/2, which is shown in figures 4.27B and 4.28B where (4.2) has been added as the two purple lines to show this behavior.

4.6 Impact of PD Aging on the Complex Permittivity

The tested materials behave differently and it is not possible to find any common trends that is seen in all materials and aging times. The trend that is repeated the most is the -1/2 slope of $\varepsilon''_{r,\text{App}}$ for low frequencies. Some materials does not seem to be affected very much by the PD aging, like the long time aging of PVC, see figures 4.5 and 4.6. There does not appear to be any major penetration of charges into the material which would affect the DS measurements. Then there is PC, see figures 4.12 and 4.13, where the DS measurements show very similar values of $\varepsilon''_{r,\text{App}}$ for low frequencies and increasing values at high frequencies compared to the non-aged sample, then when the short time aged
Figure 4.27: Permittivity of PA6 top disc at different times during long aging. (A) shows the complex permittivity. In (B) $\varepsilon_\infty$ has been subtracted to display the dynamic part $\Delta\varepsilon'_r$ of the real part of the complex permittivity and straight lines (purple) with -1/2 slope have been added.

In PA6, see figures 4.27 and 4.28, the opposite behavior is seen in $\varepsilon''_{r,\text{App}}$. At high frequencies the loss factor displays very similar values, with a small decrease compared to the non-aged sample after 108 h, and at low frequencies the dispersion of the loss factor increase.

An initial hypothesis was that the dielectric behavior of the materials could to some extent be related to their placement in the Triboelectric series. This was the reason why the sample was remeasured it was found that the change in the loss factor had disappeared, see figure 4.10, which indicate that the change was caused by space charges in the bulk.
Figure 4.28: Permittivity of PA6 bottom disc at different times during long aging. (A) shows the complex permittivity. In (B) $\varepsilon_\infty$ has been subtracted to display the dynamic part $\Delta \varepsilon'_r$ of the real part of the complex permittivity and straight lines (purple) with -1/2 slope have been added.

PA6 and PTFE were chosen, since they are located at either end of the Triboelectric series. No clear evidence was found in the results to support this hypothesis. PA6 has a larger dispersion of $\varepsilon''_{r,App}$ at lower frequencies than PTFE. Whether this is due to their placement in the Triboelectric series or some other reason cannot be concluded without further testing and comparison of the materials.

All measurements was done without any extra weights on the top electrode to get rid of any unwanted air-gaps between the electrodes and the test sample. It is therefore possible that some of the measurements has been affected by this, which can be seen in
Figure 4.7, where the long time aged PVC sample was remeasured and the sharp minimum disappeared. Then there is PE and PTFE which display similar behaviors with $\varepsilon''_{r, App}$ remaining more or less the same during aging up until approximately 100 h for both materials, see figures 4.17, 4.18, 4.22 and 4.23. Then for the last measurement the loss factor has decreased again for the materials. Regarding the incomplete measurements in the short time aging of PTFE and PE it is difficult to draw any conclusions about the dielectric behavior and in future work the measurements would have to be remade to try and get a measurement of the whole loss factor.

4.7 Diffusion

In most of the materials tested in this thesis $\varepsilon''_{r, App}$ was found to have a -1/2 slope or very close to it at low frequencies, the exception being the short time aging of PC shown in figures 4.8B and 4.9B which was found to have a -1 slope instead. According to (4.2) $\Delta \varepsilon'_{r, App}$ and $\varepsilon''_{r, App}$ should be equal. Therefore, it is not perfectly obvious if a diffusion mechanism is active in the materials. Although, in some measurements, see figures 4.3A, 4.3B, 4.24B and 4.25B, $\Delta \varepsilon'_{r, App}$ approaches $\varepsilon'_{r, App}$ for low frequencies and it is possible that they will become equal for frequencies below the measured frequency range. The low frequency limit of 10 mHz was set in order to save time in the DS measurement because for low frequencies the measurement times becomes very long. In future studies to perform DS measurements for much lower frequencies than was done in this thesis, then it would be possible to see if the increase in $\varepsilon''_{r, App}$ and $\Delta \varepsilon'_{r, App}$ is the beginning of a loss peak or if it is in fact diffusion. The -1/2 behavior is seen in the non-aged samples too, this is an indication that even if there is a diffusion mechanism in the material it was also active in the non-aged samples, and therefore not something caused by the PD aging. The condition for diffusion to occur is that the density of a charge carrier is higher at someplace in the sample. The charges then diffuse in the material from places of higher concentration to places of lower concentration until equilibrium is reached. The presence of a diffusion behavior in the DS measurements therefore indicate that there is a larger concentration of charge somewhere in the sample.

4.8 Space Charges

A results of the discharges in the cavity space charges are deposited on the cavity surfaces in shallow traps while others diffuse into the material where they recombine or are trapped in traps embedded deeper in the material. The mean trapping time of the space charges in the material can be very long [27]. If there is space charges trapped in
deep traps it will affect the DS measurements because they have not had time to escape the traps and recombine or be diffused away. If the aged sample is then left to rest so the trapped charges are able to recombine or diffuse away the result will be different if another DS measurement is done. This can be seen in figures 4.10A and 4.10B which show the short time aged PC sample which was left to rest for 35 days before another DS measurement was performed. The measurement showed the loss factor had returned to virtually the same state from before it was aged. This suggests that the change that was seen in figures 4.8 and 4.9 was because of space charges trapped in the bulk of the material and no permanent damage of the material has occurred. In [28] the authors study space-charge characteristics of LDPE during AC aging, and they found in one of their measurements that charges are trapped in deep traps in the bulk of the material. They draw the conclusion from their study that their measurements strongly suggest that AC aging has changed the trapping characteristics of the material. Therefore, one possible explanation to the change in dielectric response of the tested materials is that the trap density and trap depths has been changed in favor of deeper traps where the charges require longer time to escape. The change in the dielectric response in, at least, the short time aged PC could then be accredited to space charges in deep traps in the bulk.

4.9 Significance of Unwanted Air-gaps in Dielectric Spectroscopy Measurements

When performing the DS measurements it is very important that the electrodes of the test cell is intimately pressed against the test sample so that no unwanted air-gap is introduced, because it will affect the results different results can be obtained for the same sample if it is removed from the test cell and then put back again and a new measurement is performed, it all depends on how large air-gap is introduced. The reason for the unwanted air-gaps is that the top electrode of the test cells shown in figures 3.1A and 3.1B is to light. Another reason is the material of the sample to be tested. Some of the tested materials were quite stiff and hard to bend and they were not perfectly flat, so the top electrode and the spring in the measurement electrode were not able to press away all air between the sample and electrodes. This can clearly be seen in figure 4.7 where the strange minimum in $\varepsilon''_{r,App}$ has disappeared when the sample was measured again but this time with extra weights on the top electrode. It can also be seen that there is also a small difference at low frequencies but at high frequencies the loss factor is more or less unaffected.
Chapter 5

Conclusions

5.1 Conclusions

The aim of this thesis was to investigate how the dielectric properties of different polymers change when subjected to prolonged PD activity in enclosed cavities. And possibly provide a suggestion on what the mechanisms behind the change is. In order to achieve the thesis aim samples of PVC, PC, PTFE, PE and PA6 was aged for different times and the dielectric response was measured using dielectric spectroscopy analyzers. From the obtained results the following can be concluded

- All tested materials are to some extent affected by the PD activity and changes in $\varepsilon''_{r,App}$ can be observed, which supports the theory presented by the author in [3] of the creation of an aged zone around the cavity, although some materials, like PVC, is not very affected by the PD aging.

- No universal behavior can be seen that applies to all materials and measurements. The trend that is seen the most is a $-1/2$ slope at low frequencies, but this behavior is only seen in $\varepsilon''_{r,App}$ and not in $\Delta\varepsilon'_{r,App}$ which it should according to (4.2) for it to be a real diffusion mechanism. In some of the materials it is seen that $\Delta\varepsilon'_{r,App}$ approaches $\varepsilon''_{r,App}$ and it is possible that they will become equal for lower frequencies than the investigated frequency range.

- Some of the change in $\varepsilon''_{r,App}$ can be attributed to space charges trapped in the bulk, possibly due to a change in trapping characteristics of the material due to the aging. When the trapped charges are neutralized the complex permittivity will return to its original state, which was observed in figure 4.10.
• In order to get consistent results the measurement electrodes must be perfectly flat against the test sample so that no unwanted air-gaps is introduced, because it will affect the results and make it harder to interpret.

In conclusion it is likely that prolonged PD activity in enclosed cavities in polymer insulation affect the dielectric response of the polymer material, although the response varies depending on the material and the degree to which it is affected by the PD activity.

5.2 Suggestions For Future Work

One of the problems in the measurements in this thesis is likely due to the top electrode not being heavy enough and the spring in the measurement electrode not being strong enough to force the sample to lie flat against the electrode surfaces. This introduces unwanted air-gaps between the electrodes and sample which affect the result. Therefore, in future work it would be good to use heavier electrodes in order to get better measurements. Another problem is the relatively large thickness of the discs and the relatively small area of the measurement electrode, this yields a small capacitance and for some materials the capacitance was so low that the dielectric spectroscopy analyzers had problem measuring the dielectric loss. In the future it might therefore be beneficial to use larger measurement electrodes in order to obtain a larger capacitance. It was found that \( \varepsilon''_{\text{r,App}} \) in many of the measurements had a -1/2 slope at low frequencies but \( \Delta \varepsilon'_{\text{r,App}} \) does not equal \( \varepsilon''_{\text{r,App}} \) as it should according to (4.2). Although for some materials it was found that \( \Delta \varepsilon'_{\text{r,App}} \) approaches \( \varepsilon''_{\text{r,App}} \) for low frequencies, and in the future it would be useful to perform DS measurements down to very low frequencies to get a more complete picture of the dielectric response of the material.
Bibliography


