



CHAPTER IV

Effect of Electric Field Strength and Temperature on Electromechanical Properties of Ethylene Propylene Diene Elastomers (EPDM)

Patcharee Intanoo¹ and A. Sirivat^{1*}

¹The Petroleum and Petrochemical College, Chulalongkorn University,
Bangkok 10330, Thailand

4.1 Abstract

An elastomer that can response to external stimuli such as temperature and electric field are known as the dielectric elastomer consisting of either a polar molecule or an unsaturated structure on its side chain. In our work, ethylene propylene diene elastomers (EPDM) were prepared as thin films. The effects of electric field strength and temperature on electromechanical properties were investigated. The electrical conductivity, the dielectric constant, the storage and loss moduli (G' and G''), the storage modulus response ($\Delta G'_{1000\text{V/mm}}$), and the storage modulus sensitivity ($\Delta G'_{1000\text{V/mm}}/G'_0$) of the elastomers of different ENB contents and of different molecular weights were measured under applied electric field strength varying from 0 V/mm to 1000 V/mm and at temperatures between 300 -380 K. In the case of different molecular weights, the storage modulus response and sensitivity increase with increasing molecular weight and dielectric constant, consistent with existing theory. However, for the case of different ENB contents, the storage modulus response and sensitivity vary inversely with the dielectric constant.

Keywords: dielectric elastomer, dipole moment, polar molecule, unsaturated structure, ethylene propylene diene elastomers (EPDM), electrorheological properties.

4.2 Introduction

Dielectric materials are typical of poor electrical conductivity; they are widely used in capacitors [1], as electroactive polymers (EAP), and as dielectric elastomer actuators (DEAs). DEAs applications have been proposed as large displacement actuators for use in mechatronic devices. They are simple, potentially of low cost, and of lightweight [2]. Dielectric elastomers typically consist of polar molecules with random orientations when no electric field is applied. An applied electric field will interact with the polar molecules by orienting the dipole moments [3]. Due to the induced dipole moments, some dimension changes are expected in such way that EAPs transform electrical energy into mechanical responses. Examples of dielectric elastomers are ethylene propylene diene elastomer [4], silicone elastomer [5], and polyurethane [6].

The acrylic acid and silicone are common dielectric elastomer. These actuators have shown excellent actuation properties: large strains up to 380%; high energy densities up to 3.4 J/g; high efficiency; high responsive speed, and good reliability and durability [5].

Kyokane. *et al.*, [6] studied the electro-contraction effect of normal polyurethane elastomer (PUE) functionalized with the hydroxyl group (doped PUE); the displacements increased with increasing applied voltage. The bendings of the doped PUE films were larger than of the normal PUE films and thus allowing operations at lower voltage. The actuation mechanism is the deformation of the polymer networks as induced by the dipole orientation of electrically mobile elements in the elastomer [7].

Feher *et al.* [8] studied the bending of a TiO₂-loaded polydimethylsiloxane (PDMS) gel measured in a uniform and a non-uniform electric field. The gel cylinder between parallel copper electrodes gradually bent to the cathode. The bending behavior was found to be reversible, when positive and negative electrodes were alternated. As one of the electrodes was modified to create a non-uniform electric field, a metal ball replaced one of the electrodes, the bendings were the same. Li *et al.*, [9] studied the mechanical properties and electrical conductivities of TiN-Al₂O₃ nanocomposite. Experiment results showed that the resistivity decreased with

increasing amount of TiN and the bending strength also increased with increasing amount of TiN.

Yun *et al.*, [10] studied the performance of electroactive papers (EAPap) with different thicknesses: 20, 30, and 40 μm . EAPap actuators exhibit bending deformation in the presence of electric field depend in To prepare electroactive material, which have flexible property from doped-permethylnpolyazine / ethylene propylene diene elastomer (EPDM) that the ethylene propylene diene elastomer used as a matrix and doped-permethylnpolyazine used as a disperse phase. Bassil *et.al.*, [11] studied the bending mechanism of the gel actuators (PAAM) caused by the pH gradient which is induced by an electric field; the gel actuator showed a volume change upon a change in its environment condition. The thickness of the actuator increased, but it needed more electrical power to move. Watanabe *et.al.*, [12] studied the electromechanical responses of a pure polyurethane with compliant electrodes. The responses were due to the difference in charge densities between the anode and the cathode. Wissler *et.al.*, [13] reported the dielectric constants of different pre-stretched VHB 4910 membranes were measured, the values decreases with increasing pre-stretched ratio. Hiamtup *et.al.*, [14] studied the dielectrophoresis force of polymer blends of polyaniline and polydimethylsiloxane with fixed copper electrodes. The dielectrophoresis force increases with increasing electric field strength but decreases with increasing polyaniline concentration.

In our work, we investigate the electromechanical responses of two classes of EPDM elastomers: EPDM with different ENB contents (NORDEL IP 3670, NORDEL IP 4570, and NORDEL IP 5565) and EPDM with different molecular weight contents (NORDEL IP 4520, NORDEL IP 4640, and NORDEL IP 4570). We determine the storage modulus response ($\Delta G'_{1000\text{V}/\text{mm}}$), and the storage modulus sensitivity ($\Delta G'_{1000\text{V}/\text{mm}} / G'_0$) subject to various electric field strengths and temperatures.

4.3 Experimental

4.3.1 Materials

Ethylene propylene diene elastomers NORDEL IP 3670 (%ENB=1.8, and MW=210,000), NORDEL IP 4570 (%ENB=4.9, and MW=210,000), NORDEL IP 5565 (%ENB=7.5, and MW=210,000) for different ENB contents, and NORDEL IP 4570 (%ENB=4.9, and MW=210,000), NORDEL IP 4520 (%ENB=4.9, and MW=115,000), and NORDEL IP 4640 (%ENB=4.9, and MW=160,000) of different molecular weight contents were provided by Chemical Innovation Co., Ltd. The properties of elastomers are summarized in Table 4.1.

4.3.2 Preparation of Specimens

All elastomer specimens were fabricated through a solution casting. The NORDEL IP 3670, NORDEL IP 4570, NORDEL IP 5565, NORDEL IP 4520, and NORDEL IP 4640 were dissolved in hexane at 30 % vol/vol. The solutions were cast onto molds and the solvent was eliminated under a vacuum atmosphere at 300 K for 48 hours. Each sample was cut into a thin disc (diameter \approx 2.5 mm, and thickness \approx 1.0 mm).

4.3.3 Thermogravimetric Analysis (TGA)

The thermal stability of the physical and chemical crosslinking cellulose gels were investigated by the thermogravimetric analysis (TA, TGA Q50). The sample mass used was about 5–10 mg. The thermal scan was performed from 30°C to 800°C at a heating rate of 10°C/min in a nitrogen atmosphere. The nitrogen purging flow rate was 100 ml/min. The weight loss of the samples was measured as a function of temperature.

4.3.4 Characterization and Testing

The specific conductivity was measured by a resistivity test fixture (Keithley 8009 resistivity test fixture) connected with a voltage supplier (Keithley, model 6517A) whose constant voltage was varied and the resultant current was measured. The conductivity measurement was performed under atmospheric pressure, 40-60 %RH, at 25-27°C, in the linear Ohmic regime. The voltage and the current in the regime were converted to the electrical conductivity by following: $\sigma = 1/\rho = 1/(6.44 \times 10^{-5} \times R \times l)$

where σ is the specific electrical conductivity (S/cm), ρ is the specific electrical resistivity (Ω .cm), t is the thickness of a sample pellet (cm), V is the applied voltage (Voltage drop)(V), I is the measured current (A). All sample thicknesses were measured by using a thickness gauge.

The effects of electric field strength and temperature, between 300 and 380 K, on electromechanical properties were measured by a parallel plate fixture attached to a melt rheometer (Rheometric, ARES) with diameter of 25 mm and the thickness of the prepared elastomers of about 1 mm. A DC voltage was applied through a DC power supply (Instek, GFG 8216A). A digital multimeter (Tektronic, CDM 250) was used to monitor the voltage input. Dynamic strain sweep tests were first carried out to determine appropriate strain by measure G' and G'' in linear viscoelastic regime at 1.0 rad/s frequency. The appropriate strain was determined to be 0.2, 0.06, and 0.3% for NORDEL IP 3670, 4570, and 5565, respectively, and 0.06, 0.05, and 0.2% for NORDEL IP 4570, 4520, and 4640, respectively. For the temporal response testing, the dynamic moduli (G' and G'') were measured as functions of time and with electric field on and off. The temporal G' response of all elastomers was carried out at $T=300$ K. Frequency sweep tests were carried out to measure G' and G'' of each sample as functions of frequency and temperature. The deformation frequency was varied from 0.1 to 100 rad/s. In each measurement, each EPDM elastomer the electric field was applied for 50 min depend on type of EPDM elastomer to ensure the steady state condition before the G' and G'' measurements.

The dielectric constant values were measured by an LCR meter (HP, model 4284A) connected to the melt rheometer (Rheometric, ARES) with 25-mm parallel plate fixture with diameter of 25 mm and the thickness of the prepared elastomers of about 1 mm. The top and bottom sides of specimens were coated with a silver adhesive to improve electrical contact between specimens and the electrodes. The measurements were carried at temperature 300 K.

4.4 Result and discussion

4.4.1 The temporal responses of EPDM elastomer films

The temporal responses of all EPDM elastomer films were studied at $E = 1\text{ kV/mm}$, $\omega = 100\text{ rad/s}$, $T = 300\text{ K}$ and $\% \text{ strain} = 0.3$, and 0.06% and are shown in Fig. 4.1, and 4.2, respectively. All elastomer samples were presheared at a low frequency, and then the electric field was applied for 50 min to ensure the formation of equilibrium polarization before G' measurement. As can be seen, G' increases with electric field on and remains constant with electric field off. The increase in G' with electric field is due to the induced dipole moments generated on the unsaturated bond on the side chain. The electrostatic interaction between the dipole moments occur leading to an intermolecular interaction in elastomer molecules and the increase in G' . The Maxwell stress is not operative here since our electrodes were fixed [15]. As electric field is turned off, G' does not recover, suggesting that the EPDM material systems are irreversible, possibly due to residue dipole moments.

An induction time and a recovery time of NORDEL IP 5565 are 3735, and 0 sec, respectively in the steady state. NORDEL IP 4570 has an induction time and a recovery time of 1270, and 0 sec, respectively in the steady state. The induction times and the recovery times of both NORDEL IP 5565 and NORDEL IP 4570 are thus comparable. Wichiansee *et al.*, [16] reported the temporal response of crosslinked PDMS at the electric field strength of 1 kV/mm , the effect of electric field strength on G' ; it was irreversible system due to some residue dipole moments between PDMS molecules.

4.4.2 The effect of electric field strength on physical properties

The specific electrical conductivity values of NORDEL IP 3670, 4570, 5565, 4520, and 4640 at temperature of 300 K are 1.12×10^{-7} , 4.31×10^{-7} , 4.94×10^{-7} , 1.20×10^{-7} , and $2.53 \times 10^{-7}\text{ S/cm}$, respectively .

The effect of electric field on electromechanical properties of EPDM elastomer films was studied at $T = 300\text{ K}$ with the electric field strength varied between $2\text{-}1000\text{ V/mm}$. The storage modulus (G') vs. frequency of the EPDM

elastomer film (NORDEL IP 5565) at $\omega = 100$ rad/s is shown in Fig. 4.3. It can be seen that the storage modulus increases with increasing electric field strength at all frequencies examined. Polarization is generated on the unsaturated bonds on the side chain of the EPDM elastomer inducing dipole moments. This leads to an increase in the storage modulus. Figures 4.4, and 4.5 compare the storage modulus response, $\Delta G'$, and the sensitivity, $\Delta G'/G'_0$, of EDPM of various ENB contents and molecular weights, respectively. Both $\Delta G'$ and $\Delta G'/G'_0$ generally increase with increasing electric field strength for both sets of data. Furthermore, $\Delta G'$ and $\Delta G'/G'_0$, as shown in Figure 2b, decrease with ENB content and attain the maximum values of 4.34×10^{-5} Pa, and 0.45, respectively for NORDEL IP 3670 at $E = 1$ kV/mm. This finding can be referred to the fact that more ENB side chains available creates a larger free volume and hence reduces the dipole moment interaction at any given electric field. On the other hand, $\Delta G'$ and $\Delta G'/G'_0$, as shown in Figure 4.6, increase with increasing molecular weight and attain the maximum value of 3.99×10^{-5} Pa, and 0.18, respectively for NORDEL IP 4570 at $E = 1$ kV/mm. A higher molecular weight EDPM has a smaller number of free ends and hence a smaller free volume and hence a more effective dipole moment interaction. Table 4.2 summarizes the data and the findings with respect to the effects of ENB content and molecular weight.

4.4.3 The effect of temperature on electromechanical properties

The effect of temperature on the electromechanical properties of EPDM films was studied at electric field strengths of 0 and 1 kV/mm within the temperature range between 300 and 380 K. The storage modulus (G') of EDPM elastomers of different ENB contents and molecular weights versus frequency at electric field 0 and 1 kV/mm, and $\omega = 100$ rad/s, within temperature range 300-380 K, are shown in Figures 4.7. It can be seen that G' increases with increasing temperature only at large frequencies in conformation with the classical network theory [17];

$$G' = \nu k_B T \quad (1)$$

where k_B is Boltzmann's constant, T is the absolute temperature (K), and ν is the crosslink density ($1/\text{cm}^3$). As temperature increases, the entropy of the elastomer [17] also increases; the molecules vibrate more vigorously and hence generate a greater retractive force on the network. The increase in G' at large frequencies can thus be traced back to the elastomeric backbone of EDPM. On the other hand, G' decreases with increasing temperature at low frequencies. This behavior reflects the fact that the ENB side chain is of thermoplastic in origin.

Figures 4.8 and 4.9 show the storage modulus response, $\Delta G'$, and sensitivity, $\Delta G'/G'_0$, (100 rad/s, 1kV/mm) vs. temperature of EDPM of different ENB content and molecular weight, respectively. It can be seen that generally $\Delta G'$ (100 rad/s, 1kV/mm) increases linearly with temperature, consistent with Eq. (1) and attains the maximum values of 1.43, 1.41, 1.23, 1.17, and 1.03 for NORDEL IP 3670, 4570, and 5565, 4640, and 4520, respectively, at temperature of 380 K. Kumanuruksapong *et.al.*, [18] reported that the storage modulus response ($\Delta G'$) and sensitivity ($\Delta G'/G'_0$) of acrylic elastomers increase with increasing temperature at frequency 1 rad/s. The storage modulus response ($\Delta G'$) and sensitivity ($\Delta G'/G'_0$) of styrene copolymer increased initially and then decreased at the temperatures above T_g [18], due to the temperatures above T_g , the materials change their behavior from rubbery like to plastic like [18]. Sato *et.al.*, [19] studied rheological properties of styrene copolymer (SIS triblock copolymer) in an n-tetradecane as function of frequency and temperature without an electric field. At low temperature ($T < T_g$ of styrene segment), the styrene behavior is still rubbery like. But, with increasing temperature ($T > T_g$ of styrene segment), the system behavior become plastic and viscous like. Puvanattvattana *et.al.*, [20] reported the storage modulus response ($\Delta G'$) of various crosslinked polyisoprene systems as functions of electric field strength at frequency of 1 rad/s, the $\Delta G'$ increase with increasing electric field strength. Fox *et.al.*, [21] reported that the displacement of dielectric elastomer membrane is highest when large inflated state and low frequencies were used, that it drops off rapidly as frequency increases.

4.4.4 The dielectric constants of EPDM elastomers

The dielectric constant of NORDEL IP 3670, 4570, 5565, 4520, and 4640 at $T = 300$ K and at the frequency of 100 Hz, are 1.27, 1.88, 1.92, 1.53, and 1.53, respectively. The dielectric constants vs. frequency of all EPDM elastomers are shown in Figures 4.10, and 4.11. The dielectric constants are independent of frequency. From the data tabulated in Table 4.2, it can be seen that the dielectric constant increases with ENB content for the first data set; as more side chains are available to create greater dipole moments or to store more charges. In addition, the dielectric constant increases with increasing molecular weight or with a smaller free volume for EDPM of different molecular weights.

The increases in the storage modulus response and sensitivity, for EDPM of different molecular weights, can be correlated with the increase in the dielectric constant, consistent with the existing theory [22]. On the other hand, the storage modulus and sensitivity, for EDPM of different ENB content, monotonically decrease with the dielectric constant as shown in Table 4.2. This reflects the fact that as more side chains are available, even though creating a greater capability to store charges, the effect is outweighed by the resultant larger free volume or a greater distance for the dipole moments to interact.

Hao *et.al.* [23] studied the mechanical properties of starch/silicone oil/silicone rubber, they found that the storage modulus sensitivity ($\Delta G'/G'_0$) first increased with silicone oil concentration and then decreased. The latter is related to the fact that the silicone oil. As silicone oil concentration increased, the dipole-dipole distance increased and the electrostatic force decreased which weakens the effect of the electric field on the storage modulus.

4.5 Conclusion

In our work, electromechanical properties of EPDM elastomers; NORDEL IP 3670, 4570, 5565, 4520, and 4640 were investigated through examining the effect of electric field strength and temperature on the dynamic storage modulus (G') under

oscillatory shear mode. The experiment were carried out at electric field strength varying from 0 to 1 kV/mm in frequency and temperature sweep test modes. In our EPDM elastomers, there are unsaturated structures on the side chain which can induce electrical dipole moments, the storage modulus response ($\Delta G'$) and sensitivity ($\Delta G'/G'_0$) increase with increasing both electric field and temperature between 300 K and 380 K at high frequencies; the latter effect is due to entropic contribution of elastomeric matrices. The dielectric constant of NORDEL IP 5565 is the highest because of more unsaturated structures on the side chains which can generate more dipole moments.

4.6 Acknowledgements

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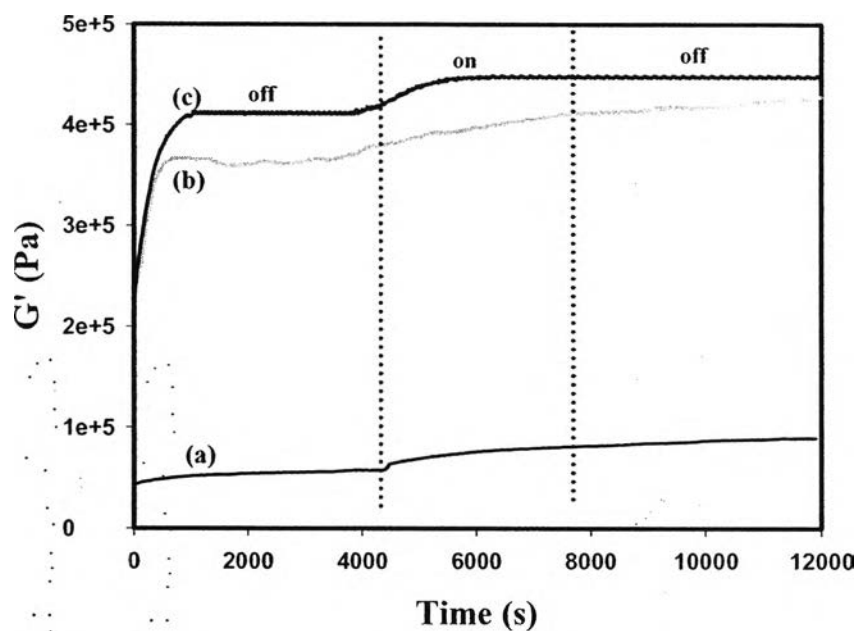


Figure 4.1 Storage modulus, G' , at 100 rad/s, 1kV/mm: vs. time of EPDM films of different ENB contents: (a) NORDEL IP 5565; (b) NORDEL IP 4570; and (c) NORDEL IP 3670.

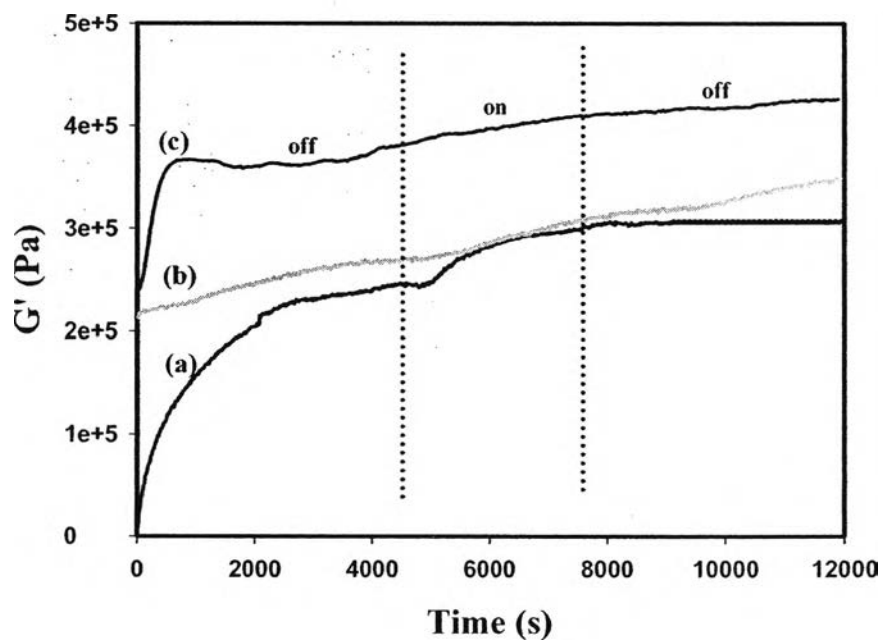


Figure 4.2 Storage modulus, G' , at 100 rad/s, 1kV/mm: vs. time of EPDM films of different molecular weights: (a) NORDEL IP 4520; (b) NORDEL IP 4640; and (c) NORDEL IP 4570.

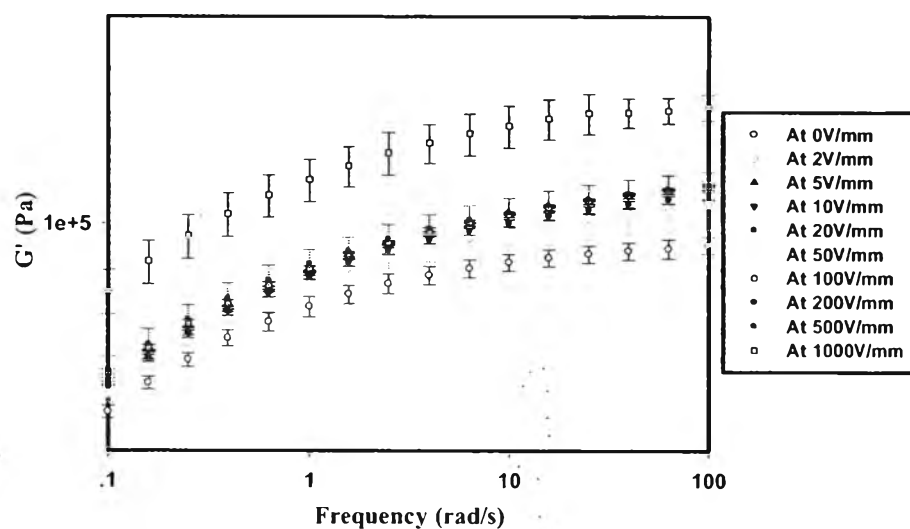


Figure 4.3 Storage modulus, G' , at $T = 300$ K, and % strain = 0.3%, vs. frequency of NORDEL IP 5565 film at various electric field strengths.

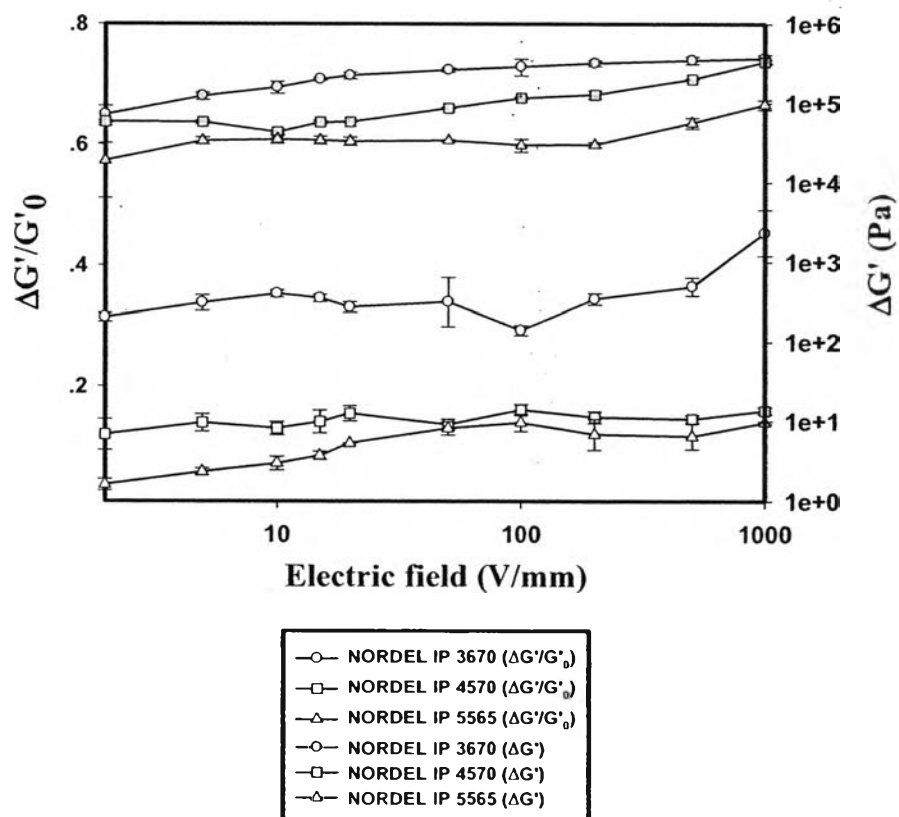


Figure 4.4 Storage modulus response, $\Delta G'$, and sensitivity, $\Delta G'/G'_0$, at $T = 300$ K, and % strain = 0.3%, of EPDM films of different ENB contents vs. electric field strength.

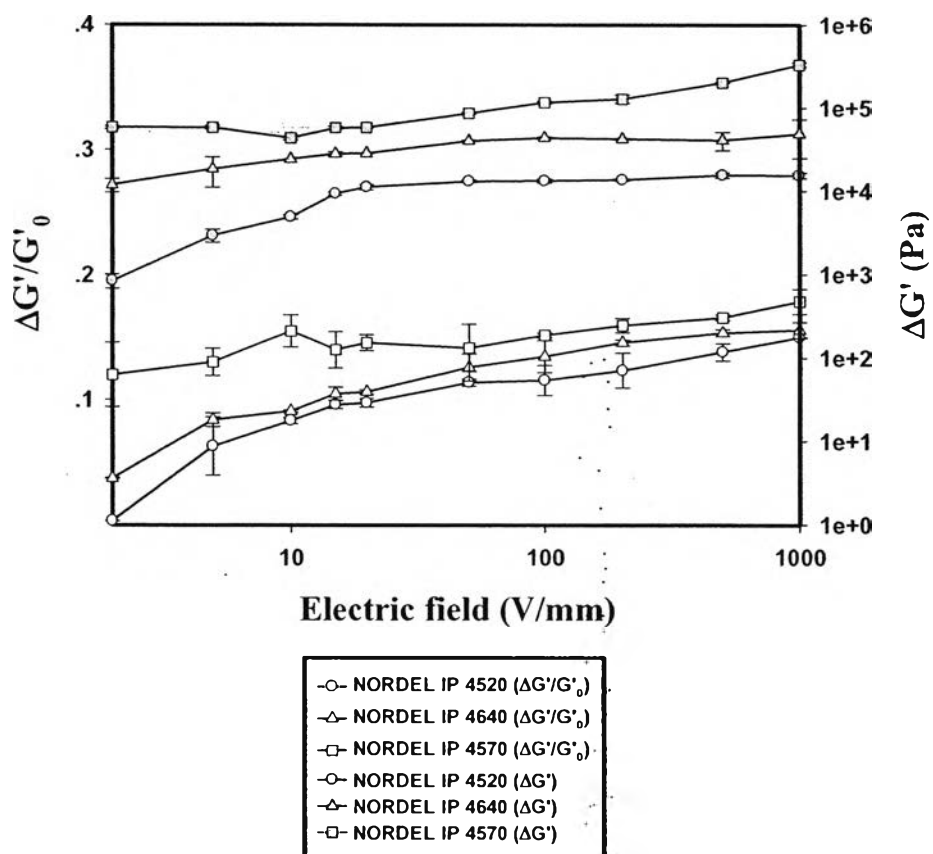


Figure 4.5 Storage modulus response, $\Delta G'$, and sensitivity, $\Delta G'/G'_0$, at $T = 300$ K, and % strain = 0.3%, of EPDM films of different molecular weights vs. electric field strength.

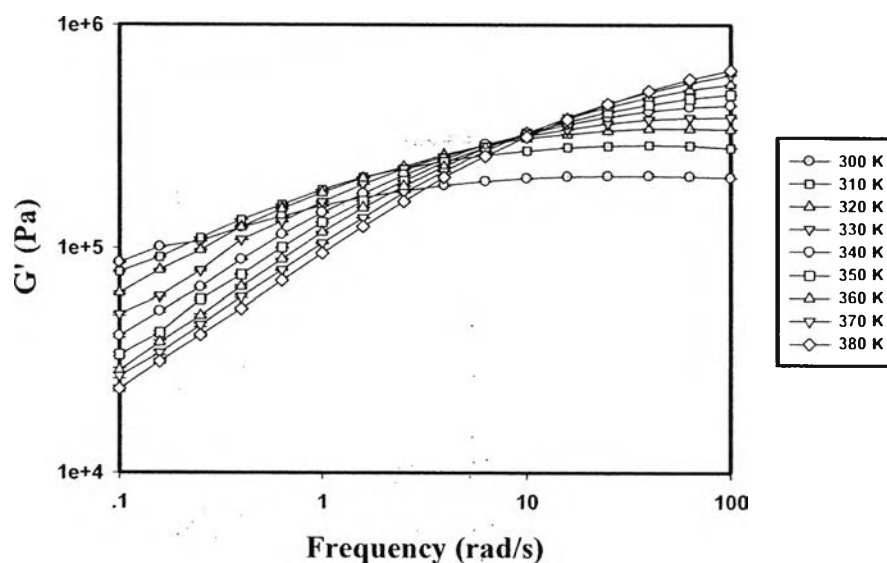


Figure 4.6 Storage modulus, G' , at 100 rad/s, 0kV/mm, and % strain = 0.5%, vs. frequency of NORDEL IP 5565 films at various temperatures (100 rad/s, 0kV/mm, and % strain = 0.5%).

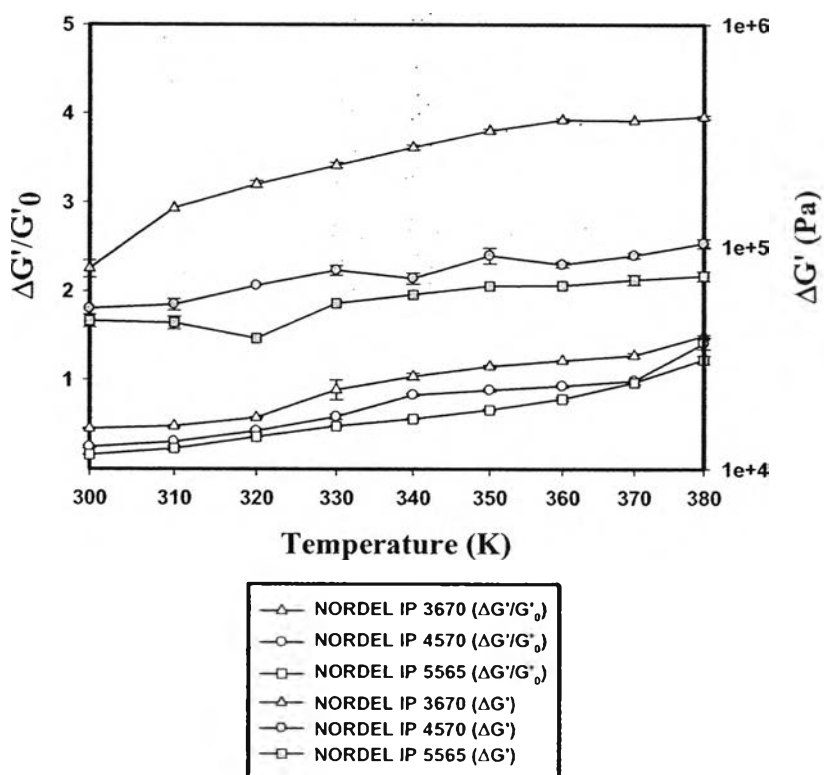


Figure 4.7 Storage modulus response, $\Delta G'$, and sensitivity, $\Delta G'/G'_0$, at 100 rad/s, and % strain = 0.5%, of EPDM films of different ENB contents vs. temperature.

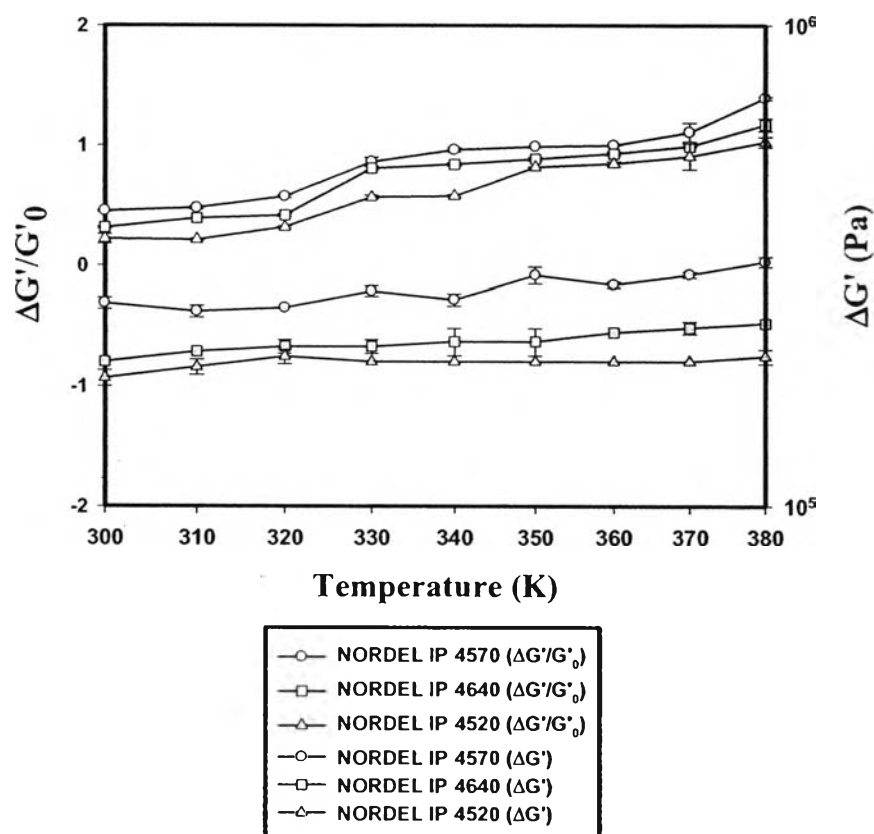


Figure 4.8 Storage modulus response, $\Delta G'$, and sensitivity, $\Delta G'/G'_0$, at 100 rad/s, and % strain = 0.03%, of EPDM films of different molecular weights vs. temperature.

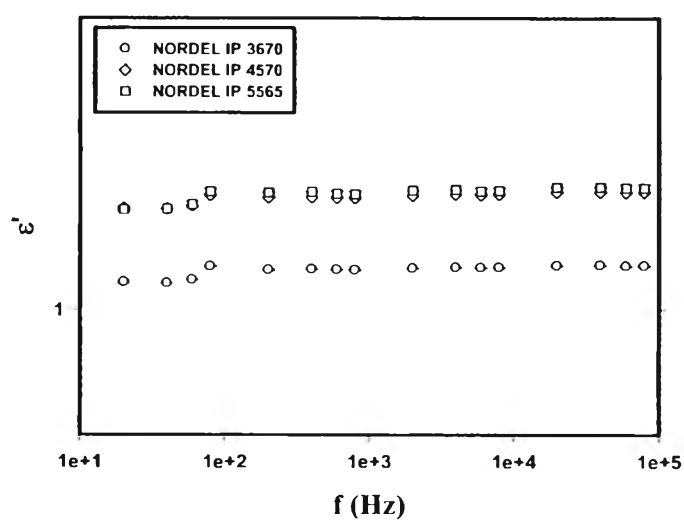


Figure 4.9 Relative dielectric constant (ϵ') vs. frequency of EPDM films of different ENB contents at temperature of 300 K.

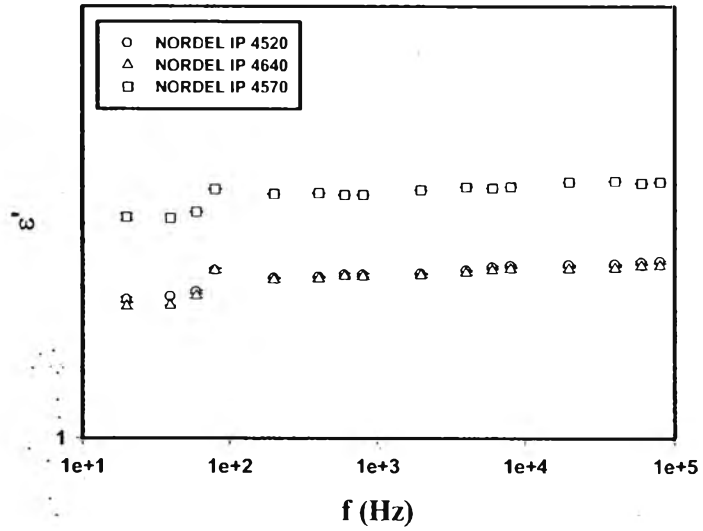


Figure 4.10 Relative dielectric constant (ϵ') vs. frequency of EPDM films of different molecular weights at temperature of 300 K.

Table 4.1 Properties of ethylene-propylene diene (EPDM) films.

Material	wt%ENB	M_w (g/mol)	Density (g/cm³)	T_d (°C)
NORDEL IP 3670	1.8	210,000	0.86	457
NORDEL IP 4570	4.9	210,000	0.86	450
NORDEL IP 5565	7.5	210,000	0.86	442
NORDEL IP 4520	4.9	115,000	0.86	440
NORDEL IP 4640	4.9	160,000	0.86	450
NORDEL IP 4570	4.9	210,000	0.86	450

Table 4.2 Comparison of storage modulus response ($\Delta G'$) and sensitivity ($\Delta G'/G'_0$) of EPDM films.

Materials	%ENB	M _w (g/mol)	Electric field (V/mm)	Frequency (rad/s)	Temperature (°C)	Storage modulus response ($\Delta G'$). Pa	Storage modulus sensitivity ($\Delta G'/G'_0$)	Relative dielectric constant (ϵ') at low frequency	Induction time (τ_{ind}). s	Recovery time (τ_{rec}). s
NORDEL IP 3670	1.8	210,000	1000	100	27	4.34×10^5	0.453	1.24	1331	0
NORDEL IP 4570	4.9	210,000	1000	100	27	3.99×10^5	0.178	1.86	1270	0
NORDEL IP 5565	7.5	210,000	1000	100	27	9.65×10^4	0.140	1.91	3735	0
NORDEL IP 4520	4.9	115,000	1000	100	27	1.24×10^4	0.149	1.50	4462	0
NORDEL IP 4640	4.9	160,000	1000	100	27	4.86×10^4	0.155	1.49	4038	0
NORDEL IP 4570	4.9	210,000	1000	100	27	3.99×10^5	0.178	1.86	1270	0