

High actuation performance offered by simple diene rubbers

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ABSTRACT

Dielectric elastomers are materials well-known for their superior actuation behavior under applied electric field. The simplicity of material fabrication and clear working principle of dielectric elastomer actuators (DEAs) can offer various applications of dielectric elastomers. In this work, we have compared a number of different types of commercially available elastomers in terms of actuation performance. It was found that well-known commercial rubbers like acrylonitrile-butadiene rubbers (NBR) can offer higher actuation performance in DEAs than the frequently used dielectric elastomers, such as acrylic rubber and silicone. The acrylonitrile (ACN) content of the NBR was found to play an important role in the dielectric and consequently actuation properties. More interestingly, we observed that addition of organic oil, such as dioctyl adipate (DOA), can greatly enhance the actuation performance.

INTRODUCTION

Development of robotic system being able to work as human muscle has been ongoing for decades. Among the ‘smart’ materials used to obtain muscle-like action, dielectric elastomers (DEs) most resemble the natural muscle in strain, actuation pressure, density, efficiency and response speed. DEs are insulators that become polarized in applied electric field, thus being subjected to an active electrostatic pressure ^[1] and being able to change its shape enabling electrical actuation. Compared to other electrically active polymers (EAPs), dielectric elastomers offer good overall performance, high strains and decent cost. ^[2] However, same as for other EAPs, the main drawback of the contemporary dielectric elastomer actuators (DEAs) is very high operating voltage that can reach 10 kV for 100 μm thick films ^[3,4], thus limiting their applications ^[5,6]. Despite of the decades of intensive research in the field of DEAs, there is still no efficient material-based solution how to reduce working voltage of such devices.

Basic operation principle of DEA, also known as deformable capacitors ^[2], is shown in Fig. 1a. The actuator consists of thin DE sheet with compliant electrodes on its both sides. When the voltage is applied, charge is built up on the electrodes until the phase difference is compensated. The attraction of opposite charges presses the elastomeric film in thickness direction. Due to incompressible nature of rubbers, the film area covered by the electrodes enlarges simultaneously. When the film thickness decreases, electrical energy is converted into mechanical energy in the form of electrostatic pressure across the electrodes given by the Maxwell pressure (σ) equation ^[7]:

$$\sigma = \varepsilon' \cdot \varepsilon_0 \cdot E^2 \quad (1)$$

where ε' describes relative permittivity and ε_0 describes permittivity of empty space. Electric field E can be expressed by applied voltage V and distance d between electrodes $E=V/d$. Therefore, from equation (1) we get final expression for the Maxwell pressure:

$$\sigma = \varepsilon' \cdot \varepsilon_0 \cdot \left(\frac{V}{d}\right)^2 \quad (2)$$

Maxwell pressure can be used to calculate compressional strain in thickness direction (s_z) that also depends on the elastic modulus of the material (Y):

$$s_z = -\frac{\sigma}{Y} \quad (3)$$

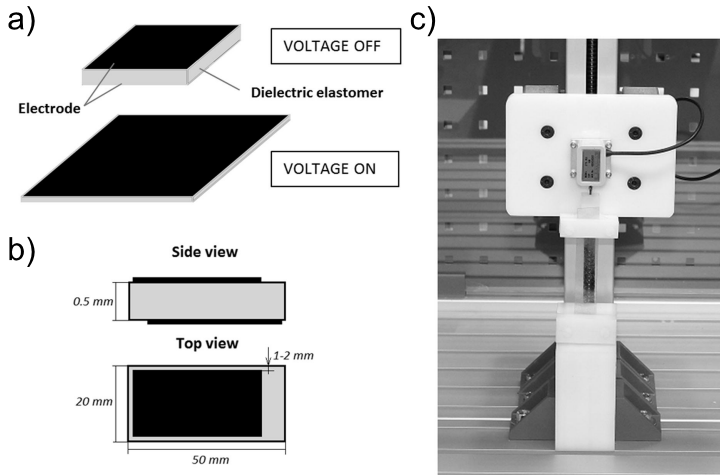


Figure1. a) Principle of DEAs b) Electrode fabrication with the rubber test samples c) Sample holder of the test equipment.

In literature, Y is often referred as Young's modulus. However, due to non-linear deformation of rubbers Young's modulus of rubber is difficult to determine and it does not give realistic value if pre-strain is used. Therefore, in current research, the elastic modulus at certain pre-strain X (Y_X) is used instead of Y for the pre-strained samples. According to abovementioned equations, high relative dielectric permittivity and low elastic modulus are required for good actuation performance. However, too low modulus may cause electro-mechanical instabilities [8]. Generally, DEs should possess the following properties in order to perform well as DEA: large actuation strain, low applied voltage, high energy density, good efficiency, and high response speed [9,10]. Moreover, light weight and low cost is preferable [11].

Rubbers are flexible polymers that are capable of significant and reversible deformation under applied mechanical stress [12]. They are generally insulators with dielectric constant ranging from

about 2 to 25^[8,9]. Elastomers more frequently studied for DEA applications include silicone, polyacrylic rubber (ACM) and polyurethane^[3]. Moreover, some research has been conducted on DEAs utilizing acrylonitrile-butadiene rubber (NBR)^[10], natural rubber (NR)^[13], chloroprene (CR)^[14], ethylene-propylene-diene (EPDM)^[15,16] and fluorinated^[8] rubbers. However, different testing approaches, equipment set-ups, sample dimensions and conditioning used in the abovementioned studies make it rather difficult to compare those versatile rubber materials. Therefore, a study involving a large selection of commercial elastomers for a DEA application may be in demand.

This study is aiming to compare different rubber materials in terms of actuation properties in order to show that the range of dielectric elastomer materials is not limited to the most widely used silicones and polyacrylates. The main goal of the current research is to utilize readily available, affordable and conventional materials in order to obtain material capable of significant deformations in response to an applied moderate electric field. The role of dielectric material is very important in providing proper insulation and low dielectric losses, as well as avoiding current leakage^[17]. In this work, first of all, different types of commercial rubber compounds are compared to find the most suitable material for dielectric actuators. Finally, one of the high performance rubbers i.e. NBR is selected and the effect of acrylonitrile (ACN) content of NBR as well as the effect of different plasticizers is studied.

EXPERIMENTAL

Materials

Several different polymers are used in the study. The studied materials include polymers discussed in earlier papers concerning DEA applications as well as new polymers which suitability for DEAs has not been studied earlier. In addition, the polymers with different polarities were chosen. The polymers are two different types of ACMs, NBRs with different ACN content, carboxylated NBR

(XNBR), epichlorohydrin homopolymer (CO) and terpolymer (GECO) as well as fluorosilicone (FMQ) and NR. Details of the materials and their curing packages are presented in Table 1.

Three different plasticizers are studied at the concentration of 5 phr: Fomblin Y (F), Liquid-NBR (LNBR), and Dioctyl adipate (DOA). F is dielectric perfluoropolyether oil provided by Sigma-Aldrich. LNBR (Nipol 1312) with ACN content of 28% was provided by Zeon Chemicals L.P. and DOA by BASF.

Sample preparation

The polymers and their ingredients were compounded in a laboratory scale mixer (Brabender W 50 driven by Brabender Plasti-Corder) with tangential rotors. The rotor speed of the mixer was 60 rpm and the starting temperature was 40°C. The details concerning the mixing of the different rubber compounds are presented in Table 2. If the rubber compound contained plasticizer, it was added after 2 min mixing. Mixed compounds were formed to 0.5 and 1 mm sheets and cured. The curing times and temperatures are presented in Table 1.

Characterizations

Tensile tests of the samples were carried out with a Messphysik Midi 10-20 universal tester according to ISO 37 using dumb-bell test specimen type 3.

Dielectric permittivity, dielectric loss and electrical conductivity were measured with a Novocontrol Alpha-A. The frequency sweep was done from 1 MHz to 1 Hz. For measuring dielectric properties of plasticizers, 100µm silica spacers were used to set the distance between electrodes.

Actuation measurements were conducted with two different set-ups. Actuation measurements of different elastomers were performed at VTT (Technical Research Centre of Finland) with the 20x50x1 mm samples. Carbon black (type N-234, Evonik) was used as an electrode. The samples were pre-stretched until 0.1 N force was reached. The force was measured with the 0.3 N load cell (SS2, Sherborne Sensors). The voltage from 0 to 8 kV was applied via conducting copper plates

from high voltage amplifier (TREK model 20/20C). The frequency used was 0.5 Hz. The change in force was measured when the voltage was applied to the samples. The used equipment had very limited capability to measure force and allowed only a few percent pre-strain. Therefore, another type of equipment was build in-house. The actuator (20x50 mm) was prepared by using 0.5 mm thick elastomer film. Electrodes were made by carbon black-toluene mixture spread on both sides of elastomer and then toluene was fully evaporated (Fig. 1b). The DC voltage from 0 to 5 kV was applied to the electrode in 0.5 kV steps via conducting copper plates from high voltage amplifier (TREK model 10/10B-HS). The samples were pre-stretched to 100%. The sample holder is presented in Fig. 1c. During the measurements, changes in force when voltage was applied were measured with a 10 N load cell (LTS-1KA, Kyowa). The measurement was controlled via LabVIEW 2012 software.

RESULTS AND DISCUSSION

As the dielectric permittivity (ϵ') describes material's ability to polarize in the presence of electric field ^[18] and the actuation stress is directly proportional to this value, it is interesting to compare the dielectric properties of different types of rubbers with different chemical structures. Fig. 2a shows the relative permittivity values as a function of frequency. All polymers except GECO show plateau-like behavior at the intermediate frequency ($10 - 10^5$ Hz) and the relative permittivity values in this region can be expressed in the following order NR < ACM dual < ACM chlorine < FMQ < XNBR < CO < NBR3330 < NBR3945 < GECO. At lower frequencies (<10 Hz) some effect of interfacial polarization, occurring at the interfaces of microscopic boundaries where the charges built up, on the relative permittivity can be observed, especially for NBRs and epichlorohydrin rubbers. For polyepichlorohydrin terpolymer ^[19], this effect is explained by the charge build-up at the boundaries of structured blocks in copolymer - poly(epichlorohydrin-co-ethylene oxide-co-allyl

glycidyl ether), as well as to the presence of curatives and some impurities in rubber. The similar phenomena can be attributed to nitrile rubbers [20].

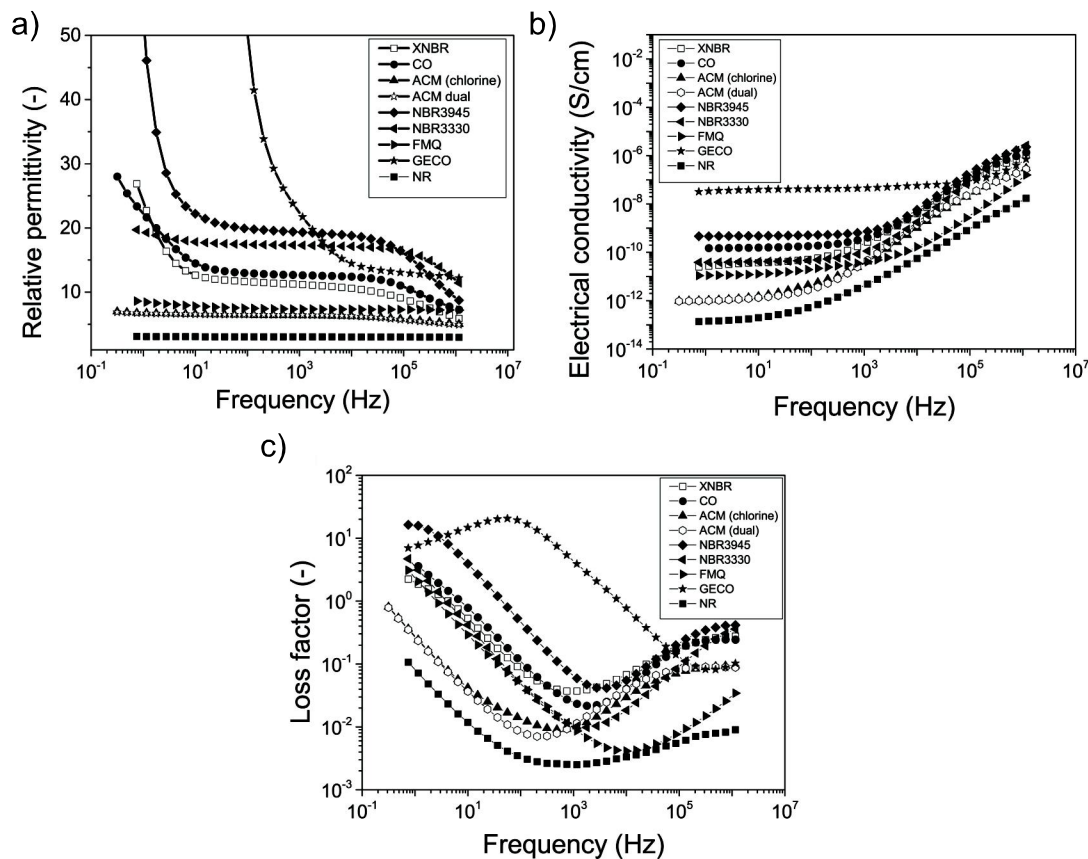


Figure 2. Frequency dependence of a) relative permittivity; b) electrical conductivity; and c) loss factor on different types of crosslinked rubber samples

It should be mentioned here that the most studied and widely used DE material is considered to be ACM, but in the Fig. 2a it is shown that many common commercial rubbers have higher relative permittivities than ACM. NBR with ACN group as a pendant to the macromolecular chains performs quite well due to high polarity of ACN groups, as compared to other rubbers. NR shows the lowest relative permittivity values due to its non-polar nature. FMQ has polar fluorine atoms in the chemical structure, but relative permittivity values stay rather low as fluorine may increase free volume that is known to decrease relative permittivity [21].

In case of GECO a significant increase in relative permittivity is possibly mostly due to the increased electrical conductivity. This terpolymer having poly-oxyethylene in the backbone shows some semi-conductive nature as seen in Fig. 2b in which the electrical conductivity of GECO is $3.39 \cdot 10^{-8}$ S/cm at 1 Hz frequency. The electrical conductivity of all studied materials is dependent on frequency at higher frequency regions which indicates non-conducting nature of the materials whereas at low frequencies they go towards DC conductivity. The frequency dependence of the electrical conductivity of materials is explained by correlated barrier hopping model. [22,23]

Therefore, the electrical conductivity study showed that GECO is not dielectric material and is thus unsuitable for dielectric elastomer actuators. The other polymers studied are insulators and they can be used as dielectric actuator. A low dielectric loss is a desired property for DEAs. It improves actuation performance and increases lifetime of the actuator. The relation of dielectric losses to relative permittivity is expressed by a loss factor. The loss factor of the polymers is presented in Fig. 2c. It can be seen that NR and ACMs have the lowest loss factor, but due to low permittivity values, the generated actuation stress may be lower than of materials having higher loss factor. As dielectric losses of NBR could be reduced by using e.g., peroxide or electron beam curing instead of sulphur curing, it was chosen for further studies. The peak of GECO differs from the others due to semi-conducting nature of the polymer with increased charge mobility meaning that charge carriers respond faster to the applied electric field. Thus loss peak of the material, associated with α -relaxation, is shifted towards higher frequencies [19]. For dielectric materials α -relaxation loss peak is normally observed at low frequency region, around 0.01-10 Hz. [24]

Maxwell pressure (Eq. 1) shows that the actuation stress developed by a constant electrical field is largely dependent on the polarizability of the polymer chains. On the other hand, under a constant stress, the actuation strain depends on the elastic modulus of rubber, assuming a linear relationship of stress-strain behavior, particularly, at very low deformations. In the comparison of different

polymers, the samples were kept under a constant pre-stress when the electrical field was applied. A plot of actuation stress as a function of applied electric field is shown in Fig. 3a.

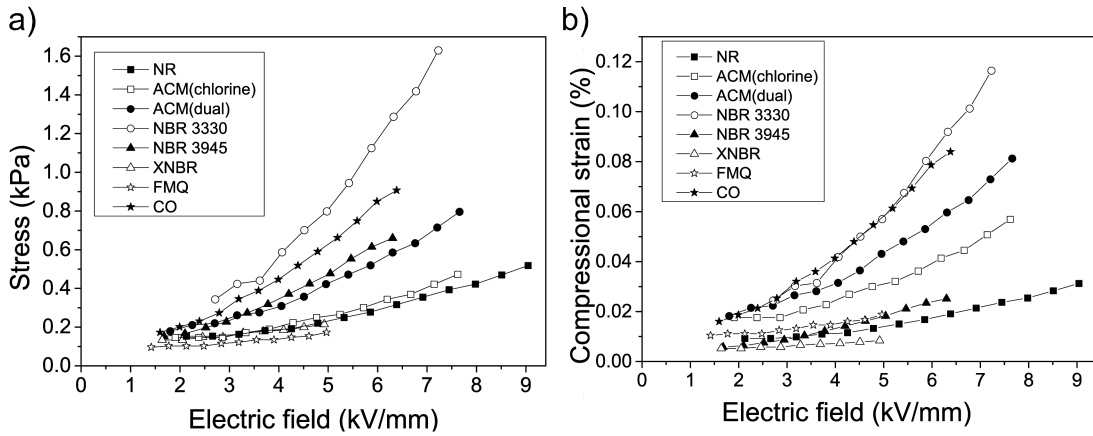


Figure 3. a) Actuation stress of different rubbers and b) Calculated compressional strain of different rubbers.

First of all, it is noted that the stress follows a non-linear pathway against electric field. Due to their viscoelastic nature, the rubbers are showing very complex non-linear behavior in terms of stress or strain. It is also evident from this figure that the actuation stresses differ largely from one another at a given electric field. According to the Eq. 2, the material having the highest relative permittivity should have the highest actuation stress in a certain electric field but this is not valid in our case, indicating the dependence of actuation stress on the modulus values of the materials and other factors. Compressional strain values calculated from Eq. 3 are plotted in Fig. 3b against applied electric field in order to include the effect of modulus into the estimated actuation performance. As seen from the comparison with other rubbers in Fig. 3a and Fig. 3b, a nitrile rubber (NBR3330), commercially available elastomer processed through conventional way, confirms its higher actuation performance and thus is used in the further study. Moreover, it is seen that without pre-strain applied to the test samples the obtained values of actuation stress and strain are notably low. Therefore, it is decided to pre-strain the samples uniaxially to 100% in planar direction during the

following actuation tests in order to reduce elastic modulus and achieve better actuation performance.

Further studies are carried out with different NBRs with variable ACN content. Relative permittivity values of different NBR compounds are presented in Fig 4a. Relative permittivity of NBR enhances when ACN content, and also polarity of rubber, increases owing to the dipolar polarization mechanism that occurs due to rotation of growing number of polar side groups. Actuation stress and calculated actuation strain of the compounds are presented in Fig. 4b and Fig. 4c, respectively. Here, the actuation stress is converted into a compressional strain according to Eq. 3 in order to correlate the dielectric permittivity and the actuation performance. As pre-strain of 100% was applied during actuation measurements, corresponding modulus Y_{100} is used in the calculations. It can be seen that the compressional strain values of the NBR composites are largely depend on the ACN content of the rubber, while stress values show no direct correlation due to the incomparable elasticity of the different rubbers. Different ACN content strongly influences the modulus of the rubbers (Table 3) and an increase in modulus can negatively affect the actuation strain. Thus, at a given electric field, the NBR with the lowest ACN content shows the highest actuation strain regardless of its considerably low permittivity value.

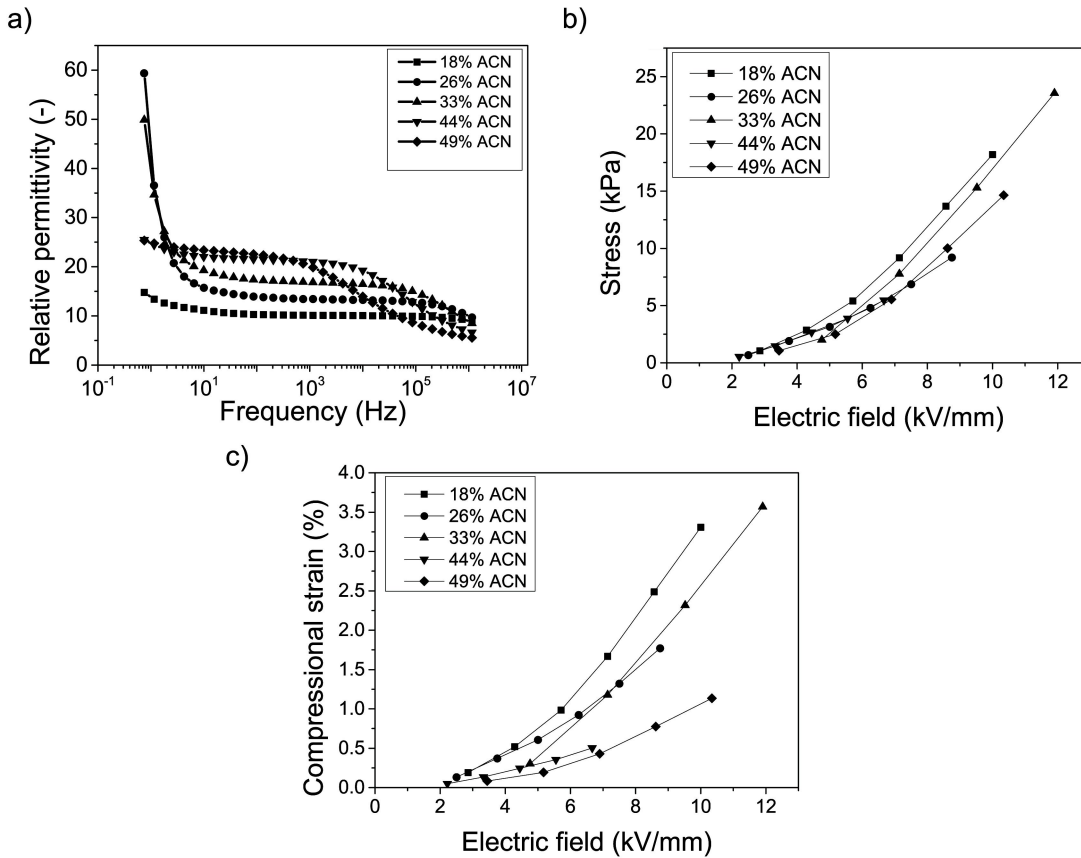


Figure 4. a) Relative permittivity of NBR with variable ACN content b) Compressive strain of different NBRs.

In order to obtain better actuation performance, it would be important to decrease the elastic moduli of the NBR with high relative permittivity by adding plasticizers. For this reason three different plasticizers are studied. All studied plasticizers have lower relative permittivity than NBR, thus some decrease in permittivity values may be expected when 5 phr of plasticizers are incorporated into NBR. DOA is traditional plasticizer used in NBR but it has comparably low relative permittivity (4.58 at 1 Hz). Two other plasticizers, LNBR and dielectric oil F have relative permittivity 16.16 and 1.13 at 1 Hz respectively. The relative permittivity values of plasticized compounds are presented in Fig. 5a. All compounds containing plasticizer show lower permittivity values than the NBR compound without plasticizers. At the plateau region, DOA decreases the relative permittivity more than F and LNBR as seen in small figure in Fig. 5a but there are no

significant differences between the different plasticizers. When the actuation performance of plasticizer containing compounds is studied, one sees that the actuation strain increases even when the permittivity of the compound decreases, as moduli is decreased even more (Fig. 5b). The moduli values for the compounds containing DOA, LNBR, and F are 2.28, 2.17, and 2.40 MPa, respectively, when the modulus of the pure NBR compound is 2.41 MPa. Among the studied plasticizers, DOA - traditional plasticizer used in NBR, gives significantly higher actuation strain than the other plasticizers. This is due to good plasticizing effect with NBR causing decrease in elastic modulus of rubber and due to higher relative permittivity at 1 Hz frequency than LNBR.

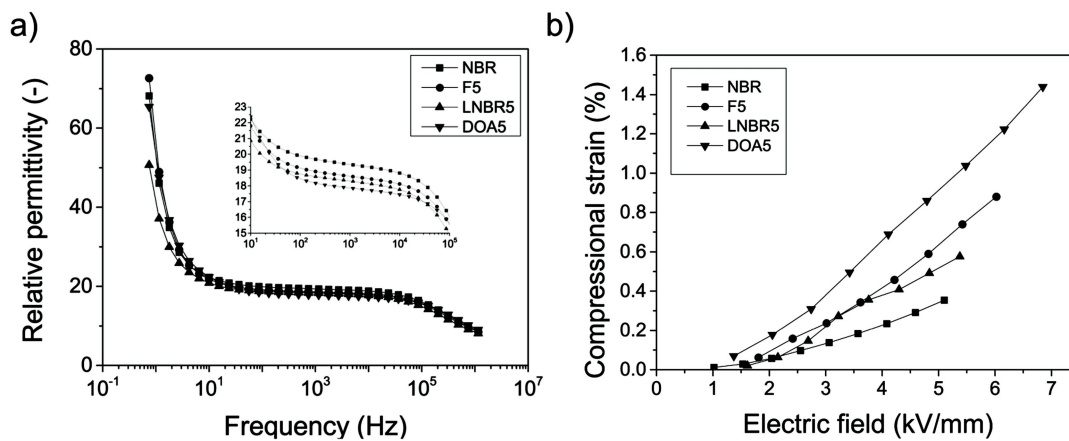


Figure 5. a) Relative permittivity of NBR3945 containing 5 phr of different plasticizers, b) Compressional strain of NBR3945 with different plasticizers.

As NBR containing DOA shows much better actuation performance than NBRs with the other plasticizers, the effect of DOA content on actuation is studied. The relative permittivity of the compounds decreases when the content of the plasticizer increases above 100 Hz frequencies (Fig. 6a insert) as expected due to lower relative permittivities of plasticizers. However, at low frequencies, the trend is not so clear. The relative permittivity increases at higher DOA loads due to increased Maxwell-Wagner polarization (Fig. 6a).

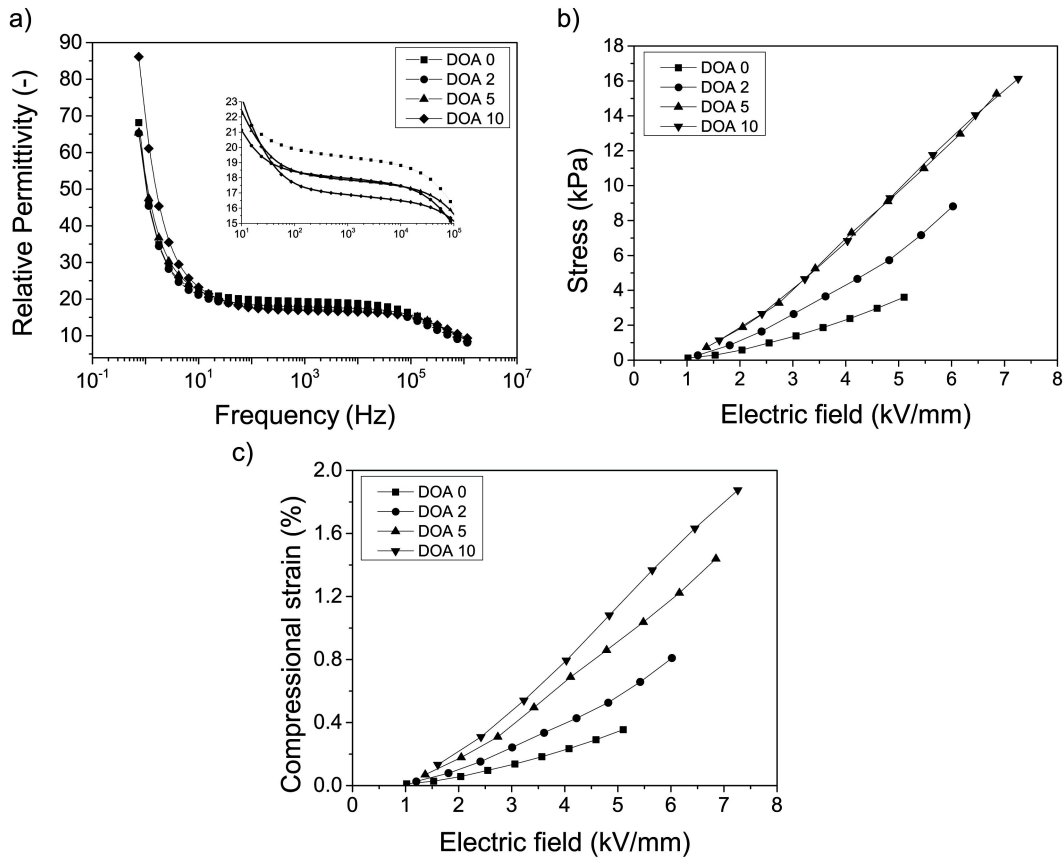


Figure 6. a) Dielectric permittivity of NBR-DOA compounds at different DOA loads, b) Plots of compressional strain against electric field.

Nevertheless, addition of DOA leads to a significant increase in the actuation stress (Fig. 6b). The compounds with relatively high amounts of plasticizer show about three-fold increase in actuation stress at 4 kV/mm compared to the material with no plasticizer. It can be noticed that although incorporation of 5 and 10 phr of DOA results in very similar actuation stress levels, the material with higher amount of plasticizer can be capable of generating larger compression strain. In Fig. 6c the influence of plasticizers on the electromechanically achievable strain is demonstrated with the plots of compression strain vs. electric field. As the DOA only marginally changes the dielectric permittivity of the composite but has large effect on the moduli it can highly affect an actuation strain which is mostly dependent on the ratio of these two material properties. The higher strain is

then achieved by the lowering the elastic modulus of rubber. The result indicates that a simple diene elastomer such as NBR can be exploited to develop high performance electro-mechanically responsive actuators.

CONCLUSIONS

For DEAs, the high relative permittivity and low modulus are desirable properties. In the present study different types of commercially available elastomers are considered for dielectric actuator fabrication to be functioning at relative low electric fields and it is found that NBR has a promising property combination for the actuation performance. The current work revealed that:

- addition of traditional DOA plasticizer is preferred over liquid-NBR and dielectric vacuum pump oil (high- and low-permittivity plasticizing agents respectively) in its effect on actuation performance of the resulting dielectric elastomers
- addition of 10 phr DOA results in more than three-fold increase in the actuation stress and compressional strain at 4 kV/mm with
- increased amount of acrylonitrile content in NBR leads to increase in both relative permittivity and modulus with the latter one having more pronounced effect on actuation performance

Therefore, the material selection of dielectric elastomers for actuator application should not be limited to only well-known silicone and acrylic commercial materials. Fine tuning of actuation performance can be easily achieved by addition of plasticizers and by varying amount of acrylonitrile content of the basic polymers. This type of actuator can be utilized either to generate stronger force or larger strain according to the need of the application.

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Table 1: Polymers and recipes used in the study

Polymer	Trade name	Supplier	Sulphur, phr	ZnO, phr	StA, phr	MBT, phr	TMTD, phr	NaStA, phr	CaCO ₃ , phr	Triazine, phr	Peroxide, phr	Curing time, min/ temp, °C
NBR3330	Europrene N-3330	Versalis	1	5	2		1					8/160
NBR3945	Europrene N-3945	Versalis	0.5	3	2	1						15/160
XNBR	Krynac X740	Lanxess	0.5	5	2	1						15/160
ACM (dual)	HyTemp® 4051EP	Zeon Chemicals L.P	0.3		0.5			3				15/175
ACM (chlorine)	HyTemp® AR 715	Zeon Chemicals L.P	0.3		0.5			3				15/175
CO	Hydrin® H-45	Zeon Chemicals L.P							5	1		15/175
GECCO	Hydrin® T3108	Zeon Chemicals L.P	1	5	2	1						15/175
FMQ	Silastic LS63U	Dow Corning									1	15/170
NR	SMR10		1	5	2		1					8/150
ACN 18%	Krynac 1846	Lanxess	0.5	3	2	1						15/160
ACN 26%	Krynac 2645	Lanxess	0.5	3	2	1						15/160
ACN 33%	Krynac 3345	Lanxess	0.5	3	2	1						15/160
ACN 44%	Krynac 4456	Lanxess	0.5	3	2	1						15/160
ACN 49%	Krynac 4975	Lanxess	0.5	3	2	1						15/160

phr: parts per hundred rubber

SMR: Standard Malaysian Rubber

StA: stearic acid

NaStA: sodium stearate

MBT: 2-mercaptobenzothiazole

TMTD: tetramethyl thiuram disulfide

Peroxide: Trigonox 101-45D-pd

Table 2. Mixing order of the rubber compounds

Polymer	Addition of ingredients, time, min								
	ZnO	StA	Sulphur	MBT/ TMTD	NaStA	CaCO ₃	Triazine	Peroxide	Total mixing time, min
NBRs	2	2	3	3					4.5
XNBR	1	1	2	2					4.5
ACM (dual)		1.5	1.5		1.5				3.5
ACM (chlorine)		1.5	1.5		1.5				3.5
CO						1	1.75		3.5
GECO	0.25	0.75	1.5	1.5					3
FMQ								0.75	2.5
NR	1.5	1.5	2.5	2.5					

Table 3: Moduli of NBRs with different ACN contents

Sample	Young's modulus (Y), MPa	Stress at 100% elongation (Y ₁₀₀), MPa
18% ACN	1.48	0.55
26% ACN	1.51	0.52
33% ACN	1.84	0.66
44% ACN	2.29	1.09
49% ACN	3.09	1.29