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Modifying the MRI, Elastic Stiffness and Electrical Properties of Polyvinyl Alcohol Cryogel Using Irradiation

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ABSTRACT

The aim of this work was to study the effect of radiation on the elastic stiffness, electrical, and MRI properties of polyvinyl alcohol (PVA)-based cryogel (PVA-C). The PVA-C samples were irradiated with a ^{60}Co γ -source, at 2.18×10^6 Rads. The indentation measurements (an indication of elastic stiffness) reduced by about 14.6 % for PVA-3C and 5.7% PVA-6C after irradiation, indicating that the material became harder/stiffer. It was found that MRI relaxation times provide an alternative and non-destructive method to evaluate the radiation effect on PVA-C. The T_1 of PVA-C that had undergone 3 freeze thaw cycles decreased with irradiation by 10%, 25%, and 35% at 1T, 1.89T, and 3T respectively. The T_1 of PVA-C that had undergone 6 freeze thaw cycles decreased with irradiation by 18%, 15%, and 11% at 1T, 1.89T, and 3T respectively. The T_2 of PVA-C decreased with irradiation only at 1T, however this change is hypothesized to be due to the interaction of two spin pools in the gel. The electrical conductivity (σ) and permittivity constant (ϵ) of the unirradiated and γ -irradiated PVA-C samples were measured at different frequencies in the range 40 Hz to 1 MHz. The results demonstrated that the conductivity increased with irradiation by 50% for PVA-3C (3 freeze-thaw cycles) and 75% for PVA-6C (6 freeze-thaw cycles) at frequencies greater than 1KHz. The permittivity decreased with irradiation up to 25% for 3C and 35% for 6C at frequencies less than 1 KHz.

Keywords: PVA-C, irradiation, Conductivity, MRI relaxation times

INTRODUCTION

A PVA-cryogel (or PVA-C) is a hydrogel generated by freezing (-20°C) and thawing ($+20^{\circ}\text{C}$) an aqueous PVA solution [1]. The cryogel molecular organizational structure changes during the freeze-thaw cycles as hydrogen bonds form between water and the hydroxyl groups on the PVA molecules. As the number of freeze-thaw cycles (FTCs) increases the degree of hydrogen bonding increases. A PVA-C gel phantom has been successfully used to mimic organs and tissues in MR and ultrasound imaging studies [2]. Polymer cross-linking is a possible mechanism to explain the hardening of PVA cryogels. Three models have been proposed in an effort to explain the mechanisms inducing the solidification and the cross-linking. They involve hydrogen bonding, polymer crystallite formation, or a liquid-liquid phase separation process [3]. The factors affecting the PVA-C electrical and mechanical characteristics are the number of freeze-thaw cycles, PVA concentration and additive materials.

The methods for determining elastic stiffness include rheological (storage and loss moduli), materials tensile testing, indentation, and ultrasound propagation. Wan, Campbell et al [4] showed a statistically significant difference among 1 to 6 freeze-thaw cycles for materials tensile testing. The indentation method was chosen in this study as the measure of elastic stiffness since it can provide a single measurement with sufficient sensitivity to compare the relative changes of elastic stiffness for each sample.

Campbell et al, have demonstrated that the electrical properties of PVA-C can be changed by adding water soluble salts [5]. The measurement of electrical properties is one of the most convenient methods for studying the polymer structure [6]. These properties are affected by the structure of polymer, type of doping agent, concentration, and manufacturing process [7]. In an earlier work, we demonstrated that there are significant changes in the electrical properties of PVA-C that undergoes 3FTCs as compared to 6FTCs [Moran et al, submitted to Magnetic Resonance in Medicine, Aug 2005].

In this same manuscript we had presented MRI results of plain 3C and 6C (15%) PVA-C. We measured PVA-C that had undergone 3 or 6 freeze thaw cycles at three different fields, 1T, 1.89T, and 3T. It was determined that T_1 increased linearly with field (419ms, 755ms, and 1069ms for 3C, and 391ms, 602ms, and 929ms for 6C). It was also found that T_2 remained relatively constant around 50-60ms, except at 1T where T_2 was approximately 125ms. It was hypothesized that two different spin populations are present in the gel, and that at 1T the 125ms represents an average over two different components. At higher fields, the component hypothesized to be due to the mobile water has an increased T_2 so that it is no longer measured at the low TE's.

Other groups have performed similar measurements. For example Lukas et. al. [8] have determined the MRI relaxation times at 1.5T of a 10% PVA-C over 1-2 freeze thaw cycles. They estimate for example a T_1 of approximately 1050ms and a T_2 of 150ms at 35°C for 10% PVA-C that has undergone 2 freeze thaw cycles. Surry et. al. [2] also measured MRI relaxation times of 10% PVA-C, however they measured PVA-C that had undergone from 1 to 4 freeze thaw cycles. For 3 freeze cycles for example, they measured a T_1 of approximately 680ms, and a T_2 of 115ms at 1.5T.

It has been demonstrated that the PVA-C properties make it suitable for the construction of medical imaging phantoms [2,9,10]. PVA-C polymer offers the opportunity to meet the phantom requirements suitable for impedance imaging techniques, such as Electrical Impedance Tomography (EIT)(Wtorek[11]). Tissue mimicking phantoms made from PVA-C have been used: for MRI to simulate normal and diseased tissue [12], for temperature dosimetry in MRI [8], and for simulations of ultrasound guided breast biopsy [13].

Past studies have examined the affect of radiation induced polymerization (cobalt-60 [14,15] , electron beam[16,17]) of PVA-C hydrogels. Degradation rather than crosslinking occurs with PVA alone. However, PVA-C hydrogels can be crosslinked by irradiation. It has been shown in rheological experiments that both the elastic properties of the storage (G') and the loss (G'') moduli increased with absorbed radiation dose from 0 to 30 kGy [18]. There has not been any study to elucidate the combined affects of freeze-thawing and radiation processes on the properties (electrical impedance, magnetic susceptibility and elastic) of PVA-C.

Ionizing radiation is an efficient tool for sterilization. A great part of single-use medical products are sterilized by this technique [19]. In order to use irradiation to sterilize PVA-C phantoms, it is crucial that the changes in the material properties, in particular the elastic stiffness, the conductivity, and the MRI properties be characterized.

The long term goal of this research is to fabricate PVA-C gels to be utilized in medical devices and to be used as tissue mimicking phantoms for imaging research. A requirement of the imaging studies is that the phantom constructions be visible with sufficient contrast, when viewed with both MRI and impedance imaging. The purpose of the present work is to determine

the changes induced in the elastic stiffness, the MRI properties, and the electrical properties of the cryogel, resulting from γ - irradiation.

MATERIALS AND METHODS

PVA-C Manufacture:

In this study 15% PVA-C specimens were prepared that had undergone either 3 or 6 freeze-thaw cycles. The details of the manufacture have been detailed elsewhere [Moran et al, submitted to Magnetic Resonance in Medicine, Aug 2005]. Samples will be designated “PVA-3C” for 15% PVA-C with 3 freeze-thaw cycles or “PVA-6C” for 15% PVA-C with 6 freeze-thaw cycles.

Elastic stiffness (indentation):

An indenter was devised that applies a minor load of 0.1 N and a major load of 0.71 N to the flat surface of a PVA-C specimen through a 2.5 mm diameter hemispherical foot. The relative depth of penetration between the major and minor loads was recorded for 10 measurements on each specimen. This method is similar to the method described in ASTM D 1415 – Standard Test Method for Rubber Property – International Hardness [20]. However the major load applied according to ASTM D1415 was judged to be too severe for the cryogels used in this study thus the major load of 0.71N was used. A smaller depth of penetration indicates a harder/stiffer material.

MRI:

MRI was performed on all of the samples at field strengths of 1T (OrthoOne, ONI corp, Wilmington MA, www.onicorp.com), 2T (Magnex magnet, SMIS console), and 3T (IMRIS 3T, Innovative Magnetic Resonance Imaging Systems, Winnipeg, Canada). The spin-lattice relaxation time, T_1 , and the spin-spin relaxation time, T_2 were measured at each field strength.

For T_1 measurements a standard 2D inversion recovery sequence was repeated with different inversion times (TI). The imaging parameters were as follows: 6cm FOV, 64x64 matrix, TR=4000ms, TE=13.8ms, TI=20, 60, 100, 200, 400, 600, 800, and 1000ms at 1T. At 2T and 3T, the same parameters were used except TR=5500ms, and TI=20, 60, 100, 200, 300, 400, 500, 600, 700, 800, 1000, 2000 and 3000ms. Signal intensity was determined in a cross-sectional region of interest in each image for each of the PVA-C samples. The T_1 recovery can be described by the equation:

$$S(TI) = S_0 (1 - 2 \exp(-TI/T_1)) \quad (1)$$

where $S(TI)$ is the signal intensity of the selected ROI of the image and S_0 is the signal at time $TI=0$. This equation can be rearranged to form:

$$TI/T_1 = -\ln [\frac{1}{2} - S(TI)/(2 S_0)] \quad (2)$$

from which T_1 can be obtained by a linear regression where both S_0 and T_1 are fitted parameters.

T_2 was measured at 1T using a 2D spin echo sequence with: a 6cm FOV, 64x64 matrix, TR=4000ms, and TE=25, 50, 100, 200, 400, and 500ms at 1T. The same parameters were used at 2T and 3T except TR=5500ms. The peaks of the echoes were fit to the equation:

$$S(TE) = S_0 \exp(-TE/T_2) \quad (3)$$

where $S(TE)$ is the signal intensity of the selected ROI, and S_0 is the signal intensity at $TE=0$. In a previous manuscript, [Moran et al, submitted to Magnetic Resonance in Medicine, Aug 2005] we had postulated the presence of 2 different T_2 components. At 1T, there was not sufficient resolution in the time domain to fit to 2 exponentials. However at 2T and 3T the data was fit assuming a bi-exponential behaviour.

Impedance Analysis:

An Agilent (Agilent Technologies, Mississauga, ON) 4294A Impedance analyzer was used together with an Agilent 16452A liquid test fixture to measure the conductivity and permittivity of the PVA-C. The details of the procedure have been outlined elsewhere [Moran et al, submitted to Magnetic Resonance in Medicine, Aug 2005].

The conductivity, σ , was calculated using:

$$\sigma = t / (ARp) \quad (4)$$

where t is the spacing between the electrodes, A is the electrode area (radius = 38.0mm), and R_p is the (parallel) resistance measured.

The permittivity, ϵ , was calculated using:

$$\epsilon = (t C_p)/(A \epsilon_0) \quad (5)$$

where C_p is the parallel capacitance measured in the liquid fixture, and $\epsilon_0=8.85e^{-12} C^2/(Nm^2)$ is the permittivity of free space.

Irradiation:

The PVA-C samples were irradiated with the calibrated ^{60}Co γ -rays source in the McMaster Nuclear Reactor. The source was a cylindrical Cobalt-60 of approximately 5600 Curies (single 'pencil'). The samples were arranged equidistant upon a circle of 15.2 cm from the centerline of the source. The high dose rate was determined to be 51.4×10^4 Rad/hr. The total delivered dose was 2.18×10^6 Rads.

Statistics:

Multiple electrical measurements were made to determine the average and standard deviation. Standard t-tests were performed to determine if measurements before and after irradiation were different at the $p<0.05$ level.

RESULTS

The results of the elastic stiffness (indentation) procedure are summarized in Figure 1.

The MRI relaxation time measurements are summarized in Figures 2 and 3. For unirradiated samples as is shown in Figure 2 there is an increase in T_1 with field strength while T_2 is less sensitive to field strength. This behaviour has been documented before [Moran et al, submitted to Magnetic Resonance in Medicine, Aug 2005]. Figure 3 shows the T_1 and T_2 of irradiated PVA-3C and PVA-6C samples measured at the different field strengths. γ -irradiation appears to have decreased the T_1 of the PVA samples at all field strengths. At 1T, the T_2 data fit a single exponential well whereas at 2T the data was better fit by a bi-exponential behaviour. The higher T_2 component (300ms) is not reported. At 3T the T_2 data fit a single exponential relaxation.

The electrical properties are summarized in Figures 4, 5 and 6. Figure 4 shows the conductivity spectra for the PVA-C samples before and after γ -irradiation in the frequency range of 40Hz-1MHz. Figure 4 indicates that there is a significant decrease in the conductivity with freeze thaw cycles for unirradiated samples and the conductivity has significantly increased with radiation. The resistance plot (Fig 5) shows an equivalent change in behavior as conductivity. The irradiated PVA-C has lower resistance than the unirradiated case.

In Figure 6, the permittivity is plotted versus frequency. Note that in this plot, the error bars fall within the data symbols. There is a significant decrease in the permittivity for both PVA-3C and PVA-6C at the lower frequencies. For frequencies greater than approximately 1KHz, this difference in the permittivity of the samples becomes less.

DISCUSSION

The results indicate a change in the elastic stiffness, the MRI, and the electrical properties of PVA-C between 3 and 6 freeze thaw cycles as well as before and after γ -radiation.

For the unirradiated specimens the indentation in the PVA-6C is significantly less than PVA-3C, indicating that the PVA-6C specimen is harder/stiffer. The difference is statistically significant difference ($P < 0.001$). This supports the tension measurements reported by Rosiak et al [21] showing that PVA-6C was stiffer than PVA-3C. The irradiated specimens for both the PVA-3C and PVA-6C were harder/stiffer by 14.6% and 5.70% respectively, than the unirradiated samples. The difference (pre and post radiation) for the PVA-3C was confirmed statistically ($P < 0.001$), but the difference for PVA-6C was not statistically significant ($P > 0.05$).

The increased hardness/stiffness after irradiation may be explained by an increased crosslinking induced by the irradiation. Since PVA-6C has undergone more entanglements than PVA-3C resulting from the freeze-thaw process, the smaller change in stiffness after irradiation (which was not significant) may be attributable to fewer available sites for crosslinking.

Both the spin-lattice (T_1) and the spin-spin (T_2) relaxation times can provide information regarding the nature and frequency of molecular motion occurring within materials. The spin-lattice relaxation time in a polymer is independent of molecular weight and is mainly determined by the main chain motion. In contrast, the spin-spin relaxation time decreases with increasing molecular weight and hence viscosity [22]. T_2 is normally determined by the slowest motions occurring within the sample [23-24]. This will depend also upon the radiation dose, since a polymer may be cross-linked using ionizing radiation. The number of cross-links is proportional to the radiation dose, r , and the efficiency of the process which is stated as a G value, $G(X)$ [25].

The G-value is defined as the number of reactions or events (in this case number of cross-links) per 100 electron volts (eV) of absorbed energy. The initial effect of radiation is to increase the molecular weight, M_w ,

$$M_w = M_w^0 / (1 - \delta) \quad (6)$$

Where M_w^0 is the initial molecular weight and δ is the number of cross-links units per weight average molecule [22]. The gelation dose, r_g , is reached when $\delta=1$. One crosslink involves two cross-linked units, one on each molecule. Any energy absorption from radiation of $(0.624 \times 10^{20} r_g) \text{ eVg}^{-1}$ produces $N_0/2M_w$ cross-links per gram, where N_0 is Avogadro's number and therefore the gelation dose is as follow [22]

$$r_g = 4.84 \times 10^5 / M_w G(X) \quad (7)$$

G-values for many reactions are in the range from 0.1 to 10, with 1 being a typical value for cross-linking pure polymers.

The spin-lattice relaxation in a polymer has been described by a single exponential relaxation time. In the unirradiated and low molecular weight case, because of the great flexibility of the polymer chain, and because protons are in rapid motion, the spin-spin relaxation decay has a single exponential behavior (fast exchange). For the irradiated polymer, particularly above the gelation dose, the spin-spin relaxation decay can be described by a double exponential function of the form [21-22]

$$A(t)/A(0) = fe^{-t/T_{2L}} + (1-f)e^{-t/T_{2S}} \quad (8)$$

Where $A(t)/A(0)$ represent the normalized (at $t=0$) signal amplitude and T_{2L} and T_{2S} refer to the long and short relaxation times respectively and f is the fraction of protons relaxing with T_{2L} . As the dose increases f decreases and more of the signal decay comes from the short

component. The dose at that point where the short component starts to decrease depends on the initial molecular weight of the polymer [25]. The cross-linked network can decrease the T_2 which reflects the more restricted nature of translational chain motion of the network structure. So the spin-spin relaxation is sensitive to the cross-linked network structure.

At the highest dose, T_1 [25], which is most sensitive to molecular motions occurring at frequencies near to the resonance frequency can be reduced. At higher fields a larger decrease in T_1 relaxation time is expected which was indeed observed in MRI measurements at 2T and 3T.

Note that at 1T, the T_2 data fit a single exponential well, indicating that if 2 water pools (one hydration layer and one free mobile water) are present, that there is not sufficient resolution in the time points to separate these components. At 2T however the data was better fit by a bi-exponential behaviour. A higher T_2 component (300ms) is postulated to be due to more mobile water, whereas the second component (approx 50ms) is due to water associated with the PVA-C. At 3T the T_2 data fit a single exponential relaxation yielding a T_2 of approximately 50ms. This fits a single exponential probably because the mobile water component has a larger T_2 at 3T, and was not adequately resolved at the lower TE values.

T_2 changed little following radiation except at 1T. It is postulated that this is resulting from the 1T measurement arising from an average of a shorter (50ms) water component associated with the PVA-C with a longer (150ms) more mobile water component. As discussed above, if the signal corresponds to a recovery such as that indicated in equation 8, then radiation will not modify the T_2 's of these components, but rather the weightings of these components. Thus at 1T following radiation, a T_2 decrease from 96ms to 70ms for the 3C sample for example indicates an increase in the weighting of the short component as expected.

The data for conductivity shows that the conductivity increases with γ -radiation. It is speculated that this is due to radiation induced polymeric cross-linking. This cross-linking reduces hydrogen bonding between the PVA and water, creating energetic free electrons, ions, and radicals, which are able to migrate through the polymer, eventually changing the electrical conductivity. A clear description for conduction mechanism of disordered polymers has not been adapted to the data due to complexity of their structure [26].

CONCLUSION

The elastic stiffness, MRI relaxation times, and electrical properties have been measured for unirradiated and irradiated PVA-C samples (3 or 6 freeze-thaw cycles). All of these techniques illustrated clear differences due to radiation exposure on the PVA cryogel polymer.

Irradiation of PVA-C samples increased their elastic stiffness by 14.6% for PVA-3C and by 5.7% for PVA-6C.

The T_1 relaxation times were decreased by irradiating the samples. The T_1 of PVA-C that had undergone 3 freeze thaw cycles decreased with irradiation by 10%, 25%, and 35% at 1T, 1.89T, and 3T respectively. The T_1 of PVA-C that had undergone 6 freeze thaw cycles decreased with irradiation by 18%, 15%, and 11% at 1T, 1.89T, and 3T respectively. The T_2 of PVA-C decreased with irradiation only at 1T, however this change is hypothesized to be due to the interaction of two spin pools in the gel.

Irradiation increased the conductivity of PVA-C by up to 50% for 3C and 75% for 6C at frequencies greater than 1 KHz. This can be explained from the basis that radiation induces cross-linking and therefore ions and free radical are formed which can be trapped in the bulk of

material. The permittivity was also decreased with irradiation up to 25% for 3C and 35% for 6C at frequencies less than 1 KHz.

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Figures:

Figure 1: Variation of the elastic stiffness (indentation) of unirradiated and irradiated PVA-3C and PVA-6C

Figure 2: Variation of T1 and T2 values for unirradiated PVA-3C and PVA-6C for various field strengths

Figure 3: Variation of T1 and T2 values for irradiated PVA-3C and PVA-6C for various field strengths

Figure 4: Variation of electrical conductivity for unirradiated and irradiated PVA-3C and PVA-6C versus frequency

Figure 5: Variation of electrical resistance for unirradiated and irradiated PVA-3C and PVA-6C versus frequency

Figure 6: Variation of permittivity for unirradiated and irradiated PVA-3C and PVA-6C versus frequency

Fig 1

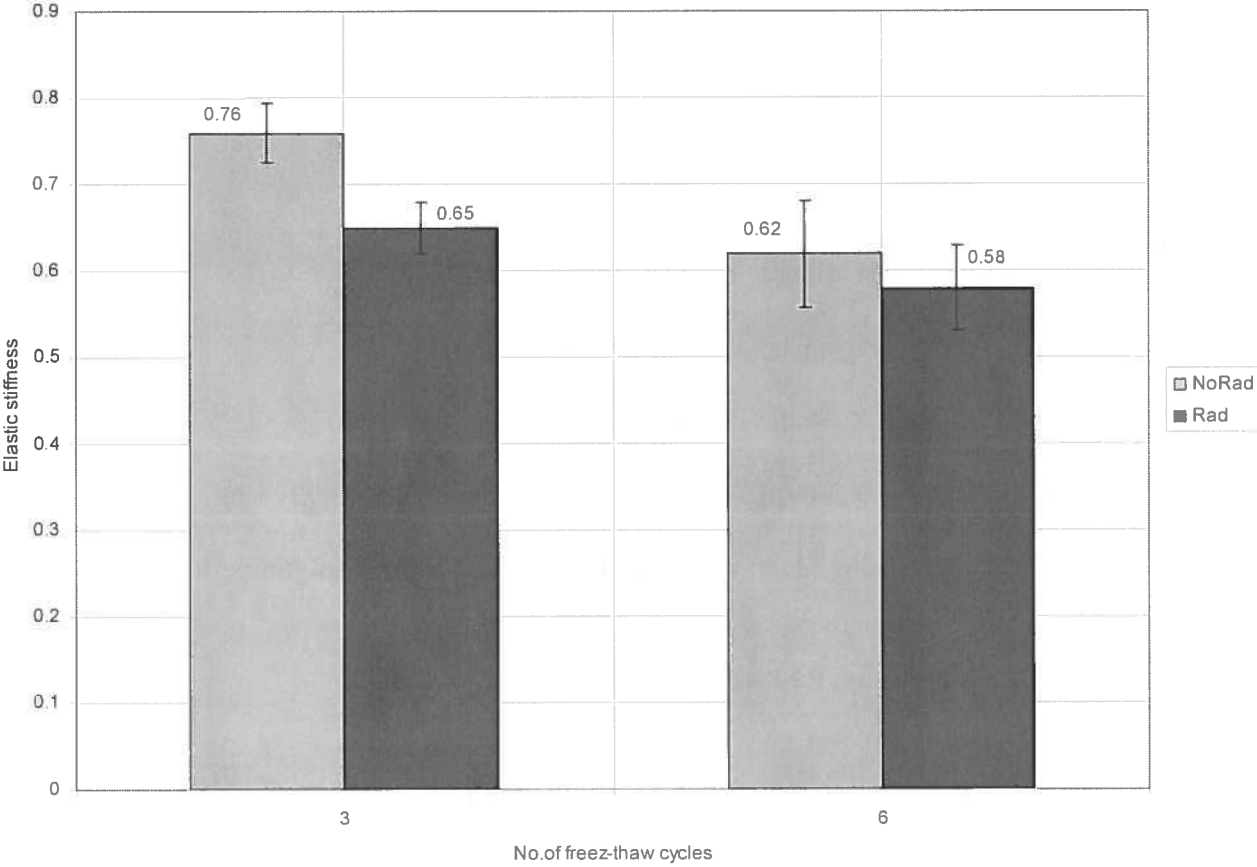


Fig 2

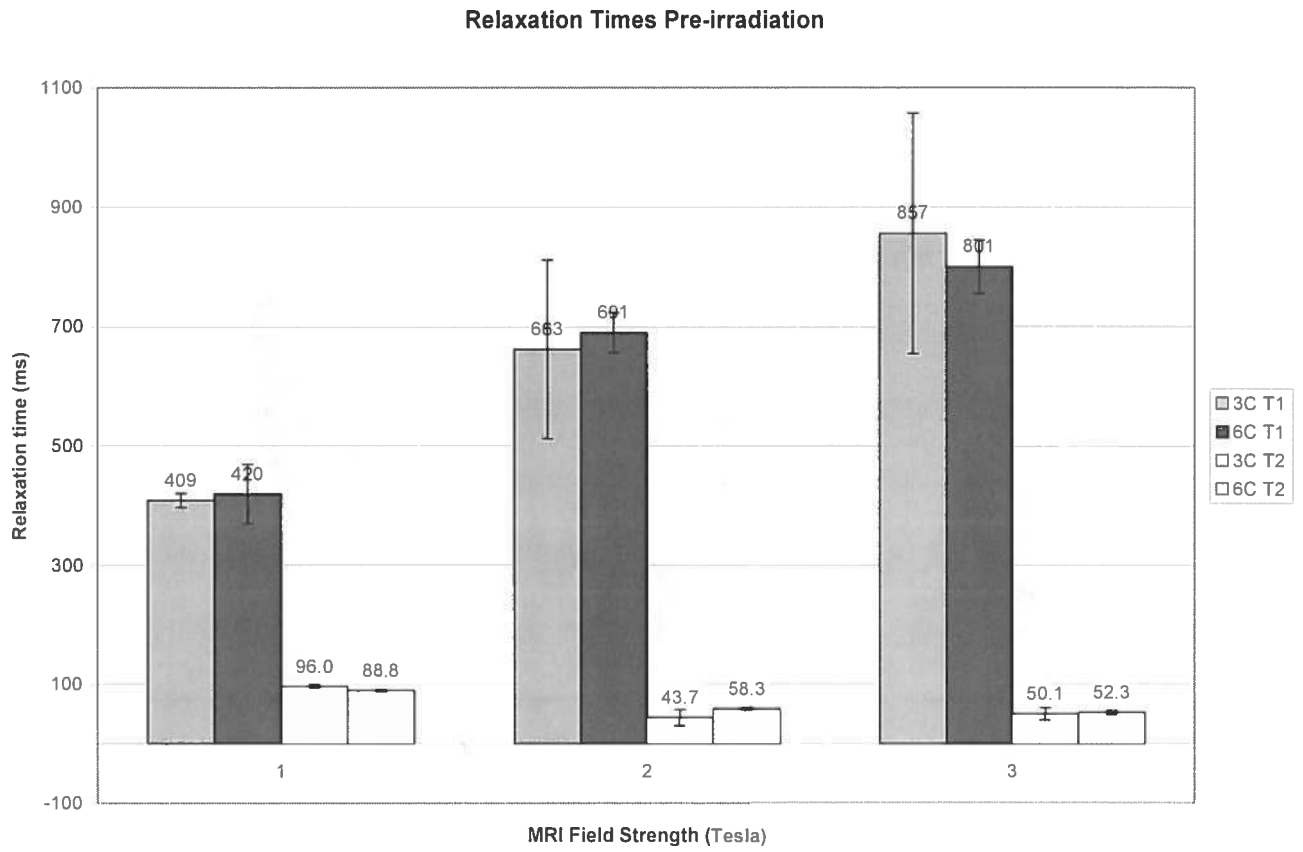


Fig.3:

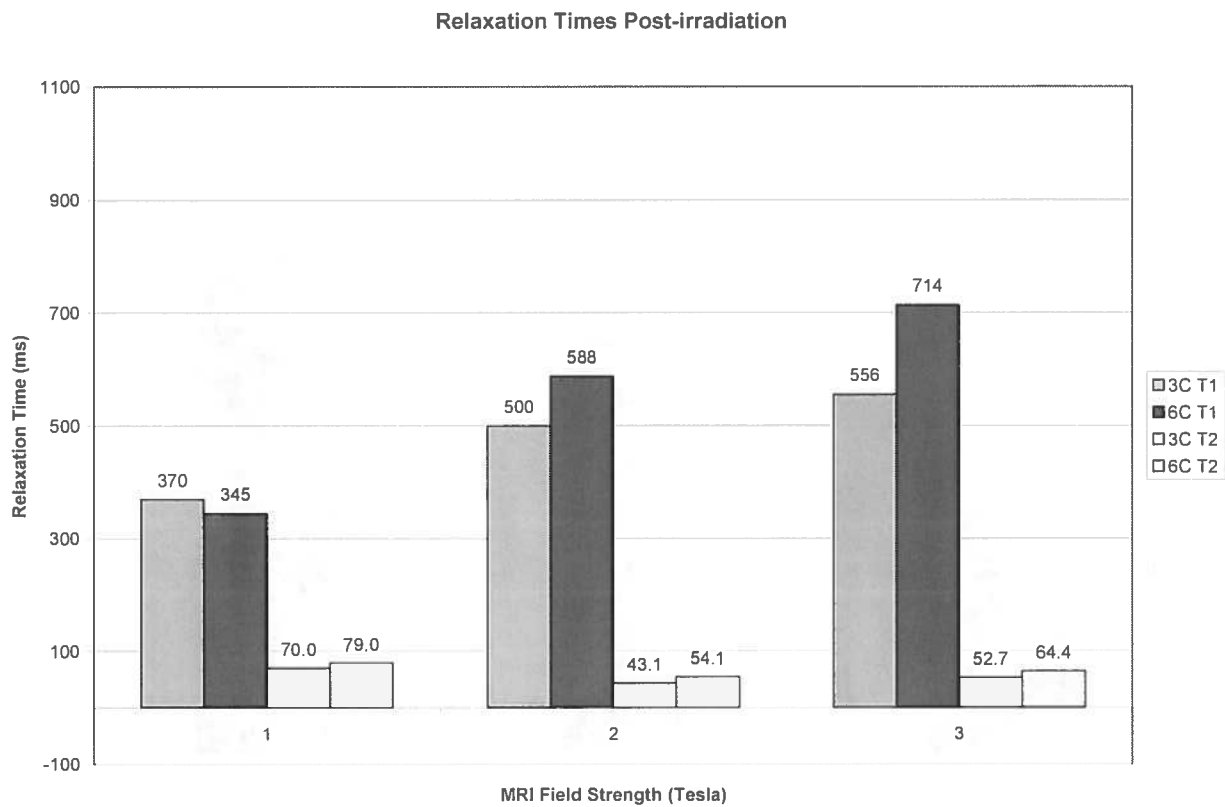


Fig.4:

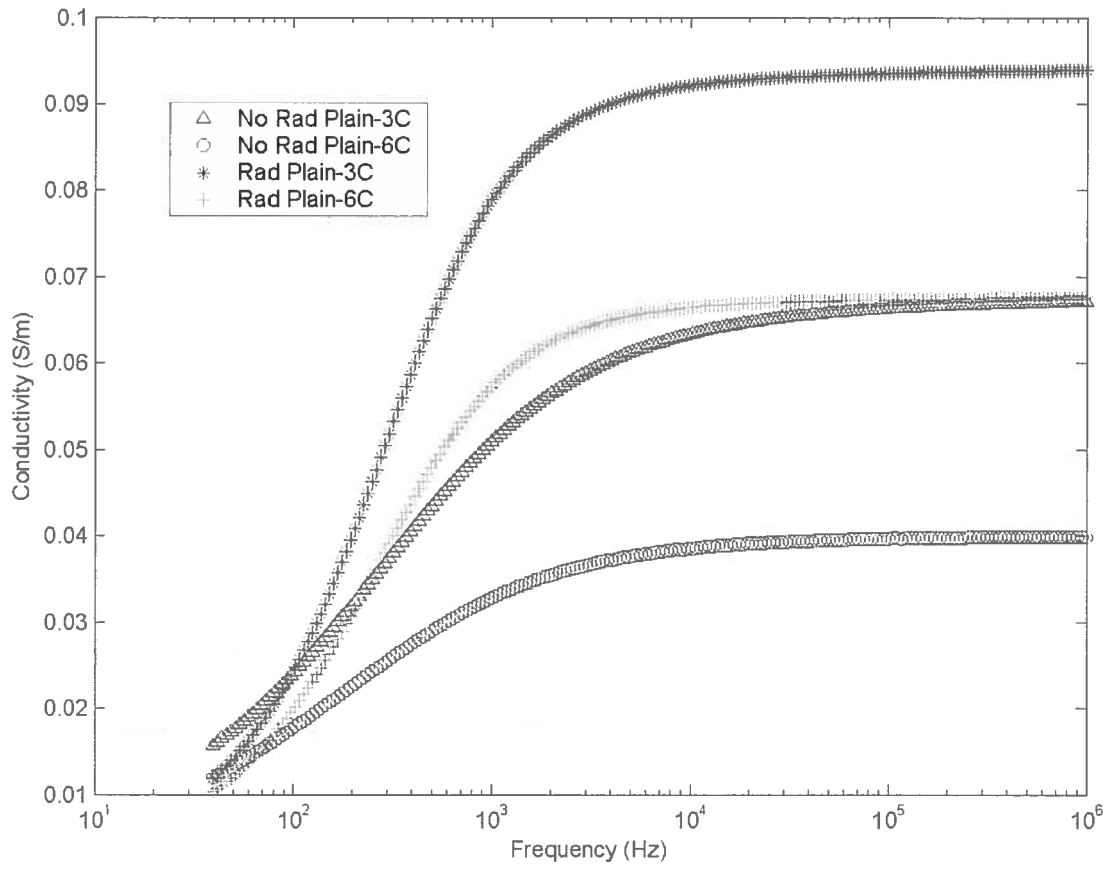


Fig.5:

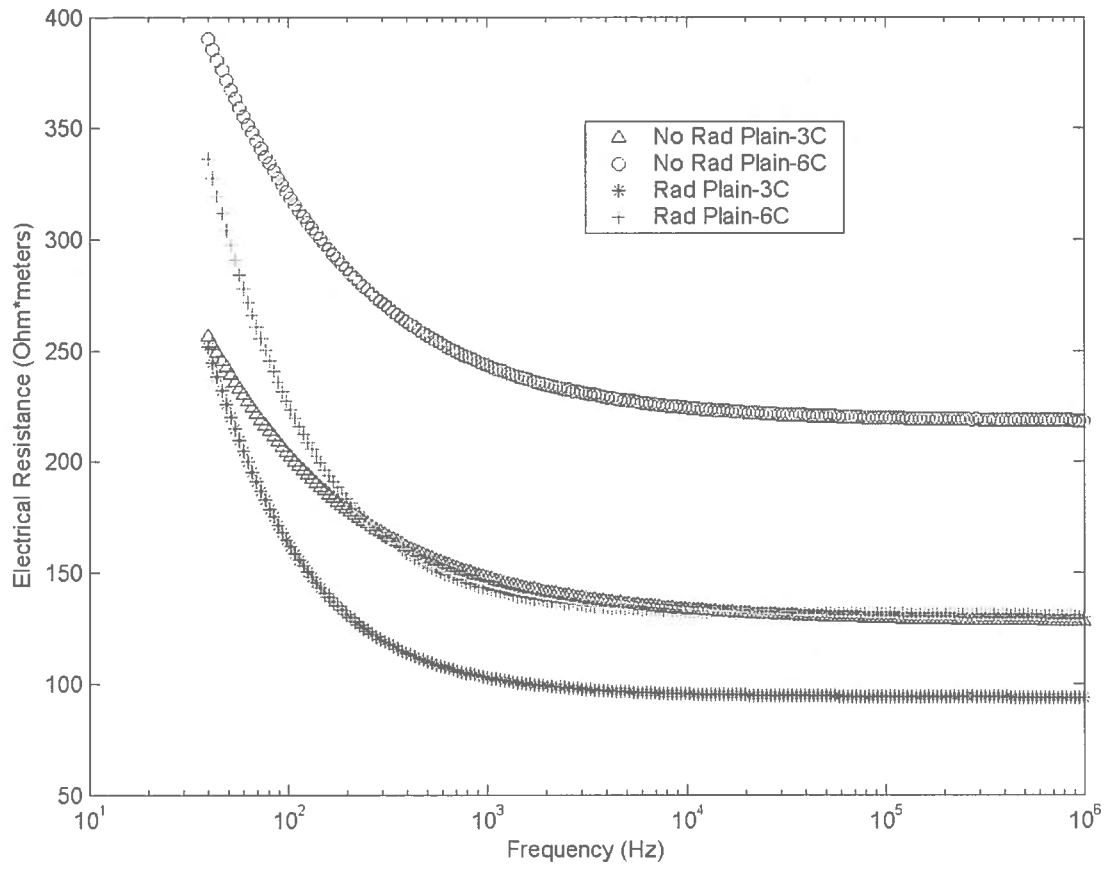


Fig.6:

