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Considerations for Contractile Electroactive Materials and Actuators

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ABSTRACT

Ras Labs produces electroactive polymer (EAP) based materials and actuators that bend, swell, ripple, and now contract (new development) with low electric input. In addition, Ras Labs produces EAP materials that quickly contract and expand, repeatedly, by reversing the polarity of the electric input. These recent developments are important attributes in the field of electroactivity because of the ability of contraction and contraction-expansion to produce biomimetic motion. The mechanism of contraction is not well understood. Radionuclide-labeled experiments were conducted to determine the mechanisms during contraction of these EAPs.

Keywords: electroactive, EAP, contraction, artificial muscle, plasma treatment, electrode, actuator

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1. INTRODUCTION

There has been a great deal of interest in the search for materials that can transfer electrical energy directly into mechanical energy, analogous to our muscles converting electrical and chemical energy into mechanical energy to produce movement. Advances in these endeavors have been achieved around the world, using materials such as poly(vinyl alcohol), ionized poly(acrylamide), poly(acrylic acid), poly(acrylic acid)-co-(poly(acrylamide)), poly(2-acrylamide-2-methyl-1-propane sulfonic acid), poly(acrylic acid), poly(methacrylic acid), poly(styrene sulfonic acid), quarternized poly(4-vinyl pyridinium chloride), and poly(vinylbenzyltrimethyl ammonium chloride), and numerous other materials. SRI International holds many patents (US Patents 7,064,472,¹ 7,049,732,² 6,940,211,³ 6,891,317,⁴ 6,882,086,⁵ 6,812,624,⁶ 6,781,284,⁷ 6,768,246,⁸ 6,707,236,⁹ 6,583,533,¹⁰ 6,545,384,¹¹ 6,376,971,¹² 6,543,110¹³) and publications^{14,15,16,17,18} in this area. Patents are additionally held by the Nippon Zeon Corporation (JP Patents 03234770,¹⁹ 2001258275²⁰) the Nitta Corporation (US Patent 5,977,685,²¹ JP Patents 2001258275,²² 07216244²³), and many others. Piezoelectric materials have also been investigated for use as electrically responsive materials; however, most piezoelectric materials undergo length changes of only a fraction of one percent.

Cross-linked, ion-containing electroactive polymeric (EAP) materials developed by Ras Labs have shown good results in terms of strength and electroactivity, with the ability of some of these EAP materials to contract more than 80 % within 1 minute at 50 V, and then return to their original size and shape when the electric input is stopped, repeatedly. Recently, stronger EAP materials were produced that quickly contract and then expand, repeatedly, by reversing the polarity of the electric input. The contraction-expansion cycles can be cycled repeatedly, lending these EAPs to applications where life-like motion is desired.

One of the challenges of EAPs is that when they undergo motion, the electrodes, even if embedded, can become detached, which causes actuator failure. Plasma treatment and other metal treatments were explored to improve the interface between the electroactive material and the embedded electrodes. By improving the metal-polymer interface, the EAP material and the electrode(s) can work as a unit, where the electrode(s) delivers the electric input and can also serve as a tendon between the EAP material and a lever.

The mechanism of movement in EAP materials is somewhat understood, particularly for bending,^{24,25} however, the mechanism of contraction in EAP materials is less common and not as well understood. Tanaka,²⁶ Osada,²⁷ Smela,^{28,29,30} and others have investigated the contraction behavior of smart materials from change in pH, change in solvent, and electric input. An experiment with radionuclide-labeled electrolyte and tritium labeled water was performed to definitively determine the chain of events that occur when the EAP materials developed by Ras Labs contract.

2. EXPERIMENTAL

Electroactive polymeric materials: The EAP materials were synthesized using the monomer methacrylic acid (Aldrich Chemical Co., Inc., 99 %), the initiator 1-hydroxycyclohexyl phenyl ketone (Aldrich, 98 %, or Irgacure 184® from Ciba Geigy, Inc.), and the cross-linking agents ethylene glycol dimethacrylate (Aldrich, 98 %), poly(ethylene glycol) dimethacrylate (Aldrich), or 1,1,1-trimethylolpropane trimethacrylate (Aldrich, technical grade). Inhibitors were removed using a column of inhibitor removal beads (Aldrich). Typically, 1 weight percent 1-hydroxycyclohexyl phenyl ketone was used. The amount of cross-linking agents varied between 0.5 and 10 weight percent depending upon the physical properties desired. Glycerol or distilled, oxygen-free water was used as a diluent. Different formulations were used to produce EAPs that bend, EAPs that swell, and EAPs that contract. Multi-phasic gels, with different zones having different physical properties, were also synthesized. A custom built, mercury bulb, ultraviolet oven ($\lambda = 300 - 450$ nm) was used to photopolymerize the cross-linked networks for typically 3 to 10 minutes. The reaction mixtures were polymerized in glass vials or test tubes. Molds were used for more complex shapes. For EAP materials with embedded electrodes, the electrodes were positioned in the monomer/initiator/cross-linking agent(s) mixture prior to the photopolymerization. Once the material had polymerized, electrolyte solution(s) was added to the material, which typically swelled over a finite amount of time, varying from overnight to a few days to reach final volume. For non-water based systems, gels could be dried, and then reconstituted with electrolyte(s).

Testing for electroactivity: A 50 V Edvotek modified mini-electrophoresis apparatus was used for preliminary testing for electroactivity. Weight was determined before and after electric impulse. Physical movement of these EAP materials was visible to the naked eye and could also be measured using a simple grid. A HQ Power® adjustable DC-regulated power supply, 0-15V/2A, was used for testing at lower voltages.

Metal electrodes: Stainless steel aircraft grade (McMaster-Carr, ASTM-A580, 302/304, 8860K11) lockwire (0.015 gauge) and foil (0.002 inch thick) was used because this is a non-hardening stainless steel with good corrosion resistance and high temperature service. Titanium (ESPI, Knd1222, G2) wire (0.001, 0.003, and 0.005 inch diameters) and foil (0.002 inch thick) were used because of the high strength and good corrosion resistance of titanium.

Plasma treatment: Nitrogen, synthetic air, hydrogen, helium, and oxygen plasmas were used on stainless steel and titanium foils. Nitrogen and oxygen plasma were used on stainless steel and titanium wires. The plasma was generated using a 25 MHz RF power source at 10 to 25 W in a near vacuum (typically 1 to 2 mTorr). The metal samples were attached to an I-shaped current probe that inserted into the plasma chamber source with -35 V (0.5 to 5 mA) electric potential, which attracted the bombardment of the positive ions towards the metal samples. All metals were degreased prior to plasma treatment.

Radionuclide-labeled experiments: Solutions containing a known amount of tritium (H-3) and sodium-22 (Na-22) in the form of sodium chloride (NaCl) in water were used. A standard stock solution of Na-22 (NaCl in water) with 9.957 microcuries/g (Isotope Products) was diluted in a 1 g standard stock solution: 99 g unlabeled 0.5 % w/v NaCl solution, producing a final activity of 0.1 microcuries/mL. A standard solution of H-3, 1.010 microcuries in 50 g of water, from PPPL's stock calibration inventory in the Princeton Environmental Analytical Radiological Laboratory (PEARL), was diluted using 8.6 g stock H-3 solution: 91.4 g unlabeled 0.5 % w/v NaCl solution, producing a final activity of 0.001126 microcuries/mL (2500 dpm/mL) in 100 g of water. 20 g of the diluted Na-22 solution was mixed with 20 g of the diluted H-3 solution (total volume of 40 g), with a specific activity of 0.05 microcuries/g Na-22 (111,000 dpm/g) and 0.0056 microcuries/g H-3 (1250 dpm/g). Dried samples of the EAPs were immersed in this aqueous NaCl solution containing both Na-22 and H-3 radionuclides for over 24 hours. The EAPs absorbed the liquid and the overall size of the EAPs increased. Immediately before the contraction experiment, the EAP samples were placed in a solution of unlabeled 0.5 % w/v NaCl solution. The EAPs were activated with electric input (50 V), with aliquots taken from the media surrounding the EAPs at 5, 15, 30, 45, 60, 75, and 90 seconds. For the control experiment, EAPs were placed in an unlabeled 0.5 % w/v NaCl solution and, with no electric input, aliquots were taken from the media surrounding the control EAPs at the same timed intervals. Liquid scintillation chromatography (LSC, Perkin Elmer Tri-Carb Detector) was used to measure H-3 with a standard H-3 in water protocol of 1 mL sample/aliquot for 10 mL aqueous LSC mixture. Gamma-ray spectroscopy (GRS, high resolution Intrinsic Germanium Detector with a beryllium window) was used to measure Na-22 by placing 1 mL sample/aliquot on filter paper in a 47 mm Petri dish and counting with a GRS unit calibrated to this geometry. Following each contraction experiment, the EAP (including control EAPs) were placed in a clean unlabeled 0.5 w/v % NaCl solution for about a week. This equilibration step was to determine the amount of Na-22 and H-3 left in the EAP after the contraction experiment and also to determine if there is any leaching of the radionuclides from the EAPs. After the contraction experiment and the equilibration, the EAPs were analyzed using GRS to determine the amount of Na-22 left remaining in the EAPs themselves.

3. RESULTS AND DISCUSSION

Cross-linked EAP materials produced from predominantly ion-containing monomers have shown good results in terms of strength and electroactivity, particularly contraction. Recent synthetic breakthroughs have produced an electroactive material that contracts 80 % within 1 minute at 50 V (Figure 1). When the electricity is stopped, the EAP returns to its original size and conformation. In addition, stronger EAPs were developed that contract to about 50 % from their original weight within 1 minute at 50 V, and when the polarity was reversed, expand back to their original weight (sometimes exceeding original weight) within 1 minute at 50 V. The contraction-expansion cycles can be cycled repeatedly. These recent developments are important in the field of electroactivity because of the ability of contraction and contraction-expansion to produce biomimetic life-like motion.

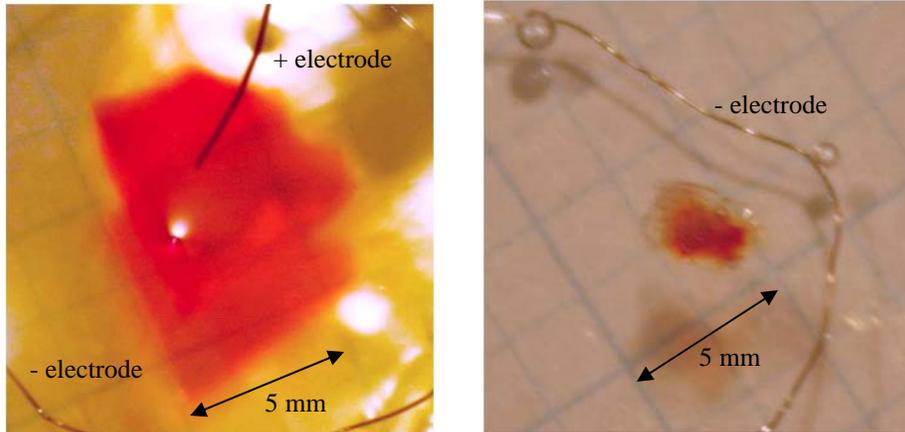


Fig. 1 A. Weight ($t = 0$) = 0.11 g B. Weight ($t = 1$ minute, 50 V) = 0.01 g
 Note: Red dye added to (uncoated) EAP to improve visualization. EAP surrounded by electrolyte solution. Electrode removed after experiment for weight determination.

Using EAP materials in an actuator presents some interesting challenges. For actuators designed to act like an artificial muscle, the EAPs were synthesized with at least one embedded electrode. If the material was particularly electroactive, the material would move so quickly that the embedded wire would disengage, causing the actuator to lose its electric impulse (Figures 2 and 3).

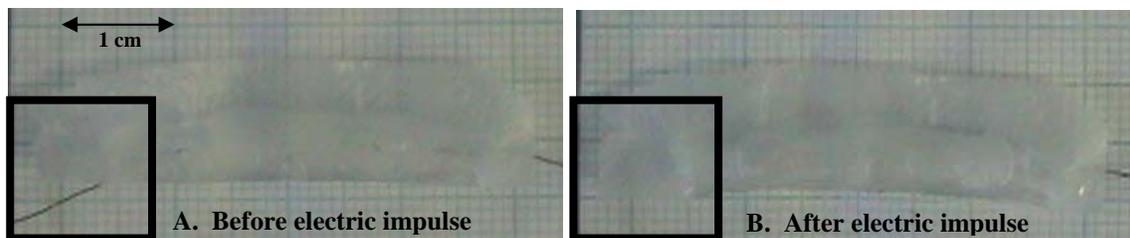


Fig. 2. Coated EAP material with embedded electrodes.

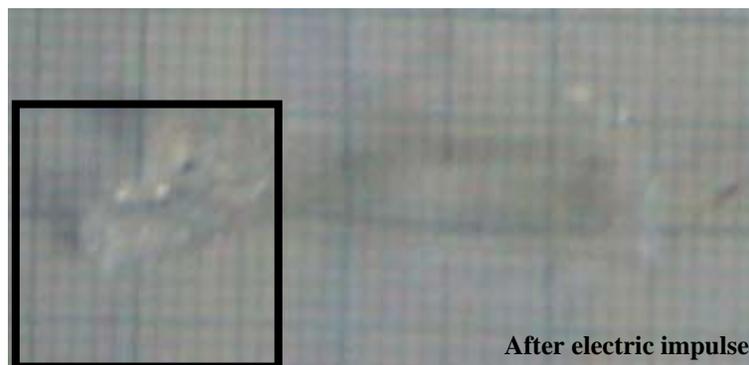


Fig. 3. Damage to EAP material in boxed area.

Plasma treatment was used to improve the interface between the electrode metal and the electroactive material. Plasma is partially or wholly ionized gas with about an equal number of positively and negatively charged particles. Some scientists have named plasma the "fourth state of matter."³¹ While plasma is neither gas nor liquid, the properties of plasma are similar to those of both gases and liquids. Sterilization and improving the adhesion between two surfaces are common applications. Good adhesion requires strong interfacial forces by chemical compatibility and/or chemical bonding. Plasma surface treatment can create chemically active functional groups such as amine, carbonyl, hydroxyl and carboxyl groups, which can greatly improve interfacial adhesion. Plasma is used to improve bonding on substrates such as glass, polymers, ceramics, and metals. Plasma treatment can also enhance performance. Surface crosslinking is

often used to enhance the performance of polymers, particularly in terms of strength and toughness. Plasma treatment can create a higher crosslinking density within the material to depths of a few thousand angstroms.³¹ The resulting increase in hardness and chemical resistance can substantially enhance performance.

Preliminary experiments explored the bonding between these EAP materials with plasma treated metals provided by PPPL. The results were encouraging, with much better bond strengths in the plasma treated metals compared to the untreated control. In 2007, a CRADA was formed between Ras Labs and PPPL to expand upon improving the attachment of the embedded electrodes to the EAP materials in these actuators using plasma treatment and other treatments to non-corrosive metallic leads. For both stainless steel and titanium, oxygen plasma treatment provided the best metal-polymer interfaces for the embedded electrodes.

Most of the theory of EAPs concerns bending. Contraction is not as well understood. A radionuclide experiment was performed to determine, in as close to real time as possible, the chain of events that occurs when electricity is applied to these contractile EAPs, both in terms of water loss and electrolyte flow during contraction (Figure 4). EAP samples were immersed in aqueous NaCl solutions containing known concentrations for both tritium (H-3) and sodium-22 (Na-22) radionuclides and allowed to swell. For the electroactive experiment, the EAP sample was then placed in an unlabeled aqueous NaCl solution and subjected to electric input. Aliquots of the surrounding solution were removed in timed intervals during the electric input. A control experiment was also performed, where no electricity was applied to control EAP sample while aliquots were removed in the same timed intervals. LSC was used to detect tritium levels and GRS was used to detect Na-22 levels.

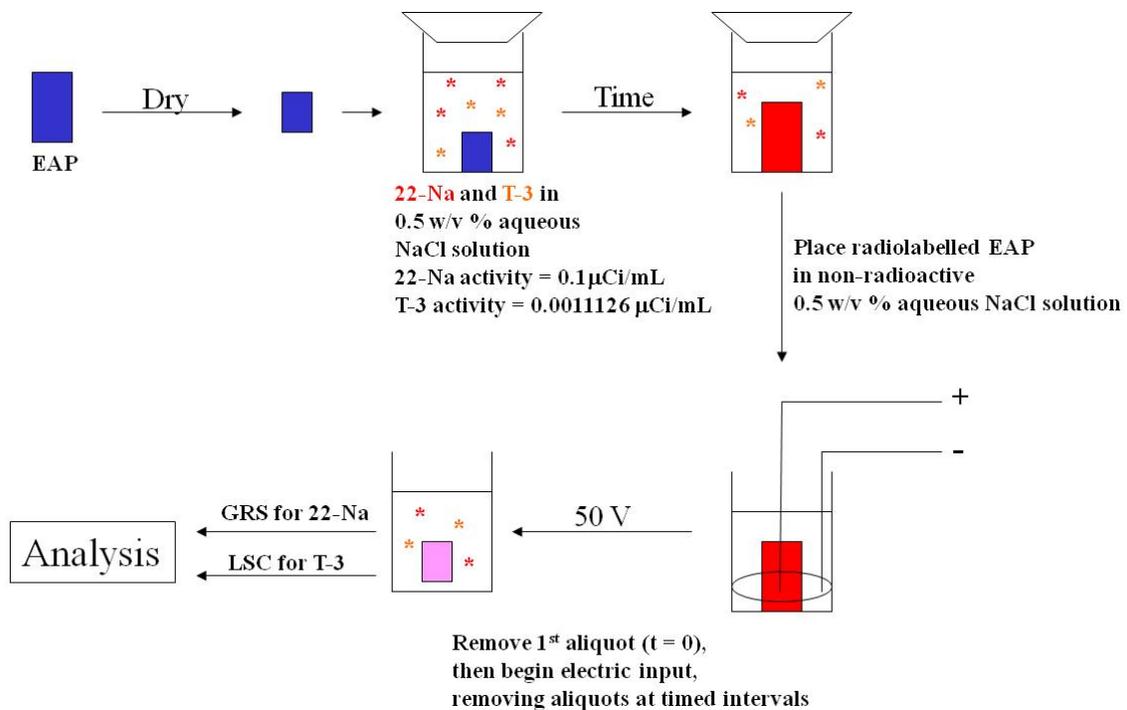


Fig. 4. Radionuclide experiment following EAP contraction.

The results from the radionuclide experiment indicate that during the electric input, Na-22 leaves the contractile EAP very quickly, with significant outflow of the positive sodium ions towards the external negative electrode within 5 seconds. The outflow plateaus around 75 seconds. Based on the control (no electric input), the Na-22 is retained very tightly to the EAP material, while the tritium has some baseline mobility in and out of the EAP. Once the EAP is activated by electricity, the positive sodium ions and water move very quickly and simultaneously out of the EAP material, leading to a very fast, pronounced contraction (Figures 1 and 5). Following water loss in real time shows a similar trend (Figure 6).

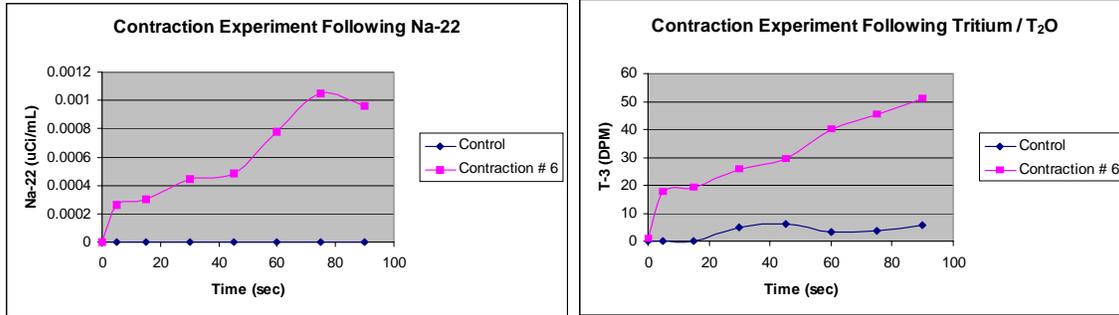


Fig. 5. Contraction experiment following radionuclide-labeled Na-22 and T-3 electrolytic solutions.

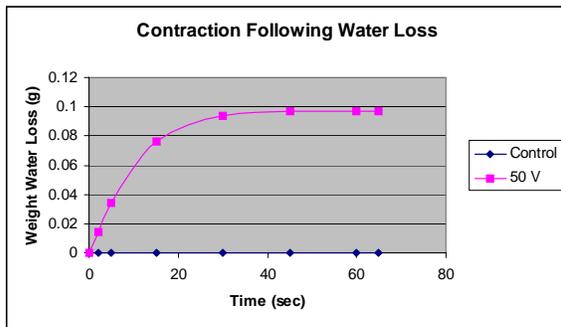


Fig. 6. Contraction experiment following water loss.

After the electric input part of the radionuclide-labeled experiment, the EAPs were placed in fresh unlabeled aqueous sodium solution, allowed to equilibrate for about a week, and then aliquots taken from each equilibration media (Figure 7). GRS found no Na-22 activity in the equilibration media, while tritium levels were fairly high, particularly for the control (Figure 8). Again, indicative that the sodium ions are very tightly bound to the EAPs, unless there is an electric input, while the water molecules are much more mobile.

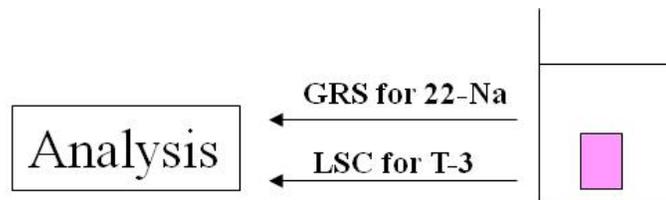


Fig. 7. Radionuclide equilibration part of experiment after contraction.

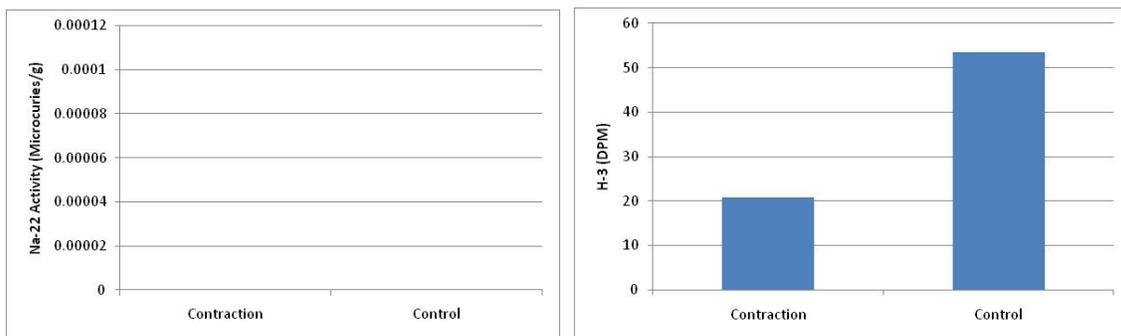


Fig. 8. Equilibration part of experiment following radionuclides Na-22 and T-3.

Finally, GRS was used to analyze the EAPs themselves (the contraction EAP and control EAP), well after the contraction and the equilibration parts of the experiment, to account for the location of all ²²Na, particularly in the control which had no electric input. The control EAP contained 6 times the amount of Na-22 compared to the EAP that had undergone contraction (Figure 9).

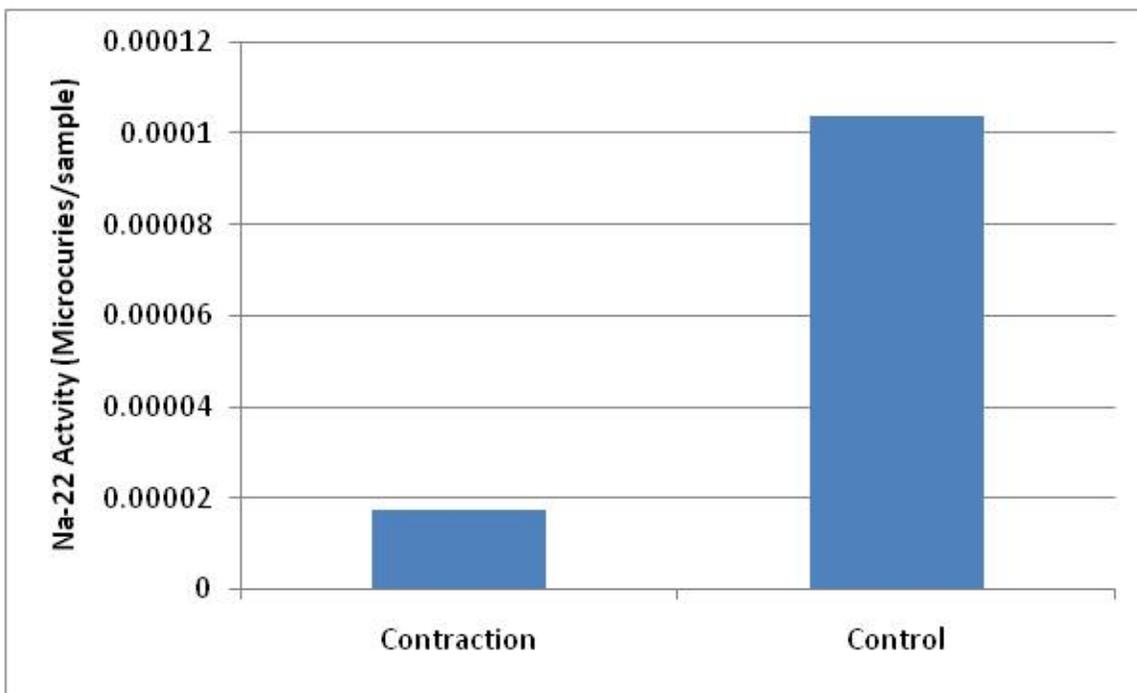


Fig. 9. GRS Analysis of the EAPs themselves following radionuclide Na-22.

Electricity is required in order for sodium ions to flow out of these contractile EAPs. With electric input, sodium ions are attracted to the external negative electrode and repelled by the internal positive electrode. Sodium ions and water simultaneously leave the EAP, causing a rapid collapse and contraction of the EAP. In the control experiment, there was some movement of tritium out of the control EAP, which can be attributed to baseline Brownian motion. Without electric input, there is no movement of Na-22 out of the EAPs, which was clearly demonstrated with the control EAP, which had no electric input. After an extended equilibration in fresh solution after the experiments, there was some tritium detected, but no Na-22 detected in the surrounding equilibration media. The EAPs themselves were then analyzed using GRS. The control EAP contained substantially higher Na-22 than the EAP that had undergone contraction. Even when submerged in an aqueous solution, the sodium ions remain very tightly associated with the ionic EAPs – unless subjected to an electric input that competes and disrupts this ionic bond with the EAP. Once the sodium ions begin migrating to the negative electrode, the water shells surrounding the electrolytes also migrate out of the EAP, resulting in rapid contraction.

4. CONCLUSIONS

Cross-linked ion-containing polymer networks have produced electroactive smart materials with good strength, durability, and electroactivity, with the ability of some of these EAP materials to contract more than 80 % within 1 minute at 50 V, and then return to their original size and shape when the electric input is stopped. Stronger EAPs were developed that contract to about 50 % from their original weight within 1 minute at 50 V, and when the polarity was reversed, expand back to their original weight within 1 minute at 50 V. The contraction-expansion cycles can be cycled repeatedly. Research and development to significantly improve the polymer-electrode interface has been performed, with significantly better adhesion between the electrode(s) and the EAP, particularly for stainless steel and titanium treated with oxygen plasma. The goal is for both the electroactive smart material and the embedded electrodes to move

as a unit, analogous to our muscles, nerves, and tendons moving together. In this analogy, the EAP material serves as the artificial muscle, and the plasma treated electrode(s) serves as both a tendon, connecting the EAP to a lever, and as a nerve, delivering electric stimulus to the EAP. Experiments using radionuclide-labeled electrolyte (Na-22) and tritium labeled water were performed to definitively determine the chain of events that occur when these EAPs contract. With electric input, sodium ions are attracted to the external negative electrode. Sodium ions and water simultaneously leave the EAP, causing a rapid collapse and contraction of the EAP. Without electric input, the sodium ions remain very tightly bound to the ionic EAPs. These recent developments, and fundamentally, the thorough understanding of this contraction phenomenon, are important in the field of electroactivity because of the ability of contraction and contraction-expansion to produce biomimetic life-like motion.

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