CHAPTER IV

A study of electromechanical properties of gelatins as an actuator or an artificial muscle

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Abstract

Gelatin is a protein produced by the partial hydrolysis of a collagen extracted from bones, connective tissues, organs, and some intestines of animals. Gelatin has been widely used in the pharmaceutical and medical fields as sealants for vascular, a carrier for drug delivery, wound dressings, and an artificial muscle. In our work, gelatin films were prepared by the film casting method using water as the solvent. The electromechanical properties, thermal properties, and the degree of swelling were measured as the function of gelatin crosslinking ratio or the gel strength, temperature, frequency, and electric field strength. The high, medium, low, and the 3% crosslinked high gel strength gelatin films possess the storage modulus sensitivity values of 2.30, 2.16, 1.26 and 0.49 respectively.

Keywords: Gelatin; gel strength; electromechanical properties

1. Introduction

The exchange of electrical energy for mechanical energy has been of scientific and technological interests for many decades. Electromechanical energy conversion has been applied in many applications such as muscle/insect-like actuators, robotics, and etc [1]. The development of electroactive materials for artificial muscle or actuator is very attractive because it has many advantages. First, the electroactive materials resemble natural living tissues more than any other class of synthetic biomaterials because to their high water contents, soft consistency, and their activation mode. Secondly it is biocompatible and not biodegradable. Third,

their physical and chemical properties vary with composition and can be tailored as desired. Fourth, they can take various shapes and they are a low-cost material.

Gelatin is a protein biopolymer derived from the partial hydrolysis of native collagens, which are the most abundant structural proteins found in the animal body: skin, tendon, cartilage, and bone [2]. Due to a wealth of merits such as biological origin, non-immunogenicity, biodegradability, biocompatibility, and commercial availability at relatively low cost, gelatin has been widely used in the pharmaceutical and medical fields as sealants for vascular prostheses, carrier for drug delivery, wound dressings, and an artificial muscle [3]. On the other hand, gelatin exhibits poor mechanical properties which limit its possible applications as a biomaterial. The improvement of the mechanical properties of drawn gelatin has been related to the renaturation level of the protein, as evaluated through the differential scanning calorimetry [4]. In the literature, several physical and chemical methods have been reported for crosslinking collagenous materials. Physical methods include the dehydrothermal treatment and the UV irradiation [5,6]; however, they are generally less efficient. Many chemicals such as formaldehyde, glutaraldehyde, carbodiimide, and dextran dialdehyde, have been used to chemically modify gelatin for biomedical applications. Amongst them, glutaraldehyde (GTA) is by far the most widely used chemical, due to its high efficiency in stabilizing collagenous materials [7]. GTA based crosslinking of collagenous materials significantly reduces biodegradation, making the materials biocompatible and nonthrombogenic while preserving biological integrity, strength and flexibility. GTA is also easily available, inexpensive, and capable of allowing the crosslinking in a relatively short time period.

In our work, we are interested in gelatin as a candidate for artificial muscle or actuator. The electromechanical properties, thermal properties, and degree of swelling were measured and will be reported as functions of gelatin strength, crosslinking ratio, temperature, frequency, and electric field strength.

2. Experimental

2.1 Materials

Gelatin powder (high, medium, and low gel strengths) (AR grade, Fluka), glutaraldehyde (50% GTA solution) (AR grade, Sigma-Aldrich) were used as starting materials for fabricating gelatin films.

2.2 Preparation of gelatin films

Glutaraldehyde–gelatin crosslinked films (GTA–Ge) were prepared by adding the appropriate volume of GTA solution to the gelatin solution 10 vol.%), with GTA concentrations varying from 0.5 to 7 vol%. Non-crosslinked gelatin films (Ge) were prepared from an aqueous gelatin solution (10%, v/v) at 50 ◦C and under continuous stirring for 40 min. The GTA-Ge and Ge solutions were poured into plastic petri dishes (10 cm of diameter). Crosslinked films were obtained after allowing a water evaporation at room temperature for a period of 4 days. Pristine gelatin films (Ge) were prepared in the similar way but without adding the crosslinking agent.

2.3 Characterization and testing of gelatin samples

2.3.1 Crosslinking density determination

In order to estimate the network crosslinking density, the number-average molecular weight of chain segments between two crosslinking points, M_c, was calculated from equilibrium water uptake experiments performed at 20 ˚C, according to the Flory–Renher equation [8]:

$$M_c = \frac{\rho V_1 \left(\phi_g^{1/3} - 2\phi_g / f\right)}{\chi \phi_g^{\,2} + \ln\left(1 - \phi_g\right) + \phi_g} \qquad (1)$$

where ρ is the density of the dry gelatin determined by picnometry, V_1 is the molar volume of the solvent, χ is the polymer–solvent interaction parameter whose value

was taken from the literature [9] (χ = 0.49 ± 0.05), and φ_g is the volume fraction of the swollen gelatin. The gelatin volume fraction in the swollen samples (φ_g) was estimated assuming the following relation:

$$\phi_g = \frac{W_0 \rho_w}{W\rho_g - W_0(\rho - \rho_w)} \tag{2}$$

where W_0 is the initial weight of the sample, W is the weight of the swollen sample, ρ_w is the density of the water at room temperature, and ρ is the density of the dry and uncrosslinked gelatin film.

2.3.2 Thermogravimetric analysis (TGA)

A thermal gravimetric analyzer (DuPont, model TGA 2950) was used to determine the amount of moisture content and the decomposition temperatures with the temperature scan from 30 to 600 °C with a heating rate of 5 °C/min, for the crosslinked films with % volumes of glutaraldehyde of 0.5,1, 3, 5, and 7, and the non-crosslinked gelatin films. The samples were weighted in varying from 5 to 10 mg and loaded into platinum pans, and were heated under a nitrogen gas flow.

2.3.2 Electromechanical properties

The electrorheological properties of the crosslinked and uncrosslink gelatins were investigated in terms of electric field strength. A melt rheometer (Rheometric Scientific, ARES) was fitted with a copper parallel plates fixture (diameter of 25 mm). A DC voltage was applied with a DC power supply (Instek, GFG8216A), which can deliver electric field strength to 1 kV/mm. A digital multimeter was used to monitor the voltage input. In these experiments, the oscillatory shear strain was applied and the dynamic moduli (G' and G") were measured as functions of frequency and electric field strength. Strain sweep tests were first carried out to determine the suitable strains to measure G' and G" in the linear viscoelastic regime. The appropriate strain was determined to be 0.2% for gelatin film samples. For the 3% crosslinked high gel strength gelatin film sample, the strain of 0.14% was used. The frequency sweep tests were carried out to measure G' and G" of each sample as

functions of frequency. The deformation frequency was varied from 0.1 to 100 rad/s. Prior to each measurement, the non-crosslinked gelatin and the 3% high gel strength gelatin film samples were presheared at a low frequency (0.04 rad/s) under an electric field for 15 min to ensure the formation of equilibrium polarization before the G' and *G"* measurements. Experiments were carried out at the temperature of 27 °C and repeated at least two or three times. The effect of temperature was studied at various temperatures between 27 and 107 °C for the non-crosslinked gelatin film sample. The temporal response experiments were carried out at 1 kV/mm for the non-crosslinked gelatin and the 3% crosslinking high gel strength gelatin film samples.

3. Results and Discussion

3.1 Determination degree of swelling, weight loss of gelatin film and molecular weight between crosslinks

Crosslinked and un-crosslinked gelatin films were prepared in the same conditions. Un- crosslinked and crosslinked gelatin films were kept in a desiccator at room temperature (27°C) for a period of 2 days before testing to prevent any further property changes during the experiments. Glutaraldehyde (GTA) is a fast-acting crosslinker for collageneous materials, easily available and inexpensive. The reaction of gelatin with different amounts of GTA was carried out at 50 °C in basic conditions [10]. All crosslinked films were stiffer than pure gelatin films and they changed in color to yellowish films. The GTA crosslinking induces a significant reduction in swelling. Swelling measurements at longer times were hindered by the solubility of the film which began to dissolve in solution. The degree of swelling and the percentage of weight loss are shown in Fig 1. As can be seen from Fig 1, the degree of swelling and the percentage of weight loss decrease drastically with increasing GTA content, due to the formation of a denser network. Gelatin films with percentages of crosslinking agent higher than 3 wt% do not possess any noticeable differences in the degree of swelling and the percentage of weight loss. The calculated average molecular weights between two crosslinking points, M_c, in terms of GTA concentration are shown in Table 1.

The thermal properties were obtained from the thermogravimetric analysis and the differential thermal analysis (TGA-DTA). The TGA data show a glass transition temperature of the gelatin samples with different bloom index at various percentages of crosslinking (0.5, 1, 3, 5, 7 %v/v). There were three transitions for the gelatin namely: the first transition (45-100°C) refers to the loss of water; the second transition (197-220°C) refers to the glass transition temperature of gelatin, and the third transition (280-320°C) refers to the degradation of gelatin. The TGA themograms of the gelatin and the crosslinked gelatin show that the decomposition temperatures are not significantly different; with 7%v/v GTA-Ge has the highest percentage of a weight residue.

3.2 Time dependence of the electrorheological response

We first show the temporal responses of high, medium, low and 3% crosslinked high gel strength gelatin films, subject to switching on and off under applied electric field strength of E=1000 V/mm. The temporal characteristic of each sample was recorded in the linear viscoelastic regime at a strain of 0.14% and at frequency of 100 rad/s. As shown in Fig. 2, the storage moduli of the gelatin films show changes in G' values at electric field strengths of 1000 V/mm during a time sweep test, in which an electric field was turned on and off alternately. The storage modulus of high, medium, low, and 3% crosslinked high gel strength gelatin increase from G'= 2,120,000 Pa to 4,570,000 Pa (1.15) at 2200 s, G= 835,000 Pa to 1,485,000 (0.79) at 5000 s, G' = 1,260,000 Pa to 2,080,000 (0.65) at 2500 s, and G'= 8,060,000 Pa to 10,900,000 Pa (0.35) at 2000 s, respectively. Fig. 2(a)-(d) shows the temporal response of gelatin film at electric field 1000 V/mm the storage modulus increases. With the electric field switched off, G' does not recover its original value, possibly due to the interaction between the residue permanent dipoles in gelatin molecules [11].

3.3 Effect of gelatin films under electric field strength

The effect of electric field strength on the rheological properties of high, medium, low and 3% crosslinked high gel strength gelatin films was investigated in the range from 0 to 1 kV/mm. Fig. 3 shows the storage modulus sensitivity ($\Delta G'/G_\circ$)

and the storage modulus response ($\Delta G'$) of the high, the medium, the low and the 3% crosslinked high gel strength gelatin films vs. electric field strength, at frequency 100 rad/s, strain 0.14%, and at 27 °C. The increases in $\Delta G'/G_\circ$ and $\Delta G'$ with electric field are nonlinear within the range of 0.1–1 kV/mm. The storage modulus response values, $\Delta G'$, of these systems at electric field strength of 1 kV/mm are 3030000, 1928000, 369000, and 1170000 Pa for the high, the medium, the low, and the 3% crosslinked high gel strength gelatin films, respectively. The storage modulus sensitivity values, defined as $\Delta G'/G_\circ$ of these samples (high, medium, low, and 3% crosslinked high gel strength gelatin films) at electric field strength of 1 kV/mm are 2.30, 2.16, 1.26, and 0.49, respectively, as tabulated in Table 2.

When the electrical field is applied, gelatin molecules induced dipole moments are generated, leading to intermolecular interactions. These intermolecular interactions induce the loss chain free movements and higher chain rigidity, as indicated by higher G' values [12,13]. The electric field evidently enhances the storage modulus of our samples. But the improvement of 3% crosslinked gelatin storage modulus is less than those of the uncrosslinked gelatin samples, since sample rigidity is relatively high without electric field applied. A higher electric field strength is expected to induce a higher dipole moment and to cause particle chains to pull themselves together in a tighter formation due to the greater electrostatic force as evidenced by the dramatic increases in both G′ with electric field strength [14]. And comparisons of response and sensitivity of other systems are shown in Table 3. Table 3 shows several characteristics of storage modulus sensitivity of electroactive polymers and dielectric elastomers. For comparison stating sensitivities of these materials can be seen between Table2 and Table3. In same electric field strength (1kV/mm), Styrene-isoprene-styrene triblock (D1114P) is the best in its types, the storage modulus sensitivity is 0.122, however when add particles in Styrene-isoprene-styrene triblock and Silicone elastomer, the storage moulus sensitivity increased to 0.256 and 0.250 respectivly. Table2 demonstrates, good response was achieved in gelatin system, so the storage modulus sensitivity is higher than dielectric elastomer system in same electric filed strength. In addition dielectric elastomers can operate at relatively high voltage (0.1-4 kV/mm), depending on material thickness and properties) [15].

3.4 Effect of operating temperature

Finally, the storage modulus sensitivity and the storage modulus differential response of the gelatin samples are found to decrease with increasing temperature up to 380 K. This is a general plastic-like behavior. In order to exclude the effect of gelatin samples, $\Delta G' = G'(E) - G'(E=0)$ and $\Delta G'/G_\circ$ are plotted versus temperature as shown in Fig. 4. Evidently, $\Delta G'$ initially decreases with temperature up to 306 K: at E=1 kV/mm, for high, medium, low gel strength gelatin films, $\Delta G'$ are 3024000, 1930000, and 368000 Pa at 300K; $\Delta G'$ are 2483000, 1672000, and 363000 Pa at 306° K respectively. $\Delta G'$ dramatically decreases with increasing temperature up to 380°K. As can be seen in 3% crosslinked high gel strength, its $\Delta G'$ slightly decreases between 300°K and 380°K.

4. Conclusion

In our work, the electromechanical properties of gelatin films were investigated as the function of electric field strength and operating temperature on the storage moduli under oscillatory shear mode. The storage modulus (G') increases with increasing gel strength of the gelatin samples, as the applied electric field strength is increased to 1 kV/mm. The temperature dependence of $\Delta G'$ and $\Delta G'/G°$ shows a maximum at $T \approx 300$ °K, so the $\Delta G'$ and $\Delta G'/G_0$ of gelatin samples are found to decrease with increasing temperature up. At the temperature above 300 K, they indicate the plastic-like behavior. For un-crosslinked gelatin films, the storage modulus response and the storage modulus sensitivity are higher than crosslinked gelatin films.

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6. References

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Figure caption

[1] % swelling and % weight loss of crosslinked gelatins of various crosslinking ratios, 27 °C (# samples = 3): a) High gel strength of gelatin; b) Medium gel strength of gelatin; c) Low gel strength of gelatin.

[2] Storage modulus (G') vs times of gelatin films of various gel strength (100 rad/s, 1 kv/mm): a) High gel strength of gelatin; b) Medium gel strength of gelatin; c) Low gel strength of gelatin; d) 3% crosslinked high gel strength of gelatin.

[3] Storage modulus differential and sensitivity vs Electric field strength of gelatin films at various gel strength (100rad/s, 0.14%strain, 27°C).

[4] Storage modulus differential and sensitivity vs Temperature of gelatin films of various gel strength (E = 1kV/mm, 100rad/s, 0.14% strain).

List of table

[1] Molecular weight between crosslinker of gelatins of various crosslinking ratio, 27°C. (# samples = 3)

[2] Compare storage modulus sensitivity of gelatin films at various gel strengths

[3] Compare storage modulus sensitivity of electroactive and dielectric elastomer materials technology.

Table 1. Molecular weight between crosslinker of gelatins of various crosslinking ratio, 27°C. (# samples = 3)

Sample	%crosslinking agent (v/v)	Crosslink ratio	Molecular weight between crosslink (g/mol)	Crosslink density ($mol/cm^3 \times 10^4$)
High gel strength gelatin	0.5	4.69×10^{-5}	13,700 ± 687	0.73 ± 0.04
	1	9.39×10^{-5}	12,800 ± 344	0.85 ± 0.073
	3	2.81×10^{-4}	1,920 ± 296	6.09 ± 0.085
	5	4.69×10^{-4}	690 ± 75	16.68 ± 0.33
	7	6.57×10^{-4}	320 ± 16	50.58 ± 2.03
Medium gel strength gelatin	0.5	4.69×10^{-5}	12,900 ± 647	0.77 ± 0.04
	1	9.39×10^{-5}	12,400 ± 624	0.80 ± 0.04
	3	2.81×10^{-4}	1,520 ± 76	6.56 ± 0.33
	5	4.69×10^{-4}	570 ± 29	17.45 ± 0.87
	7	6.57×10^{-4}	200 ± 10	49.26 ± 2.46
Low gel strength gelatin	0.5	4.69×10^{-5}	13,900 ± 766	0.80 ± 0.04
	1	9.39×10^{-5}	11,400 ± 542	0.75 ± 0.054
	3	2.81×10^{-4}	1,440 ± 66	5.06 ± 0.20
	5	4.69×10^{-4}	670 ± 40	16.55 ± 0.27
	7	6.57×10^{-4}	300 ± 15	49.26 ± 1.64

Table 2. Compare storage modulus sensitivity of gelatin films at various gel strength

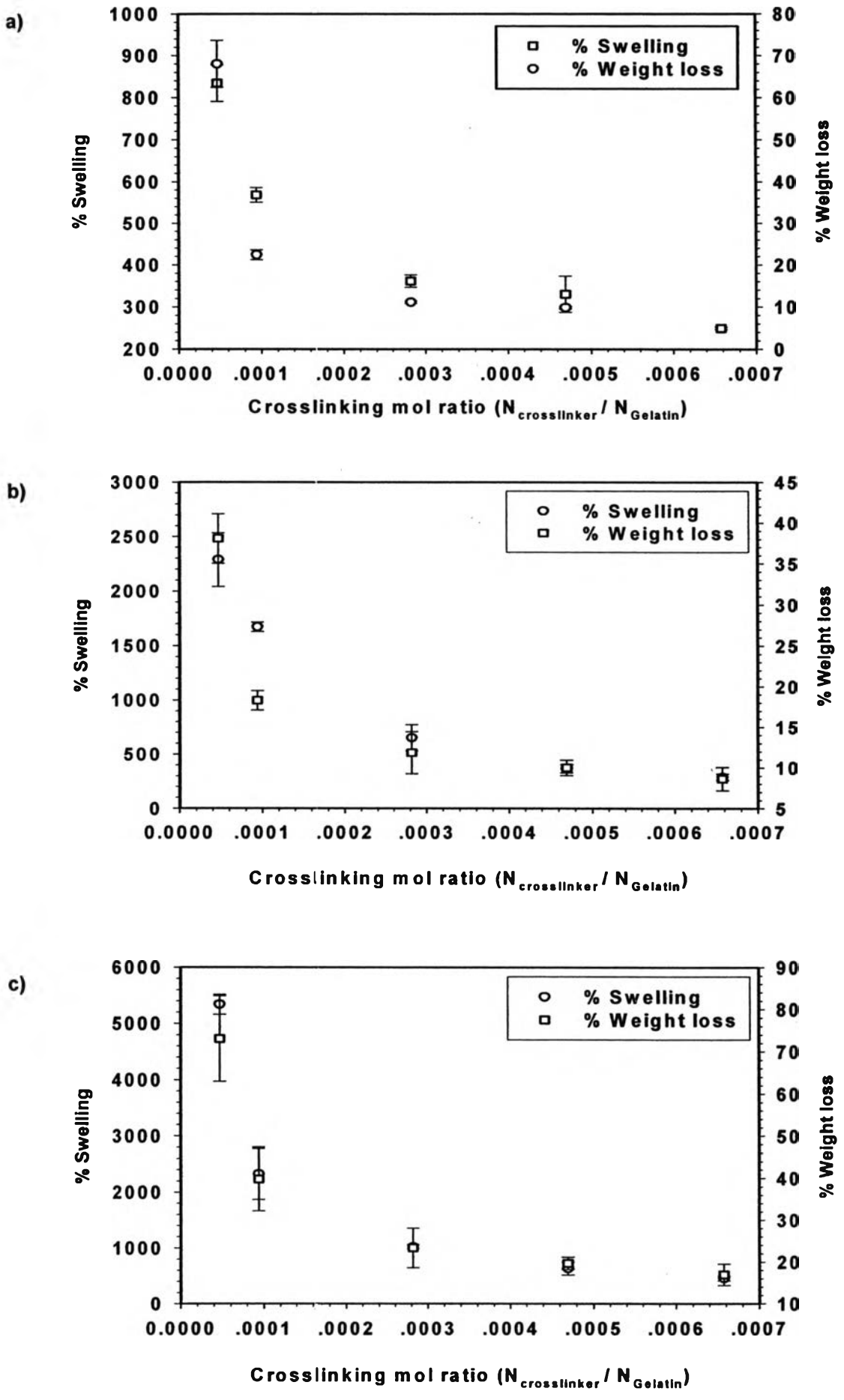
Material	Electric field (kv/mm)	Frequecy (rad/s)	Temperature (C°)	Storage modulus (G') Pa	Initial storage modulus (G°) Pa	Storage modulus sensitivity (ΔG'/G°) Pa
High gel strength gelatin	1000	100	27	4340000	1310000	2.30
Medium gel strength gelatin	1000	100		2820000	892000	2.16
Low gel strength gelatin	1000	100		292000	661000	1.26
3% crosslinked High gel strength gekatin	1000	100		3580000	2410000	0.49

Table 3. Compare storage modulus sensitivity of electroactive and dielectric elastomer materials technology

Materials	Electric field (kv/mm)	Frequecy (rad/s)	Temperature (C°)	Storage modulus sensitivity (ΔG'/G·) Pa	Reference
Acrylic elastomer 70	2000	100	27	0.439	Kunanuruksapong [15]
Acrylic elastomer 71				0.586	
Acrylic elastomer 72				0.148	
Styrene-acrylic copolymers				1.195	
Styrene-isoprene-styrene triblock D1112P				0.746	
Acrylic elastomer 71 + PPP 10%(v/v)		1		0.306	
Acrylic elastomer 71 + PPP 30%(v/v)				0.971	
Styrene-isoprene-styrene triblock D1114P	1000	1		0.122	Thongsek [16]
Styrene-isoprene-styrene triblock D1164P				0.102	
Styrene-isoprene-styrene triblock D1162P				0.050	
D114P + PDPA 5%(v/v)				0.040	
D114P + PDPA 10%(v/v)				0.256	
D114P + PDPA 30%(v/v)				0.095	
AR71/lead zirconate titanate Pb(Zr0.5Ti0.5)O3 (0.000019%v/v)	2000	1		0.149	Tangboriboon [17]
AR71/lead zirconate titanate Pb(Zr0.5Ti0.5)O3 (0.038%v/v)				0.587	
poly (dimethyl siloxane) (PDMS)	2000	100		0.104	Piyanoot [18]
poly (dimethyl siloxane) (PDMS) + PANi 20% (v/v)				0.25	
poly (dimethyl siloxane) (PDMS) + PANi 2% (v/v)				0.111	
PDMS_5%PEDOT/PSS/EG	2000	100		0.077	Wijittra [19]
PDMS_15%PEDOT/PSS/EG				0.333	

Materials	Electric field (v/mm)	Frequecy (rad/s)	Temperature (C°)	Storage modulus sensitivity (ΔG'/G•) Pa	Reference
Crosslinked Polyisoprene 3% + Polythiopene 5% (v/v)	2000	100	27	0.523	Toempong (4)
Crosslinked Polyisoprene 3% + Polythiopene 10% (v/v)				0.33	
Crosslinked Polyisoprene 3% + Polythiopene 30% (v/v)				0.435	
Silicone gel	5000	60		not response	Tahr' Shiga [21,22]
Silicone gel + PMACO 46%	1000			0.25	
Silicone gel + PMACO 46%	2000			0.75	
Silicone gel + PMACO 46%	3000			2	
Silicone gel + poly(p-phenylenes) 10%	1000	300		0.333	
Silicone gel + poly(p-phenylenes) 10%	3000	300		1.133	
Silicone gel + poly(p-phenylenes) 10%	5000	300		1.666	
poly(3-hexylthiophene) doped iodine (amorphous)	8.7	–		0.28	

figure 1


a)
% Swelling
% Weight loss
% Swelling
% Weight loss
Crosslinking mol ratio ($N_{crosslinker}$ / $N_{Gelatin}$)
b)
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% Weight loss
Crosslinking mol ratio ($N_{crosslinker}$ / $N_{Gelatin}$)
c)
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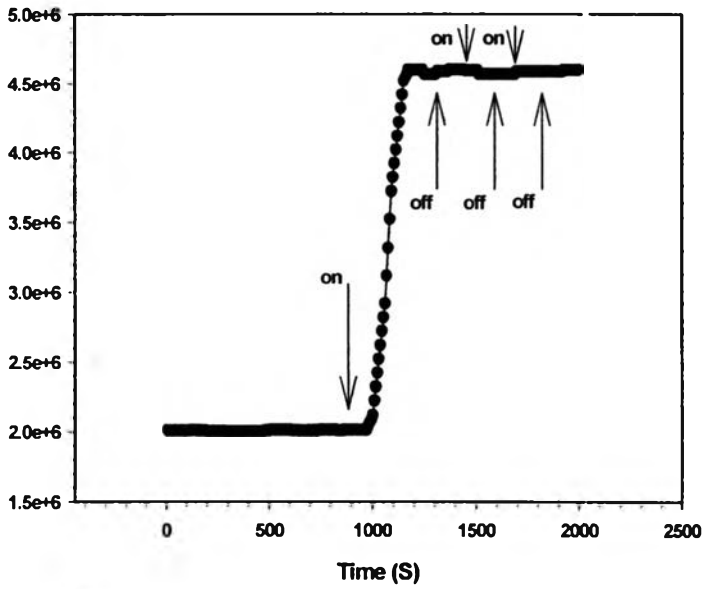
Crosslinking mol ratio ($N_{crosslinker}$ / $N_{Gelatin}$)

Figure 2a-d

a)



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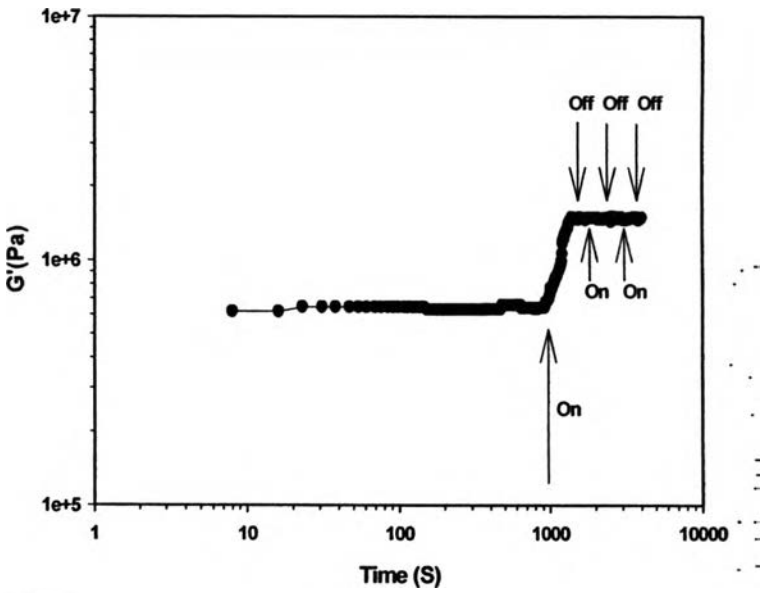

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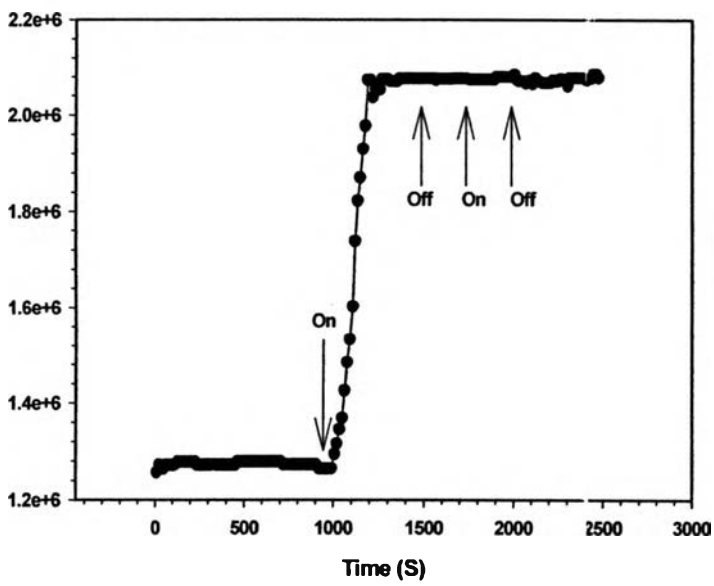

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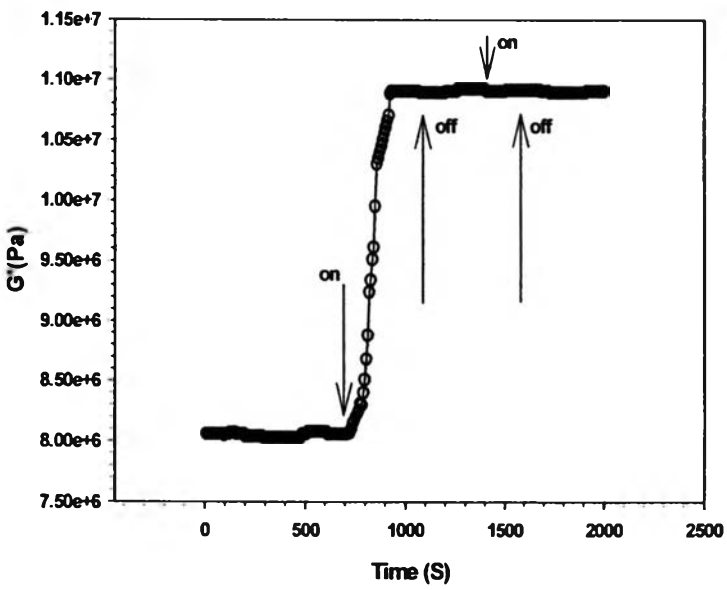
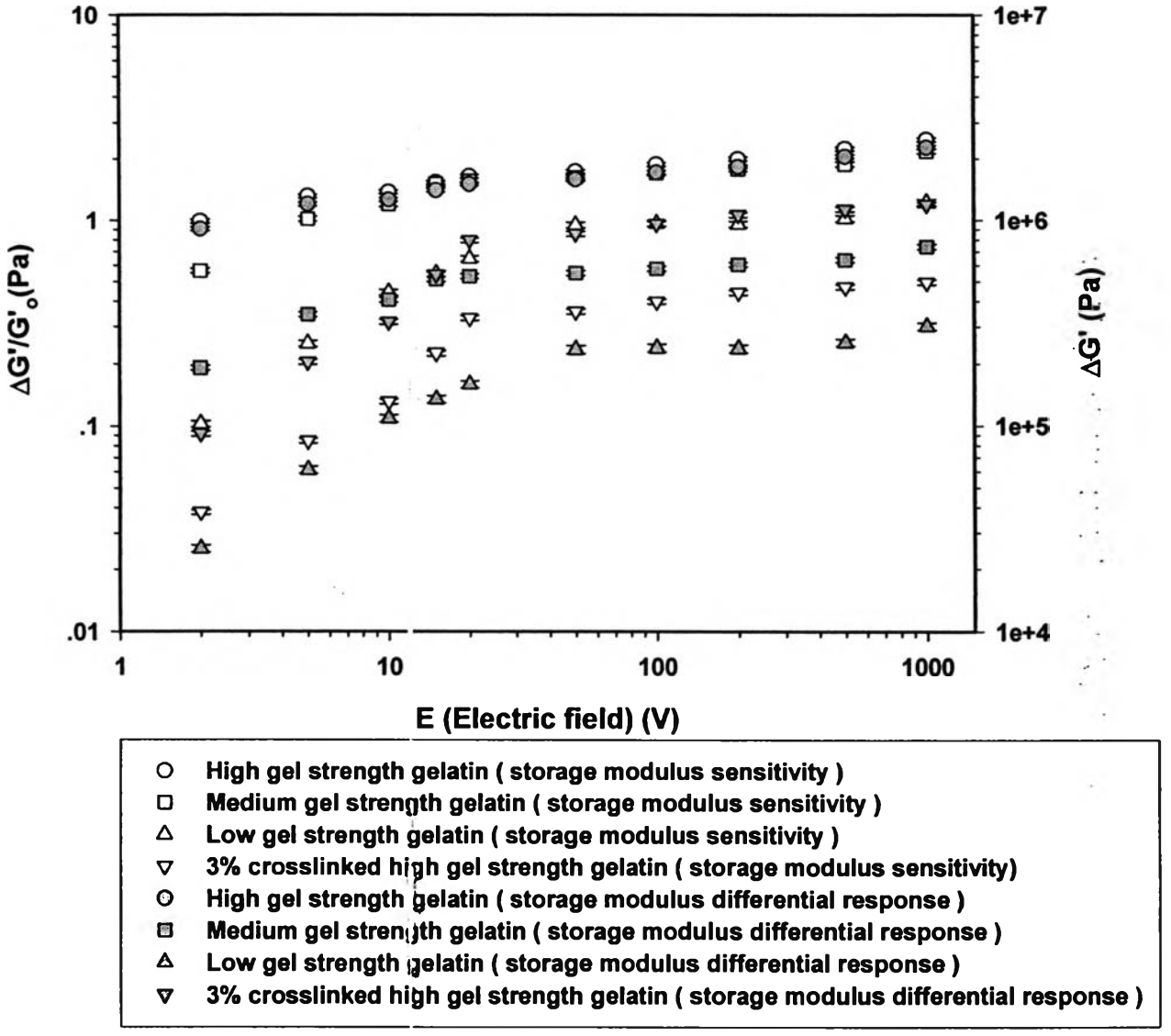


Figure 3


10
1
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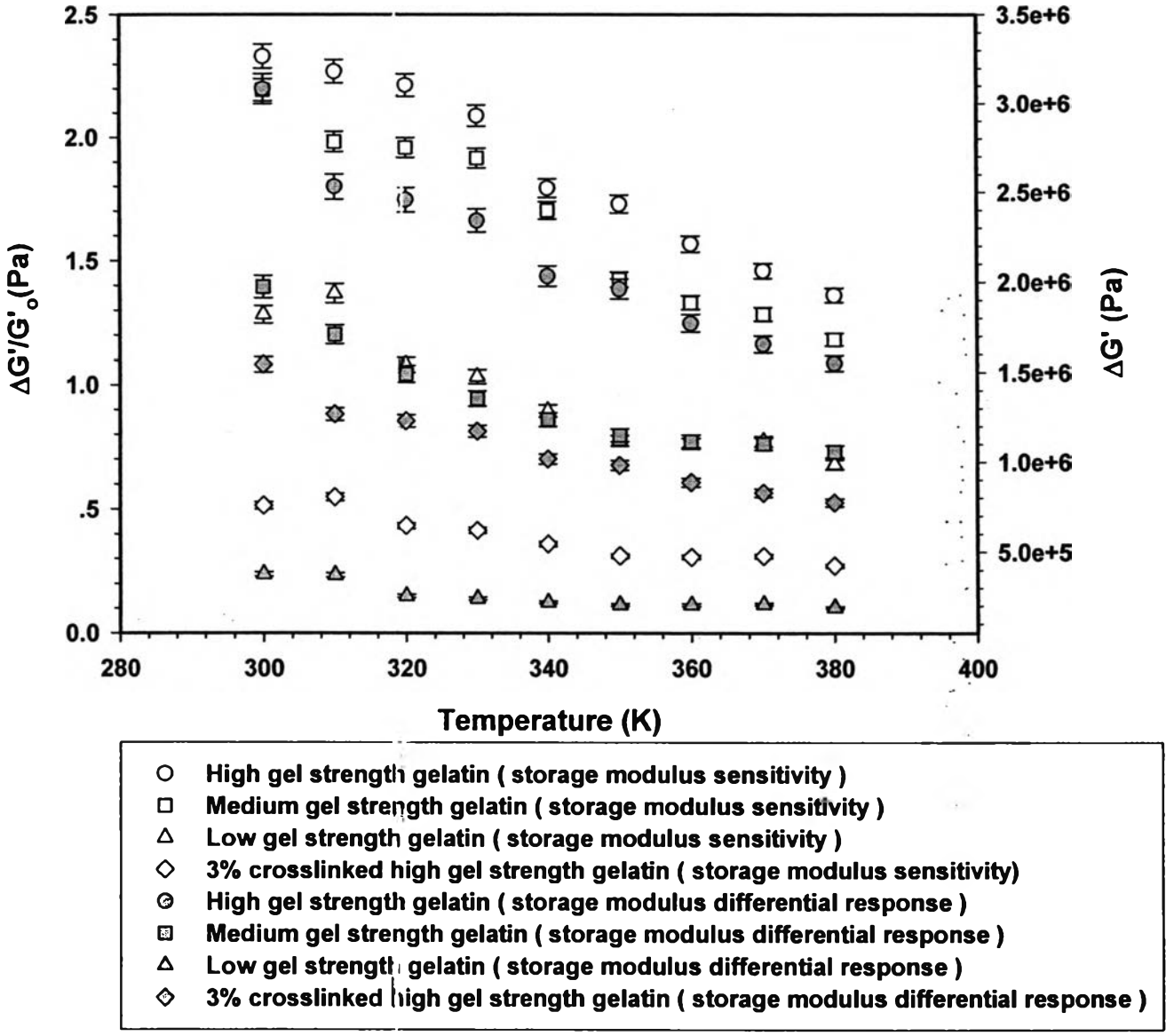
1e+6
1e+5
1e+4
1
10
100
1000
ΔG'/G'o (Pa)
ΔG' (Pa)
E (Electric field) (V)
High gel strength gelatin (storage modulus sensitivity)
Medium gel strength gelatin (storage modulus sensitivity)
Low gel strength gelatin (storage modulus sensitivity)
3% crosslinked high gel strength gelatin (storage modulus sensitivity)
High gel strength gelatin (storage modulus differential response)
Medium gel strength gelatin (storage modulus differential response)
Low gel strength gelatin (storage modulus differential response)
3% crosslinked high gel strength gelatin (storage modulus differential response)

Figure 4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1.5
1.0
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ΔG'/G'o(Pa)
3.5e+6
3.0e+6
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2.0e+6
1.5e+6
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ΔG' (Pa)
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Temperature (K)
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Medium gel strength gelatin (storage modulus sensitivity)
Low gel strength gelatin (storage modulus sensitivity)
3% crosslinked high gel strength gelatin (storage modulus sensitivity)
High gel strength gelatin (storage modulus differential response)
Medium gel strength gelatin (storage modulus differential response)
Low gel strength gelatin (storage modulus differential response)
3% crosslinked high gel strength gelatin (storage modulus differential response)