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## COMPRESSIBLE AND BENDABLE HIGHLY FLEXIBLE DOUBLE NETWORK GEL POLYMER ELECTROLYTES FOR SUPERCAPASITORS

**Abstract:** In this article, a unique self-healing, flexible double network gel polymer electrolyte derived from raw gellan gum and polyacrylamide is prepared and used as gel electrolyte in a supercapacitor. Gellan gum and polyacrylamide that have been cross-linked using Na<sup>+</sup> ions have good mechanical and self-healing characteristics. Tensile stress-strain curves before and after self-healing mechanical properties at various times - 0.3, 2, 4, 8, 16, 24 hours - and temperatures - 0, 20, 40, 60, 80, 90°C - indicated that gel polymer electrolytes have good self-healing performance and tensile characteristics. As a result of such polymer electrolyte features, supercapacitors have a high specific capacitance.

**Key words:** flexibility, polymer gel, supercapacitor, blendable, compressible, double network polymer, gellan gum, polyacrylamide, tensile stress, ionic conductivity.

**Language:** English

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### Introduction

Supercapacitors (SC) have attracted a lot of research interests as one of the best next-generation energy storage devices because of its have many advantages such as rapid charging/discharging ability, high safety, long cycle life, superior power density, and good eco-friendliness. SC devices have been developed by sandwiching the electrolyte between two electrodes [1,2]. SC devices have been developed the storing electrical charge at the interface between electrode materials and electrolytes. Among them, the electrolyte is a crucial part and one of the key components to determine electrochemical stable potential window, rate capability, and life cycle of SCs [3-6]. In the past decades, aqueous, organic, ionic liquids and redox-active gels types have been widely used for SCs. Liquid electrolytes face several

drawbacks that can be encountered such as electrolyte leakage, corrosion, or packaging difficulties [7].

Gel polymer electrolyte (GPE) as a kind of important flexible electrolyte has attention attracted increasing because of its minimum leakage compared to liquid electrolyte and much higher ionic conductivity compared to solid polymer electrolyte [4,8]. Besides, GPEs have many advantages such as ease of fabrication, the biodegradable, natural, non-toxic, non-corrosive, light-weight, variable geometry shape, and which may bring new design chances for energy storage devices in the future flexible SCs [9].

Common polymers, such as polyethylene oxide (PEO), polypropylene oxide (PPO), polyacrylonitrile (PAN), and polyvinylidene fluoride (PVDF), polyvinyl alcohol (PVA), and polyacrylamide (PAM) could be used as the matrix of GPE [10]. Among them, PAM is a promising polymer matrix for GPE which

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has many other advantages amorphous structure, higher ionic conductivity than PVA, including their low cost, reasonable chemical stability, and excellent film-forming ability, and so on. Although this polymer matrix has shown satisfactory performances, that is still mechanical properties not good. Furthermore, the long-time use of the devices under various bending, twisting, vibration, and deformation conditions may cause the SC to be irreversible damaged or accidentally breaking. All of these failures would severely limit the reliability and lifetime of SC, resulting in the maybe breakdown of the whole electronic devices. So, in the last year researchers also have been receiving attracted attention and mainly focused on the fabrication of self-healing and smart devices [11-13]. Self-healing materials, which can spontaneously repair internal or external damages, have been developed over the past decade years. This kind of ability gives consumers to use SCs under different conditions for a long time. Nevertheless, the application of GPEs in certain areas is still hindered because of their weak mechanical properties and there are few reports to improve them. However, several hydrogels method with excellent mechanical performance has been successfully developed. For example, slide-ring [14],

interpenetrating network [15], macromonomer hydrogel [16], nanocomposite [17], hydrophobically association [18], and double network (DN) structure [19-21]. Among them, DN structure soft and highly extensible hydrogels with a relatively homogeneous network structure are developed or by combining a rigid and brittle network with a soft and ductile network [21]. It is well demonstrated DN hydrogel can efficiently disperse the locally applied stress and dissipate the energy through the combination of two networks, thus enhancing the hydrogel's mechanical strength. So, DN hydrogels have captured the increasing interest and we focused to improve the mechanical properties of GPE by this method.

In the last decade, the possibility of using many natural materials as hydrogels are being investigated, such as carrageenan [22], xanthan gum [23], chitin [24], chitosan [25], guar gum [26], and gellan gum (GG) [27-29]. GG (Figure 1) is a natural anionic polysaccharide and has many advantages, such as hydrophilic, biocompatible, biodegradable, non-toxic, inexpensive, good gel-forming, thermal stable, and so on. The most important character of GG can cross-link through the ionic association between  $K^+$ ,  $Ca^{2+}$ ,  $Mg^{2+}$ , and  $Na^+$  ions at low temperatures [30].

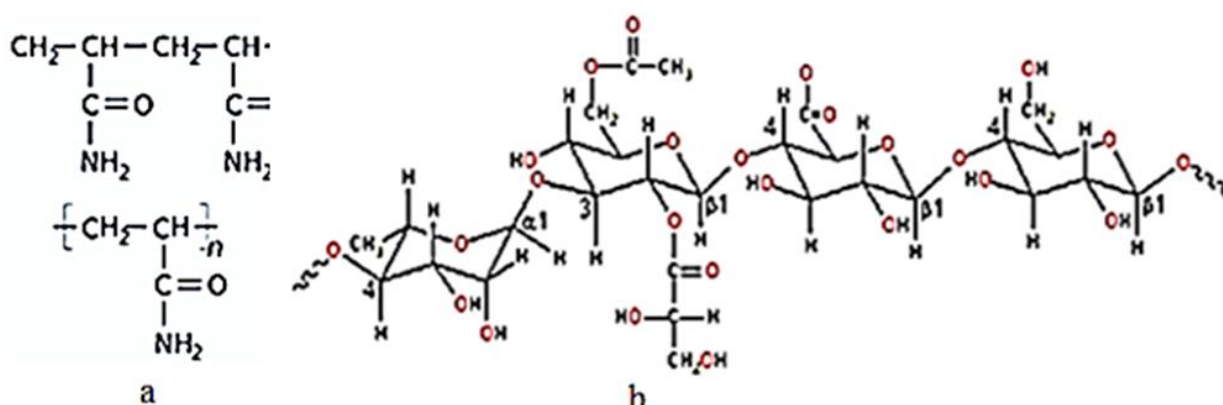


Figure 1 Chemical structure of Polyacrylamide (a) and Gellan gum (b)

Among them,  $Na^+$  exhibits high ionic conductivity also good self-healing efficiency besides high stretchability so consider by us. Herein, we report a high-performance novel double network GPE for use in flexible SC by the DN structure with excellent self-heal ability and enhanced mechanical property. DN hydrogel through a dual cross-linking strategy, with the ductile, double network, rigid sodium ion ( $Na^+$ ) cross-linked GG being the first network and PAM being the second network. GG network cross-linked by  $Na^+$  ion is mechanical stable with PAM network and then achieved remarkable stress and strain. The significance of this work, GG can be cross-link through  $Na^+$  and plays the ionic mobility role and cross-linker of the sample. The results showed high tensile stress (2 MPa), elongation break (40 mm/mm),

and good conductivity (0.29S/cm) is obtained simultaneously. Besides, the DN GPE can self-recover almost 55% at 60°C temperature and for 8 hours. SCs can be easily prepared by sandwiching the PG DN GPE with two identical electrodes, which are fabricated by electrochemical depositing active carbon, without extra components. The objective of the current study was achieve novel PG DN GPE with the combined advantages of excellent mechanical property and self-healing behaviors, which permits such flexible, wearable, and portable to be compatible for use in energy storage devices.

## 2. Materials and methods

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**2.1. Materials.** GG powder was purchased from [Shanghai Macklin Biochemical Co., Ltd.](#) (China). Acrylamide (AM) and 2-Hydroxy-4'-(2-hydroxyethoxy)-2-methylpropionophenone (Irgacure 2959, MW=224.25) were provided by Aladdin Reagent Co., Ltd. (China). Sodium sulfate (anhydrous, Na<sub>2</sub>SO<sub>4</sub>, M=142.04) was obtained from Shangyangong Co., Ltd. (China). Activated carbon (Acetylene black (CB) and binder Polytetrafluoroethylene (PTFE aqueous solution wt.60%)) were used for the made electrode. All the reagents were used without further purification.

### 2.2. Fabrication of self-healing GG-Na<sub>2</sub>SO<sub>4</sub>/PAM DN GPE.

DN GPE was synthesized by a one-pot method (Chen et al.), initially, GG (respectively 0-0.5 gr), AM (6g), Na<sub>2</sub>SO<sub>4</sub> (0.2-1 moll), and DI water (10 mL) were added into glasses beakers. The bubbling was removed in the sample by ultrasonic vibrator and vacuum pumping, heated at 95°C water, and stirred for 2 hours under stirring speed at 10 m/r. After been a homogeneous solution, then Irgacure 2959 (0.085g) added, the solution was stirred again for 10 min. After all the reagents were dissolved, the resulting solution was rapidly injected into a glass mold (10cm × 60cm). As photo-polymerized under a UV Lamp with 365 nm wavelength and 8-watt power for 3 hours, finally the sample was kept at room temperature (RT) for 2 hours to complete the polymerization process. By this method GG/PAM DN gels successfully prepared. PAM SN gels were also synthesized by the same process except for no GG added.

**2.3. Fabrication of activated carbon electrode and assembly of SC.** The active electrode was prepared as follows: activated carbon (AC, 80 wt. %) powder, acetylene black (CB, 10 wt. %) binder Polytetrafluoroethylene (PTFE aqueous solution 60 wt.%) and deionized water (DI, 10 wt. %), all the elements are inserted into the glass dish, added ethanol spirit, and stirred in the ultrasonic vibrator on few times. Then, the resultant slurry was coated on the nickel foam with an area. The sample was dried at 60 in the vacuum oven for 4 hours, the loading mass of each electrode was to be 8–9 mg, then soaked in the Na<sub>2</sub>SO<sub>4</sub> solution 24 hours.

For the two-electrode SC device assembly, the as-prepared PG GPE was simultaneously used as electrolyte and separator. Subsequently, a flexible smart SC was assembled by all the elements together in a sandwich configuration (electrode/gel polymer electrolyte/electrode). A self-made mold was used as Device 1 (Figure 2b) to assemble SC and for a test to the effects of tensile deformation, bending and low temperature on the electrochemical performance of SC prepared the Device 2 (Figure 2c) without mold. All electrochemical measurements of SCs were carried out using the two-electrode system.

### 2.4. Characterization and measurement

**Mechanical Testing:** The hydrogels were cut to dumbbell-shaped samples (Figure 2a), and determining strain and stress curves by the Universal testing machine (WSM-10kN, Changchun Intelligent Instrument Equipment Co. Ltd. China). The samples were stretched at a speed of 100 mm/min under a tensile load of 100 N under ambient conditions.

**Evaluation of Self-Healing Properties:** The DN hydrogels with a dumbbell shape (Figure 2a) were cut into two pieces. The cut discs with different colors were joined together at the cut surface and were sealed in a polyethylene (PE) bag to prevent water evaporation. Then, samples were divided into groups and stored at different temperatures (20, 40, 60, 80, and 100°C) for different periods (0.3, 2, 4, 8, 16, and 24 h, respectively). Subsequently, the healed samples were taken out for tensile tests again.

The surface morphology and energy dispersive spectrometer (EDS) of GPEs membrane were investigated by a FESEM (FEI Quanta 250, Brock AG, German) at 15kV. Before measurement, the membrane samples are fractured in liquid nitrogen and sputtered with Au. The Fourier Transform Infrared (FT-IR) spectrometer (Bruker IFS-25) spectra of non-cross-linked polymers (PAM and GG) cross-linked polymers (Na-GG and Na-GG/PAM) were obtained to confirm the existence of functional groups, was conducted in the range of 4000–400 cm<sup>-1</sup> using [potassium bromide](#) pellet.

The surface chemical composition and chemical states of the elements in GG were determined by X-ray photoelectron spectroscopy (XPS; Axis Ultra, Kratos Analytical LTD, England) with Al K $\alpha$  radiation and low energy electrons stream as the excitation source 225 W, 15mA 15kV). Water content could be estimated using the following equation:

$$Wc (\%) = (Wt - Wd) / Wt \times 100\% \quad (1)$$

where Wc is water content, Wt is the wet weight of membranes before drying, and Wd is the dry weight of membranes after drying in a vacuum oven at 60°C.

**Measurement of ionic conductivity:** The conductivity of the GPE electrolytes was measured by the electrochemical impedance spectrum (EIS) using an electrochemical workstation (Parstat 2273, Princeton Applied Research Co., USA) over a frequency range from 0.1 Hz to 100 kHz. The electrochemical stability window was determined using the cyclic voltammetry (CV) method and also measured by the electrochemical workstation in the potential of -0.09 V to 0.9 V at a scan rate of 5 to 100 mV/s. Bulk resistance R ( $\Omega$ ) was determined by the intercept with the real axis. Ionic conductivity  $\sigma$  (mS cm<sup>-1</sup>) was calculated by the following formula:

$$\sigma = L / (A \times R_b) \quad (2)$$

where L- thickness, A - area, R<sub>b</sub>- bulk resistance of the sample.

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Performance evaluation of SC: EIS measurement of SC was conducted in the frequency range from 0.1Hz to 100 kHz. Galvanostatic charge/discharge cycling was tested using a battery test instrument (CT 2001A, LAND Technology Co. Ltd., China) with different current densities in the potential range of 0.09–0.9V. The CV of the Sc was performed at

various scan rates of 5–100mV/s in the potential range of 0.09–0.9V. The Sc specific capacitance (C, F/g) and electrode specific capacitance (Cs, F/g) could be calculated from the charge/ discharge curve as follows [31]:

$$C = (I \times \Delta t) / (\Delta V \times m) \quad (3)$$

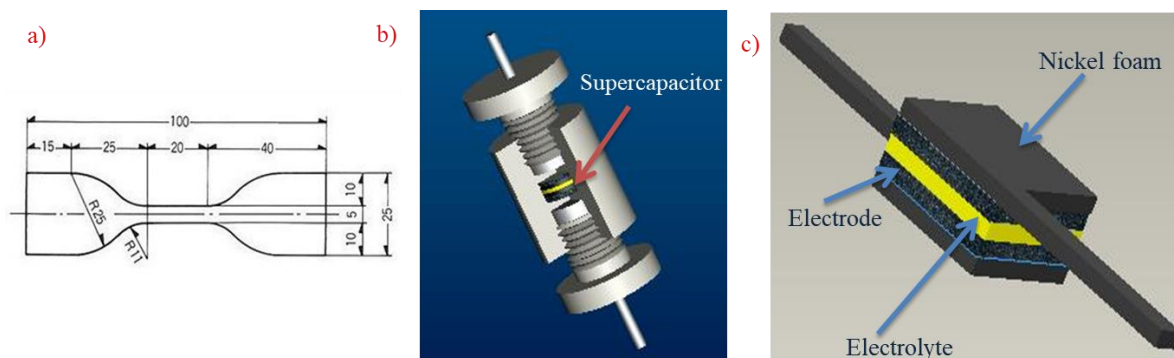


Figure 2. Schematic representation of the SC dumbbell-shape (a) Device 1(b) and Device 2 (c)

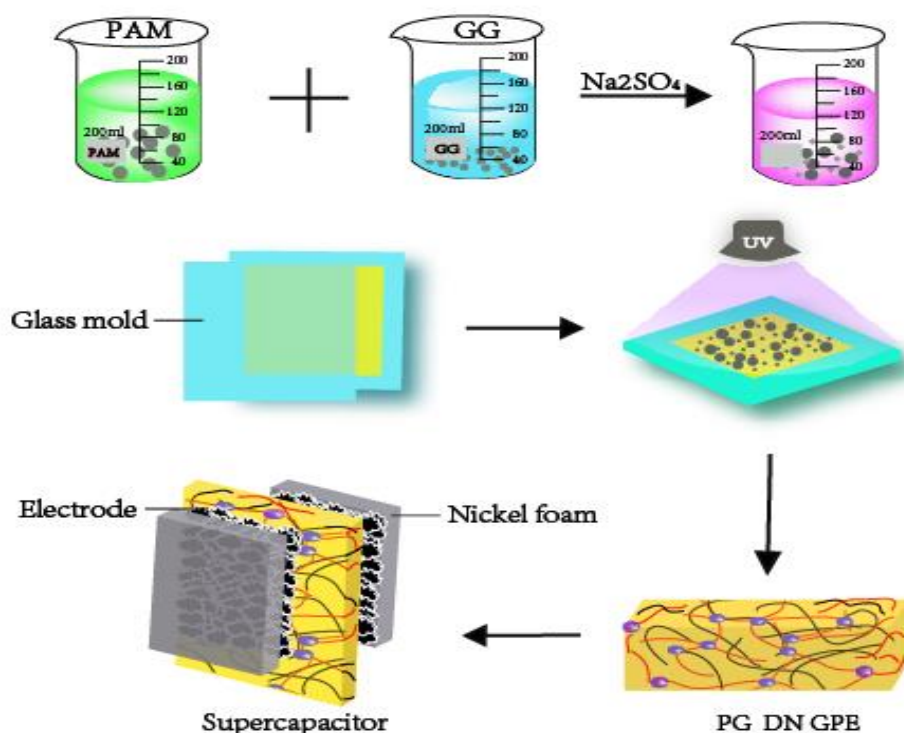


Figure 3. Schematic illustrations of double network structures of Na-GG/Pam GPE

$$C_s = 4 \times C \quad (4)$$

where I is discharge current,  $\Delta t$  is the discharge time,  $\Delta V$  is discharged voltage range, and m denotes the mass of the activated carbon in two electrodes.

### 3. Results

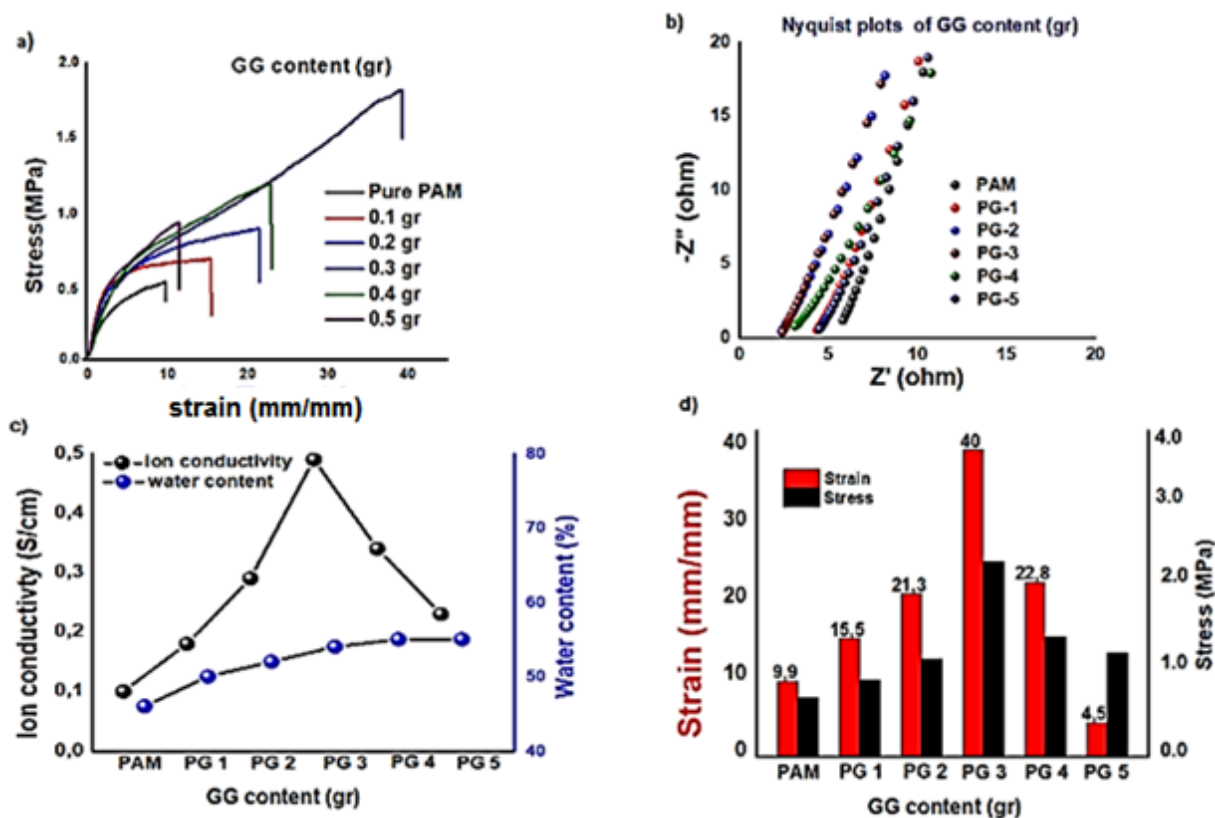
GG as a typical pure natural material is both inexpensive and environmentally friendly materials. Usually, GG widely uses in the food industry, medicine, and pharmaceutical. Nowadays, it is using as like-new material for energy storage materials. However, the mechanical properties of a pure GG hydrogel electrolyte are very weak.

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**Figure 4.** Compare tensile stress-strain curves of various GG content (a) Nyquist plots (b) water content and ionic conductivity of the samples (c) and elongation at break describes by the column graphic.

In this work, PAM was added to form the DN structure that could efficiently energy storage as a way to enhance the mechanical properties of the GG GPE.  $\text{Na}_2\text{SO}_4$ , a good neutral inorganic salt used in electrolytes of flexible SCs, is selected as the ionic conducting medium. Also, GG can cross-link each other via  $\text{Na}^+$  ions, and also this ion provides to improve the ionic conductivity of the samples. To prepare a double network, self-healing, an intrinsically stretchable and tough GPE electrolyte, we selected GG via  $\text{Na}^+$  ionic cross-linked as the first network and PAM as the second chemically cross-linked network. Herein, according to the literature "one-pot" synthesis method was employed to obtain the double-network hydrogel electrolyte one of the best ways, including heating-magnetic stirring cycle followed by UV-induced polymerization. Figure 3

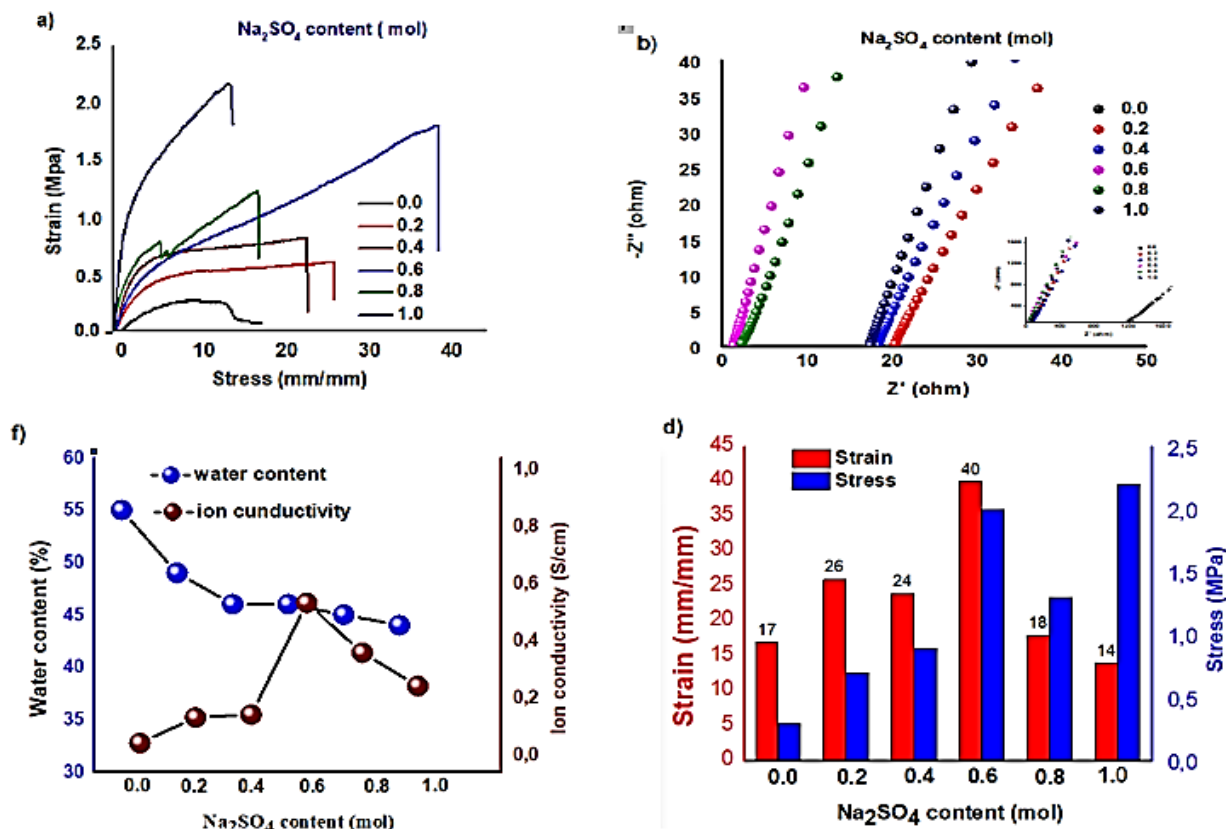
illustrated the synthesis of DN self-healable Na-GG/PAM GPEs.

### 3.1 Mechanical property of the electrolyte

First, we investigated the mechanical properties of DN gels. To prepare the DN self-healing GPEs with high ionic conductivity, a different amount of GG was introduced (denoted as PG DN GPE) and found the tensile properties of DN GPE were significantly influenced by the GG concentration, PAM and  $\text{Na}_2\text{SO}_4$  were constant at this time. Figure 4a and 4d show the typical tensile stress-strain curves of pure PAM and PG DN GPE samples. The concentration of GG is increased from 0 to 5 gr, and the result has shown the pure PAM SN electrolyte without GG was soft (elongation break- 10mm/mm, stress-0.6 MPa).

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**Figure 5.** Compare tensile stress-strain curves of various  $\text{Na}_2\text{SO}_4$  content (a) Nyquist plots (b) water content and ionic conductivity of the samples(c) and effect of  $\text{Na}_2\text{SO}_4$  content on the (f) tensile stress and elongation at break. according to Eq. (1) and Eq. (2).

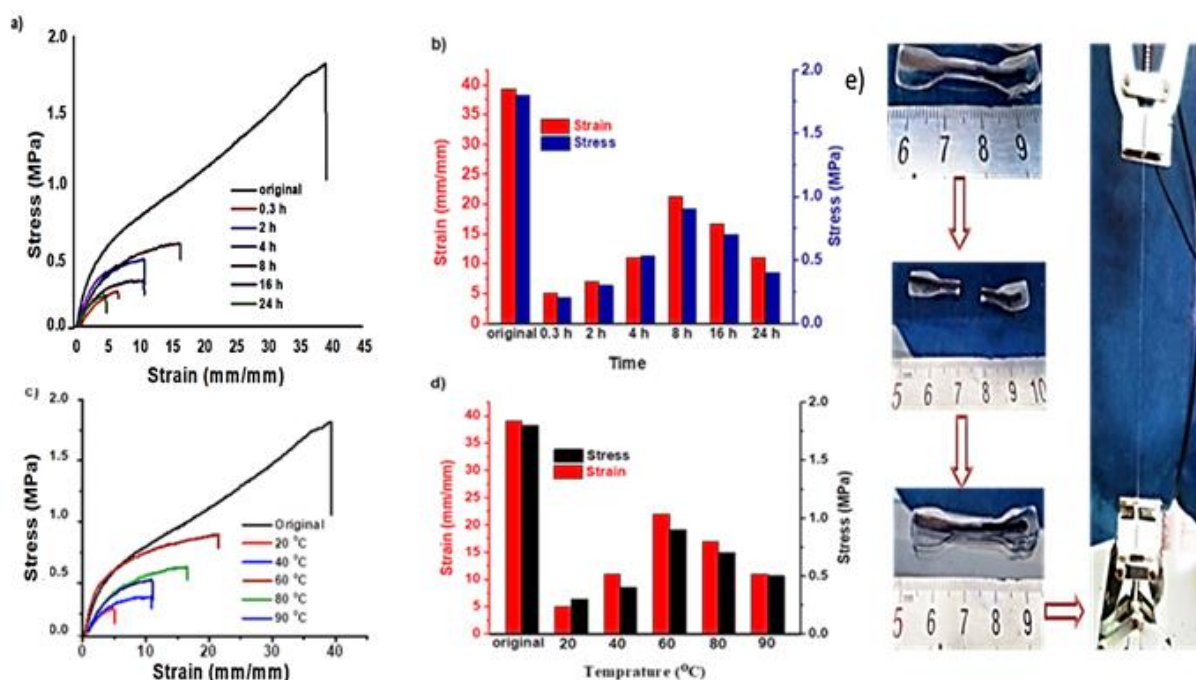
The pure  $\text{Na}^+$  cross-linked GG hydrogel without PAM was so weak that it failed to withstand tester clamping. In contrast, when the GC content reaches 3 gr (PG-3 DN GPE) achieved the highest stress reach 2 MPa, which was almost 4 times higher and the corresponding elongation break was 400%, slightly larger than that of the pure PAM hydrogel. These results demonstrate that the concentration of GG affected the mechanical properties of hydrogels greatly. After continuing to increase again GG amount, the mechanical properties began to decline. For the reason that the PAM and GG mixture solution becomes very viscous when the GG content exceeds 3gr, which may cause the inhomogeneity and a lot of tiny bubbles so that the mechanical properties decrease. The ionic conductivity and water contents exhibit similar rising trends with GG content increasing from 0 to 5 gr. The ionic conductivity and the water content vary from 0.1 to 0.29 S/cm (Fig. 4c) and from 46 to 55%

**4. Discussion.** As we know, the ionic conductivity of hydrogels without the ionic

conducting medium is very low. The key to self-healable DN GPE was the use of GG doped into the PAM solution and subsequently converted to a hydrogel by adding  $\text{Na}^+$  under basic conditions. So  $\text{Na}_2\text{SO}_4$  plays two critical roles in this case: cross-linked agent and ion mobility. Therefore, in the next step, we determined out the most suitable  $\text{Na}_2\text{SO}_4$  content to keep a balance between the conductivity of the gel and its effect on its physical performance. The ionic conductivity of the PG DN GPE rapidly increases from 0.0005 to 0.29 S/cm<sup>2</sup> with the increase of  $\text{Na}_2\text{SO}_4$  concentration from 0 to 1mol (Fig.5a/b). Cause by the salts and enhanced ionic conductivity and consequently increasing the number of conductive ions. The equilibrium water contents of the GPE (shown in Figure 5c) decreased with increasing  $\text{Na}_2\text{SO}_4$  content, the possible reason that by the salts the sample density increases and also that the formation of the double cross-linked network made the hydrogel structure more compact and prevented the penetration of water molecules.

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**Figure 6. Self-healing test: Tensile strain-stress curves after healing different time (a) and temperature(c), tensile stress and elongation at break respectively (b,d), digital photo of self-healing test process(e).**

Besides, the most important  $\text{Na}_2\text{SO}_4$  also affected the mechanical properties of the GPE. As the concentration of the  $\text{Na}_2\text{SO}_4$ , electrolyte increases from 0 to 1 mol, when reaching the concentration of  $\text{Na}_2\text{SO}_4$  0.6mol the best result showed which is the stress increases from 0.3 to 2 MPa, the elongation break from 13 to 40 mm/mm (Fig.5d). The enhanced mechanical properties may result from the enhanced interaction between GG networks  $\text{Na}^+$  serving as bridges. When the  $\text{Na}_2\text{SO}_4$  concentration increases until 1 mol, the stress increase to 2.2 MPa, however, the elongation break decreases to 15 m/mm. It may be attributed to the  $\text{Na}_2\text{SO}_4$  aggregation in high concentration, leading to the destruction of hydrogen bonds between GG networks. Therefore, the PG 3 DN GPE with  $\text{Na}_2\text{SO}_4$  concentration of 0.6 mol is selected for further investigations considering ionic conductivity and mechanical properties. Considering the physical nature of GG and PAM could demonstrate self-recovery property under appropriate conditions. Curiously, the PG 3 GPE could automatically repair itself when suffering physical damage. This kind of ability allows us to use electrolyte for a long time. For this, PG GPE was surveyed by cut the dumbbell-shape electrolyte into two pieces completely and then the pieces were physically contacted together without applying any external force to determine self-healing ability (Fig.6a-e). When the GPE was cut into two parts, hydrogen bonds were cleaved at the cut interfaces. However, once the two parts contacted together again, the broken hydrogen bonds were prone to link

together. The recombination of hydrogen bonds enabled the severed electrolyte to heal itself, leading to the recovery of the electronic conductivity along with the hydrogel. We further quantitatively evaluated the self-healing efficiency by comparing the tensile properties of original and self-healed PG 3 DN GPE at different temperatures and times. In Fig.7, it was shown tensile stress-strain curves before and after self-healing mechanical properties at different times 0.3, 2, 4, 8, 16 and 24 hours, various temperatures 0, 20, 40, 60, 80 and 90°C. The effect of healing time in Figure 5 a,b and temperature on the PG 3 DN GPE, was shown the tensile stress and elongation break of the healed GPE increased as the increase of healing time. The gel healed for 8 h at 60 °C reached indicating a high self-healing performance and excellent tensile properties of PG GPE (stress-0.38 MPa and elongation break of 420%) relative to the original length.

To improve the mechanical properties of GPE in further investigations we are planning to use the method of High-Performance Size Exclusion Chromatography [32, 33] to establish the quantitative relationship between molar mass and tensile strain-stress, elongation degree of polymers in GPE. The given method allows expressing the determination of molar masses and polydispersity degrees of GPE components and gives reliable data for the optimal choice of polymer type in GPE to reach good performance exploitation characteristics of SC product.

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## Conclusions

According to the above experiments a new GPE exhibits outstanding mechanical and electrochemical properties. Additionally, GPE has also shown promising self-healing ability under room conditions

without external situations. This kind of ability gives us GPE use for a long time under mechanical deformation conditions. So, we offer novel GPE for flexible and wearable energy storage SC devices.

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	<b>GIF (Australia) = 0.564</b>	<b>ESJI (KZ) = 9.035</b>	<b>IBI (India) = 4.260</b>
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