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Highly Elastic Slide-Ring Hydrogel with Good Recovery as Stretchable Supercapacitor

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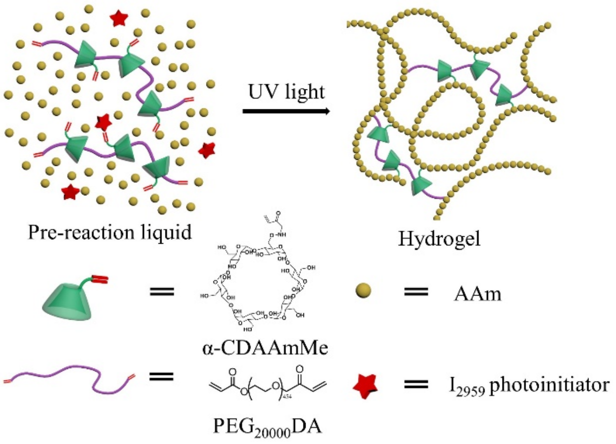
Abstract: As a new material with excellent mechanical properties and good stability, slide-ring gels have attracted more and more attention and research. However, it cannot be widely used due to its relatively complicated synthesis. Herein, we use 6–acrylamidomethylether-modified α-cyclodextrin (αCDAAmMe) and PEG$_{20000}$ diacrylate (PEG$_{20000}$DA) to construct a polypseudorotaxane. Then, the polypseudorotaxane reacts with acrylamide via a photo-initiated polymerization in-situ to conveniently obtain a slide-ring hydrogel with good elastic property and high recovery property. That is, the hydrogel can be easily stretched to 22.5 times of its original length but recovered rapidly and almost reversibly. These results enable the application of hydrogel to make an iintrinsically stretchable and ccompressible supercapacitor after doping ions and the adhesion of commercially available carbon nanotube (CNT) paper as electrodes, giving the ionic conductivity as 17.0 mS/cm (comparable to that of the commercial PVA/H$_3$PO$_4$ electrolyte) and the capacitance as 0.87 µF/cm2 (at the scan speed of 100 mV/s), and its capacitance can be further enhanced under the stretching.

Hydrogel is widely used in various fields, such as tissue engineering, drug and protein delivery, cosmetics and absorbents in waste management.[1] Many applications place increasing demands on the mechanical properties of hydrogels. However, the traditional chemical polymer gels would be broken by only 1.2-1.5 times uniaxial stretching due to the relatively weak mechanical properties, since their network can hardly disperse the internal stress.[2] Therefore, scientists developed a series of methods to improve the mechanical properties of polymer hydrogels. One of the most common strategies is to introduce the non-covalent force into the polymer matrix which can make the network easily to dissipate mechanical energy, such as hydrogen bonding, host-guest interaction, hydrophobic interaction, etc.[3] Although these hydrogels cross-linked by noncovalent bonds (called as physical hydrogels) are easy to obtain high elasticity, they may be dissolved during the long-term usage and have a large hysteresis on the repeated extension and contraction.[4] Another strategy is to optimize the polymer network structure of chemically cross-linked hydrogels to make it easily disperse internal stress. A very typical example is the double-network gel, in which two polymer networks with different chain lengths are interspersed with each other, and the short-chain polymer network is ruptured for energy dissipation, but the long-chain polymer network maintains the high elasticity of the gel.[1f,1g,5] Practice has proved that this gel have the excellent mechanical properties as well as the good stability, which is superior to the physical polymer hydrogels. However, a serious problem we must face is that the preparation method of the double-network gel hydrogels is usually complicated, which greatly limits its practical application.

Recently, the slide-ring gel, composed of a polyrotaxane cross-linked polymer network and the high content of solvent,[6] has been widely regarded as a type of highly elastic chemically cross-linked gel, where the ‘ring’ linked to the polymer chain can slide freely through the axis, giving the gel network the excellent internal stress dispersion ability.[7] However, similar to the case of the double-network gel, the synthesis of slide-ring hydrogels is also exceedingly difficult, mainly because the low synthesis efficiency of the polyrotaxane. Taking the synthesis of a commonly used PEG-αCD polyrotaxane as an example, its yield is usually 5% -10%. In addition, the dialysis operation must be used in this process, which is very unfriendly to the industrialization.[8]

In this work, we develop a simple and convenient two-stepped method to get a slide-ring hydrogel. Firstly, the threading of 6-acrylamidomethylether-modified α-cyclodextrins (αCDAAmMe) on the PEG$_{20000}$ diacrylate (PEG$_{20000}$DA) produces a polypseudorotaxane, then the polypseudorotaxane co-polymerizes with the acrylamide monomer via a photo-initiated polymerization for 15 min. As a result, a highly elastic slide-ring hydrogel with excellent mechanical properties is obtained, showing a uniaxial stretch over 2150%, a high fracture toughness of 35.84 MJ/m^3 and a high tension strength of 6.90 Mpa. Importantly, this hydrogel can be used to construct an intrinsically stretchable and compressible supercapacitor after simply doped with 1M Li$_2$SO$_4$ (to improve the ions conductivity) and the adhesion of commercially available carbon nanotube (CNT) paper as electrodes.[9]



Scheme 1. Construction of αCDAAmMe-PEG$_{20000}$DA hydrogel

Both αCDAAmMe and PEG$_{20000}$DA were synthesized via a simple one-step reaction in high purity (Figure S1-S2),[10] and they can easily assemble to a polypseudorotaxane structure in water through host−guest interactions.[11] An intuitive

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10.1002/chem.202001729

phenomenon is that both αCDAAmMe and PEG$_{20000}$DA have good water solubility, but when they are mixed in water, a large amount of precipitate gradually generates. To further confirm the formation of polypseudorotaxane, the 2D ROESY experiment was performed (Figure S3). It can be seen that the signals of αCDAAmMe (3- and 5-H protons of α-CD at 4.00-3.75 ppm) present the clear NOE correlations with PEG protons (3.69 ppm), indicating that the PEG chain is included in the cavities of αCDAAmMe.

When the αCDAAmMe-PEG$_{20000}$DA polypseudorotaxane suspension was added to an acrylamide aqueous solution, the mixture became clear and transparent after a slightly shaking. A possible reason may be that the hydrogen bonding interactions between acrylamide and cyclodextrin hinder the aggregation of αCDAAmMe-PEG$_{20000}$DA polypseudorotaxane and thereby enhances its water solubility. In addition, the 2D ROESY spectrum of αCDAAmMe-PEG$_{20000}$DA polypseudorotaxane is nearly unchanged even in the presence of high concentration acrylamide (Figure S4)，indicating that the polypseudorotaxane may be stable during the subsequent polymerization reaction.

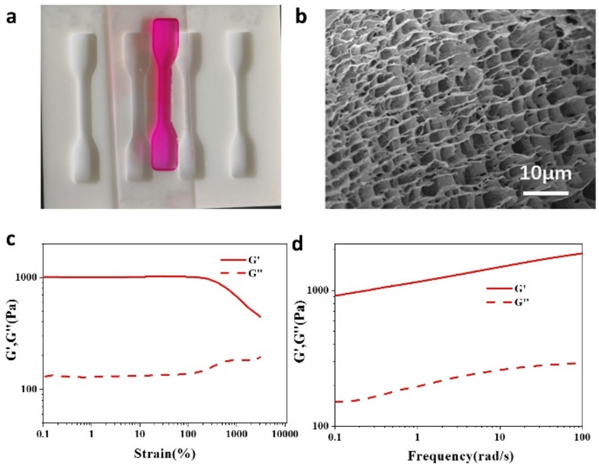

Figure 1. a) Image of αCDAAmMe-PEG$_{20000}$DA hydrogel (Stained with Neutral red). b) SEM image of frozen-dried αCDAAmMe-PEG$_{20000}$DA hydrogel. c) G′ (storage modulus) and G″ (loss modulus) as a function of strain. d) G′ and G″ as a function of frequency.

The αCDAAmMe-PEG$_{20000}$DA hydrogel was prepared by a simple photo-initiated polymerization reaction in the presence of a little amount of I$_{2959}$ in water.[12] In the polymerization process, a large amount of acrylamide monomer and a small amount of polypseudorotaxane copolymerize to form a three-dimensional network structure, meanwhile the polypseudorotaxane is capped by the newly generated polyacrylamide main chain. In addition, the polymerization efficiency is measured up to >95% by comparing the dry weight of the gel before and after excess water soaking, and this high polymerization efficiency is favorable to the mechanical properties of the hydrogel. A possible reason may be that the system is completely transparent, and no other molecules compete with the photo-initiator to absorb the UV light. FT-IR and XPS were used to investigate the chemical composition of the frozen-dried αCDAAmMe-PEG$_{20000}$DA gel. The FT-IR spectra present the characteristic absorption of amides at 3325.47 cm^{-1}, 3183.06 cm^{-1}, 1646.01 cm^{-1}, and 1604.27 cm^{-1} (Figure S5). The XPS spectrum also shows the characteristic peaks of polypseudorotaxane crosslinker and the polyacrylamide main chain (Figure S6). A SEM image of the frozen-dried αCDAAmMe-PEG$_{20000}$DA hydrogel shows the clear three-dimensional porous network structure (Figure 1b). By the way, we calculated the water content of the hydrogel as 74.5% based on the mass ratio of dry gel to the freshly made gel. To further study the stability of the hydrogel, a rheological test was performed. It can be seen that, with the strain increasing, G" (loss modulus) is always larger than G' (storage modulus) within a very wide range from 0.1% to 3000% (Figure 1c). In the frequency sweep curve (Figure 1d), with the frequency increasing from 0.1% to 100%, the relatively large G" and relatively small G' gradually increase and remain substantially parallel. These results fully demonstrate the high stability of three-dimensional network of the hydrogel. Furthermore, the swelling experiments show that the dry hydrogel can absorb about 60-times-weight water and then stably exist in water for at least 14 days without being dissolved.

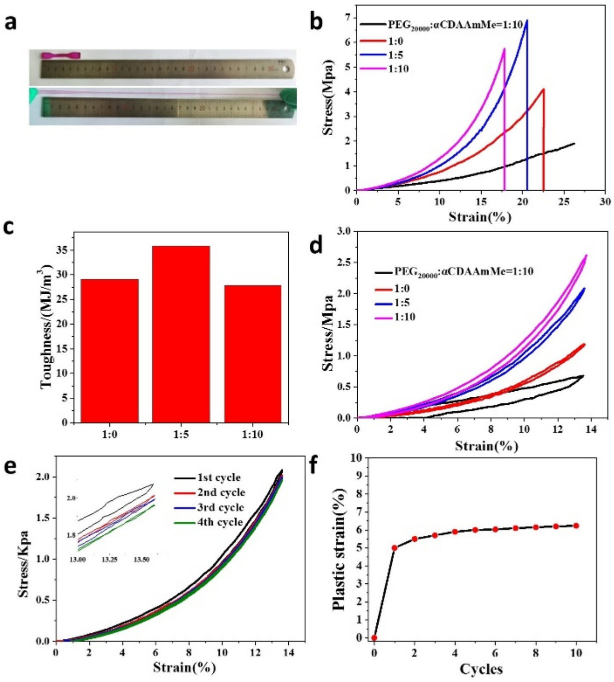

Figure 2. a) Visual photos used to show the excellent ductility (PEG$_{20000}$DA : αCDAAmMe = 1 : 5, stained with Neutral red). b) Tensile stress−strain curves of hydrogels with different polypseudorotaxane components. (black, PEG$_{20000}$: αCDAAmMe = 1 : 10; red, PEG$_{20000}$DA : αCDAAmMe = 1 : 0; blue, PEG$_{20000}$DA : αCDAAmMe = 1 : 5; purple, PEG$_{20000}$DA : αCDAAmMe = 1 : 10). c) Fracture energy of αCDAAmMe-PEG$_{20000}$DA hydrogel with the PEG$_{20000}$DA : αCDAAmMe ratio of 1 : 0, 1 : 5 and 1 : 10. d) Tensile loading−unloading curves of hydrogels with different polypseudorotaxane components at strain of 1350% (samples represented by different colors are the same as in Figure 2b). e) Cyclic loading−unloading tensile curves of the αCDAAmMe-PEG$_{20000}$DA hydrogel (PEG$_{20000}$DA : αCDAAmMe = 1 : 5) at 1350% strain for four cycles (no waiting time between cycles). f) Plastic strain as a function of cycle times at strain of 1200% (no waiting time between cycles, PEG$_{20000}$DA : αCDAAmMe = 1 : 5).

The elasticity of the hydrogel was investigated by a tensile strength measurement. A simple macro tensile test shows that the hydrogel can be easily stretched to 2150% of its original

10.1002/chem.202001729

length (Figure 2b), and can recover to 105% of the original length with almost no delay when losing external tension force. To illustrate the importance of rotaxane structure on improving the mechanical properties of the hydrogel, we prepared a series of hydrogels with different ratios between $PEG_{20000}DA$ and αCDAAmMe. In the control experiment, when PEG_{20000} is used instead of $PEG_{20000}DA$, the strength and recovery properties of the resultant hydrogel are very poor, and the plastic deformation accounts for a large proportion (29.3%) during the stretch process. A possible reason may be that αCD will be inevitably fall off from the PEG chain when the αCDAAmMe-PEG hydrogel is stretched. After using $PEG_{20000}DA$ as linker, the recovery rate of the hydrogels greatly increases owing to the formation of a complete chemically cross-linked network structure. As shown in the Figure 2d, the area of hysteresis loops of this hydrogel is much smaller than that of αCDAAmMe-PEG_{20000} hydrogel. When 5 equivalents of αCDAAmMe (compared with $PEG_{20000}DA$) is added, the strength of the resultant hydrogel increases about two times while its fracture strain only decreases less than 10%, probably because the increase of the number of αCDs on PEG chain greatly increases the crosslinking density of the hydrogel and thus leads to an increase of the hydrogel strength. In addition, the sliding of αCDs along the PEG chain also efficiently avoids the increase of the hydrogel brittleness. When the amount of αCDAAmMe continues increasing to 10 equivalents, although the strength of the hydrogel still increases, a loss of about 18.2% of the fracture strain occurs compared with the former, and the fracture stress of the hydrogel is also decrease from 6.90 MPa to 5.74 MPa (Figure 2b). This might be because that the presence of too much αCDs on PEG chain disfavors the sliding of αCDs to some extent. By calculating the fracture energy, we can see that the fracture energy of the hydrogels increases firstly and then decreases with the amount of αCDAAmMe increasing, when the ratio between $PEG_{20000}DA$ and αCDAAmMe is 1:5, a hydrogel with a relatively maximum fracture energy as 35.84 MJ/m^3 can be obtained, which is rare in such a high water content hydrogel (Figure 2c).

A cyclic tensile loading−unloading test at 1350% strain was performed to study the anti-fatigue performance of the hydrogel. As shown in Figure 2e, during the first stretching cycle, the area of the hysteresis loop is relatively large as 0.714 MPa. But in the subsequent cycles, the hydrogel can almost recover completely, and the corresponding area of the hysteresis loops decreases to < 0.20 Mpa. This might be because the presence of a small amount of polypseudorotaxane that has not been capped in the polymerization process, and this part of unstable structure will be irreversibly destroyed during the first cycle. In order to clearly show the excellent anti-fatigue performance of the hydrogel, we tested its plastic strain at 1000% strain with the cycles increasing to 10 (Figure 2f). We can see that after the first cycle with about 5% plastic strain, the hydrogel can recover almost completely in the subsequent cycles.

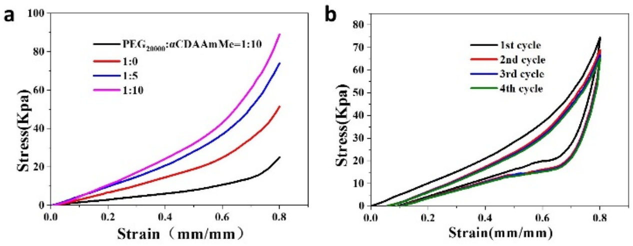

Figure 3. a) Compressive stress−strain curves of the hydrogels with different components. (black, PEG_{20000} : αCDAAmMe = 1 : 10; red, $PEG_{20000}DA$: αCDAAmMe = 1 : 0; blue, $PEG_{20000}DA$: αCDAAmMe = 1 : 5; purple, $PEG_{20000}DA$: αCDAAmMe = 1 : 10). b) Cyclic compressive curves of αCDAAmMe-$PEG_{20000}DA$ hydrogel ($PEG_{20000}DA$: αCDAAmMe = 1 : 5) at 80% strain for four cycles (no waiting time between cycles).

The same control group was used for the compressive strength measurement. The results show that the αCDAAmMe-PEG hydrogel hardly recovers after the compression, while the hydrogels with a complete polyrotaxane structure show the excellent strength and recovery properties. With the equivalent of cyclodextrin increasing from 0 to 10, the strength of the hydrogel increases gradually from 51.39 kPa to 89.01 kPa (Figure 3a). In the cyclic compressive test, except for the first cycle, a large plastic deformation occurs, and the sample can recover non-destructively in the subsequent compression cycle (Figure 3b). These results are consistent with the results of the tensile strength test.

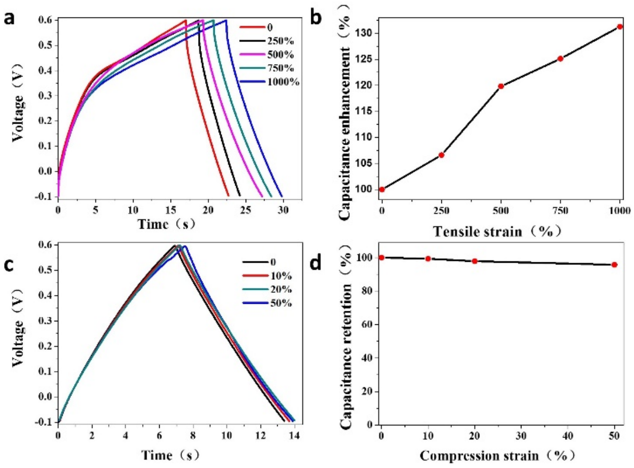

Figure 4. a) Galvanostatic charge/discharge (GCD) curves from 0–1000% tensile strain. b) Capacitance enhancement factor obtained from GCD curves as a function of the tensile strain. c) GCD curves at 0% (black), 10% (red), 20% (green), and 50% (blue) compressive strain. d) Capacitance retention obtained from GCD curves as a function of the compressive strain

The hydrogels with such excellent mechanical properties are very suitable to fabricate the flexible supercapacitor.[9]Herein, we chose the αCDAAmMe-$PEG_{20000}DA$ hydrogel ($PEG_{20000}DA$: αCDAAmMe = 1 : 5), which has a relatively larger fracture energy, as the electrolyte of the capacitor. Before the preparation of the capacitor, Li_2SO_4 was doped in the hydrogel to enhance the conductivity. The stretchable supercapacitors was prepared by a reported method,[9a] where a commercially available carbon nanotube (CNT) paper was used as the electrode to be directly stuck on each sides of the 1000% pre-stain hydrogel without any additional adhesive (Figure S7). As shown in Figures S8-9, the tensile strength, the compressive strength and the fracture strain of the Li_2SO_4-doped hydrogel reduced by 22.8%, 6.6%, and 8.4% respectively compared with those of αCDAAmMe-PEG_{20000}DA hydrogel, mainly because a part of hydrogen bonds between amide bonds are destructed by salt. However, the hydrogel remains the excellent recovery property (Figure S10). Significantly, the ionic conductivity of the ion-doped hydrogel is calculated to be 17.0 mS/cm from the electro-chemical impedance spectra (EIS, $R_0 = 5.52\ \Omega$, S = 2.1 cm^2, L = 0.2 cm, see the supporting information for the corresponding formula), which is comparable to that of the commercial PVA/H_3PO_4 electrolyte (Figure S11). Due to the good adhesion of the hydrogel, after releasing the pre-strain, the CNT-paper forms a stable wave structure attaching on the surface of the hydrogel. Owing to the good stretchable property of the hydrogel, the resultant supercapacitor is also tensile and compressive. With the capacitor in the initial state and based on the cyclic voltammetry (CV) curves at various scan rates, we can calculate that the capacitance of the supercapacitor decreases from 0.87 μF to 0.56 μF with the sweep speed from 5 mV to 100 mV. This result demonstrates the stable electrochemical performance of the supercapacitor (Figure S12-13), which can also be corroborated from the GCD curves at various currents. Interestingly, the capacitance of the supercapacitor (calculated from the GCD curves) increases 1.3 fold under the 1000% tension strain (Figures 4a and 4b), but is basically unchanged at 0 to 50% compression strain (Figures 4c and 4d). A possible reason may be that the capacitance is proportional to the effective area of the electrode. In the stretching process the CNT paper with a wrinkled structure will stretch out, and the contact area between the CNT paper and the hydrogel increases, leading to increase of the capacitance. However, in the compression process, the compression occurs in a perpendicular direction to the CNT paper, thus the effective area has no significant change. These results show that the supercapacitor can be used under certain mechanical conditions to give the high mechanical and stable electrochemical performance. It is noteworthy that, besides the lithium salt electrolytes, the hydrogel can be applicable for the conventional basic electrolyte but not acidic electrolyte, because the cyclodextrin will decompose to some extent under a strong acidic condition.

In conclusion, we developed a simple method to construct a slide-ring hydrogel that exhibits the high mechanical strength, large fracture strain and excellent recovery property. The method of using the polypseudorotaxane directly polymerizing with the acrylamide monomer can not only avoid the difficult synthesis, but also retain the excellent mechanical properties of traditional polyrotaxane hydrogel that has a good commercial prospect. Furthermore, after Li_2SO_4 is doped to improve the conductivity, the polypseudorotaxane hydrogel can be used as an electrolyte to construct a stretchable supercapacitor with excellent mechanical properties and stable electrochemical performance.

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Keywords: slide-ring • polypseudorotaxane • hydrogel • highly elastic • stretchable supercapacitor

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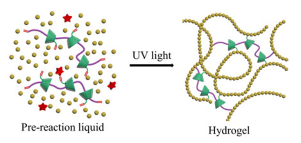

A highly elastic slide-ring hydrogel with good recovery was obtained by a two-stepped method conveniently. Firstly, the threading of 6-acrylamidomethylether-modified α-cyclodextrins (αCDAAmMe) on the PEG_{20000} diacrylate ($PEG_{20000}DA$) produces a polypseudorotaxane, then the polypseudorotaxane co-polymerizes with the acrylamide monomer via a photo-initiated polymerization. Notably, the hydrogel can be used to construct a strechable supercapacitor after simply doped with 1M Li_2SO_4 and the adhesion of carbon nanotube (CNT) paper as electrodes.

Li Feng, Shanshan Jia,Yong Chen, and Yu Liu**

Page No. – Page No.

Highly Elastic Slide-Ring Hydrogel with Good Recovery as Stretchable Supercapacitor