

Electronic Supplementary Information (ESI) Materials for RSC Advances
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Hydrogels containing layered double hydroxide nanosheets: rheological behaviour and excellent stabilities

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Experimental

Materials

Sodium isethionate ($\text{HO}(\text{CH}_2)_2\text{SO}_3\text{Na}$) was purchased from Sigma-Aldrich Chemical Reagent Co., Ltd (98% in purity), and used without further purification. All of the chemicals including $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, and $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ are of analytical pure reagent (A.R.) grade. In all of the experiments, deionized water was used.

Preparations

MgAl-LDH-NO_3 was synthesized by a typical coprecipitation method. In brief, an aqueous solution of $\text{Mg}(\text{NO}_3)_2$ (0.75 M) and $\text{Al}(\text{NO}_3)_3$ (0.25 M) and a solution of NaOH (1 M) were added dropwisely into the NaNO_3 solution (0.1 M). During adding process, the pH value of the system was kept at constant pH = 10.0 under vigorous stirring in nitrogen atmosphere. Then, the mixture was aged at 80 °C for 8 h. Finally, the resulting precipitation was filtrated, washed with deionized water, and dried in a vacuum oven at 50 °C for 24 h.

LDH-Ise was prepared according to the literature (N. Iyi, Y. Ebina and T. Sasaki, *J. Mater. Chem.*, 2011, 21, 8085–8095), in which anion exchange was repeated for three times and no NO_3^- remained could be detected by FTIR spectroscopy.

The prepared LDH-Ise powder was added into water at concentration of 10, 15, 17.5, 20, 25, 30, 40 or 50 g L⁻¹. Up to 5 min under ultrasonic treatment, it swelled automatically. After resting for a few minutes or hours, a series of translucent LDH-Ise green hydrogels were obtained.

Characterization

X-ray diffraction (XRD) measurements were conducted using a Rigaku D/max 2500 diffractometer with Cu K α radiation ($\lambda = 0.15418$ nm) at a scanning rate of 4° min⁻¹. Fourier transform infrared (FTIR) spectra (KBr pellet) were recorded using a Perkin-Elmer System 2000 FTIR spectrophotometer for 64 scans in the wavenumber range of 4000 to 400 cm⁻¹. The contents of Mg and Al were analyzed using an Axial View Inductively Coupled Plasma (ICP) Spectrometer using a diluted nitric acid solution. And the content analysis of C, H and N elements was carried out using a Flash EA 1112 apparatus. The scanning electron microscopic (SEM) images were collected using a

HITACHI S-4800 scanning electron microscope with an acceleration voltage of 15 kV. The transmission electron microscopic (TEM) images of freeze-etched sample for the LDH-Ise hydrogel (30 g L⁻¹) were collected using a JEM-1011 transmission electron microscopy with an acceleration voltage of 100 kV. The sample was frozen in liquid nitrogen and then freeze-etched by Leica BAF 060 freeze-etching machine. (A. Klaus, G. J. T. Tiddy, R. Rachel, A. P. Trinh, E. Maurer, D. Touraud and W. Kunz, *Langmuir*, 2011, **27**, 4403)

Rheological measurements were performed on an AR2000ex rheometer measured at 25 °C under nitrogen atmosphere. A pair of parallel plates with diameter of 40 mm and a sample gap size of 700 μm were adopted. Strain sweep experiments were first carried out from 0.1% to 100% at an angular frequency of 1 rad s⁻¹ to obtain the linear regime. Then, the dynamic frequency sweep measurements were performed at a strain of 0.1% at shear rate range of 0.1 to 100 rad s⁻¹.

Swelling experiments were carried out by immersing the hydrogel samples at various concentrations in an excess of deionized water at 25 °C for 3 days to reach swelling equilibrium. Subsequently, the hydrogels were weighed after removing excess water from the surface by filter paper. Thus, the relative weight swelling ratio could be measured by $(m_2 - m_1)/m_1$, where m_2 was the equilibrium weight of the swollen hydrogel and m_1 was the weight of the original hydrogel sample. For the hydrogels at each concentration, five samples were adopted, and the average values were used.

Table S1 Data of elemental analysis for LDH-Ise

	C	H	N
wt%	5.83	4.11	<0.3%

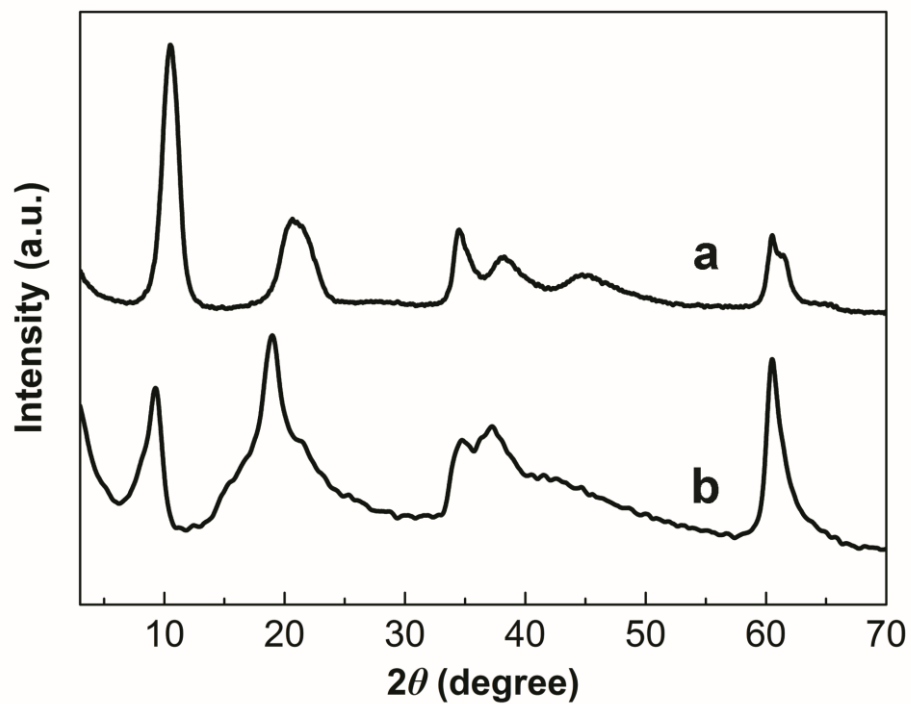


Fig. S1 XRD patterns of (a) LDH-NO₃ and (b) LDH-Ise powders.

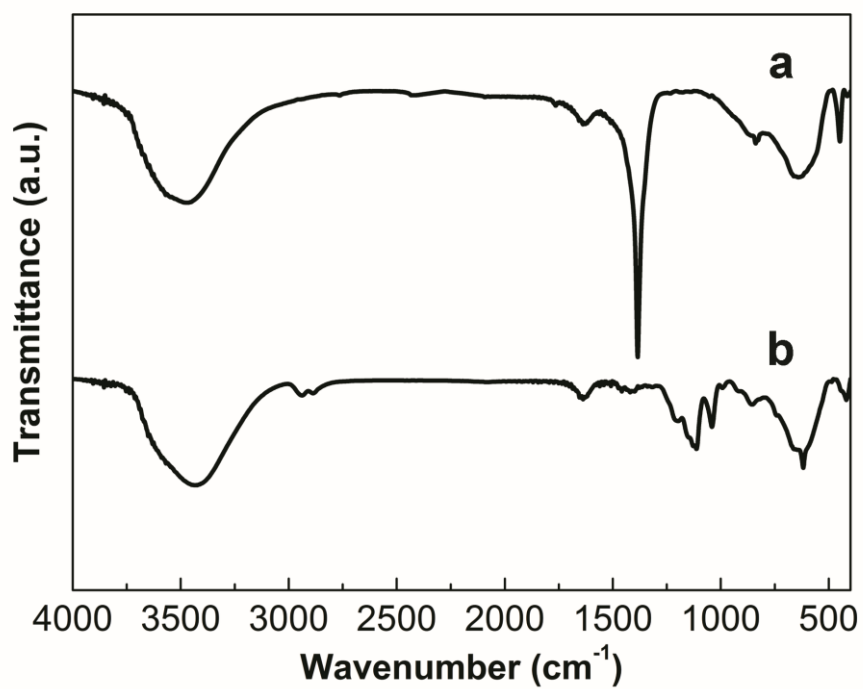


Fig. S2 FTIR spectra of (a) LDH-NO₃ and (b) LDH-Ise powders.

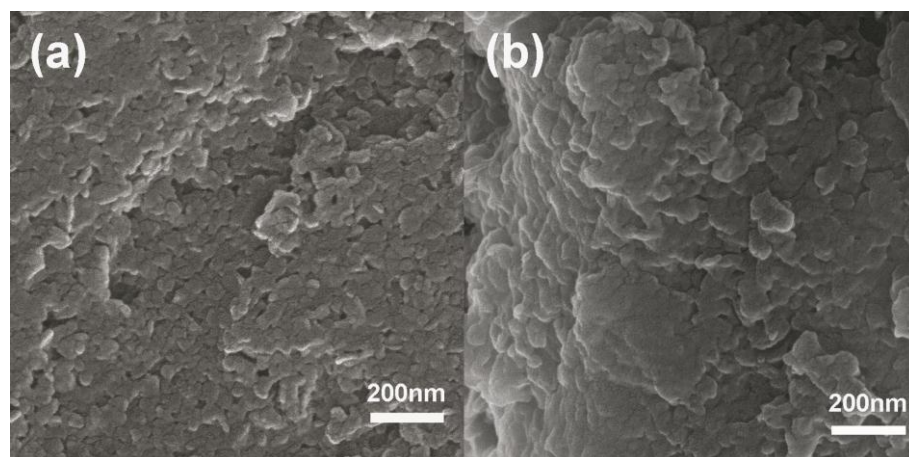
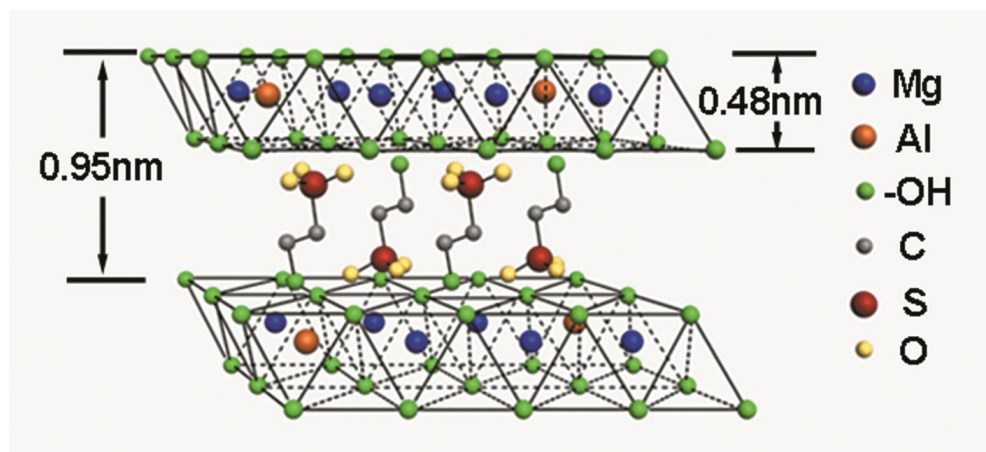


Fig. S3 SEM images of (a) LDH-NO₃ and (b) LDH-Ise powders.



Scheme S1 Schematic illustration of interlayer arrangement for isethionate anions intercalated in LDH-Ise.

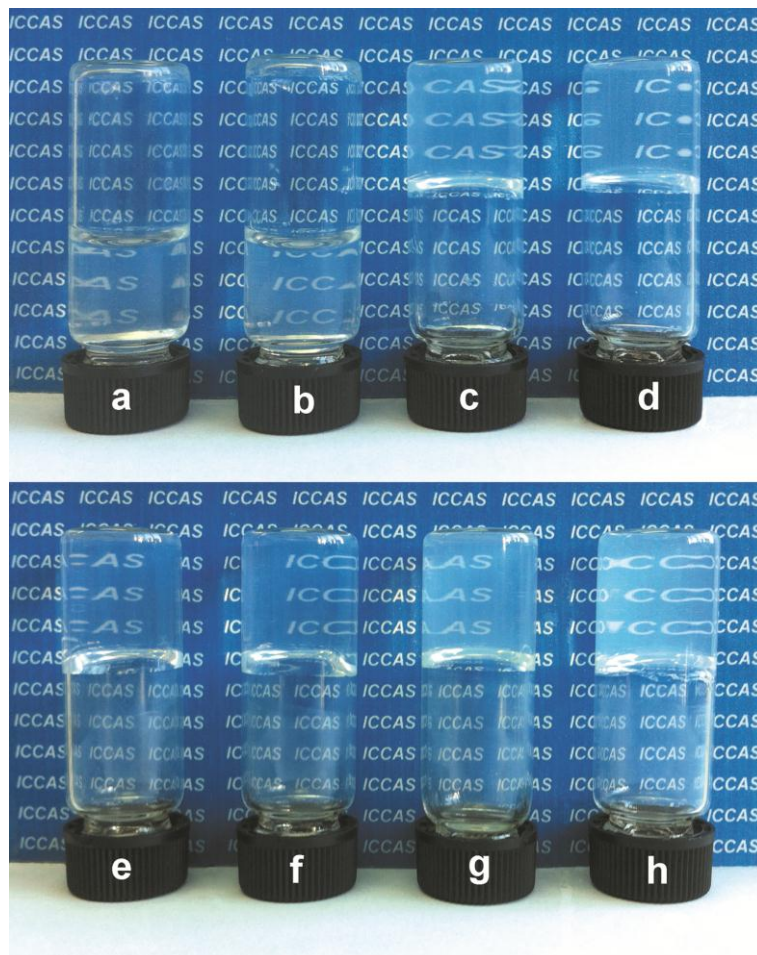


Fig. S4 Vials showing the concentration effect on sol to gel transition for LDH-Ise, where the concentrations were (a) 10, (b) 15, (c) 17.5, (d) 20, (e) 25, (f) 30, (g) 40 and (h) 50 g L⁻¹, respectively.

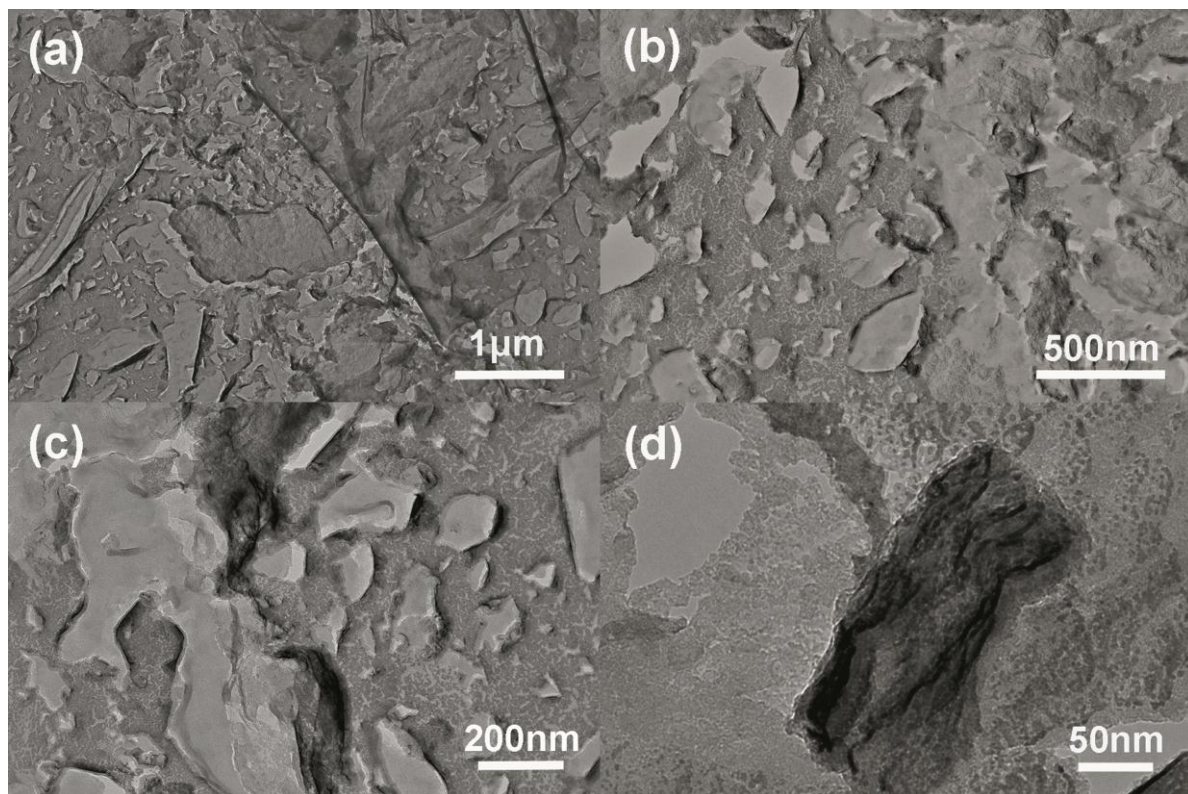


Fig. S5 TEM images of the freeze-etched sample for LDH-Ise hydrogel (30 g L⁻¹).

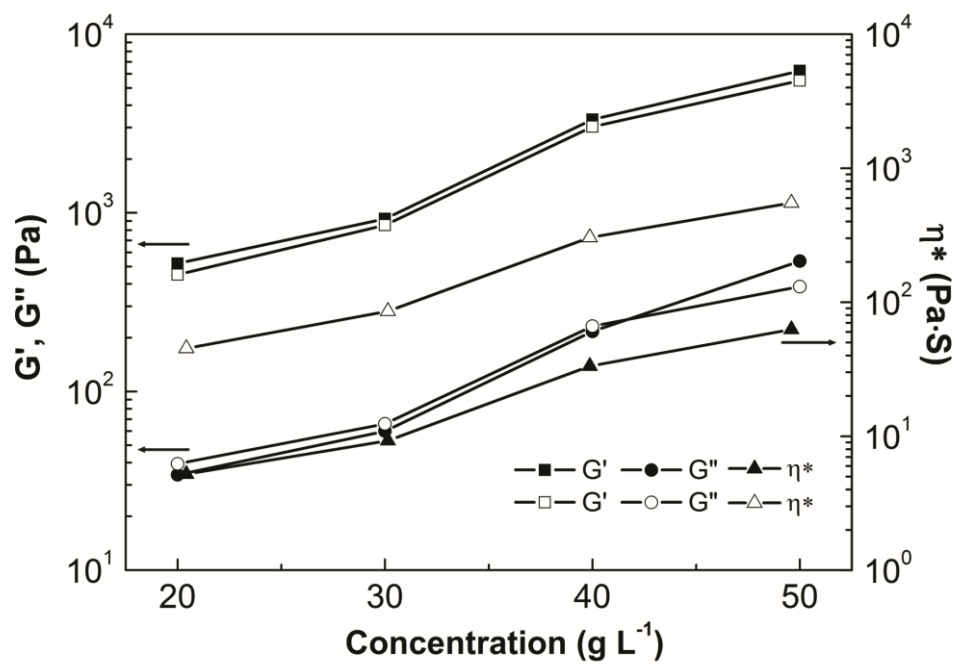


Fig. S6 At fixed frequencies, effect of concentration on the G' , G'' and η^* (black and white symbols corresponded to the frequency of 100 and 10 rad s^{-1} , respectively).

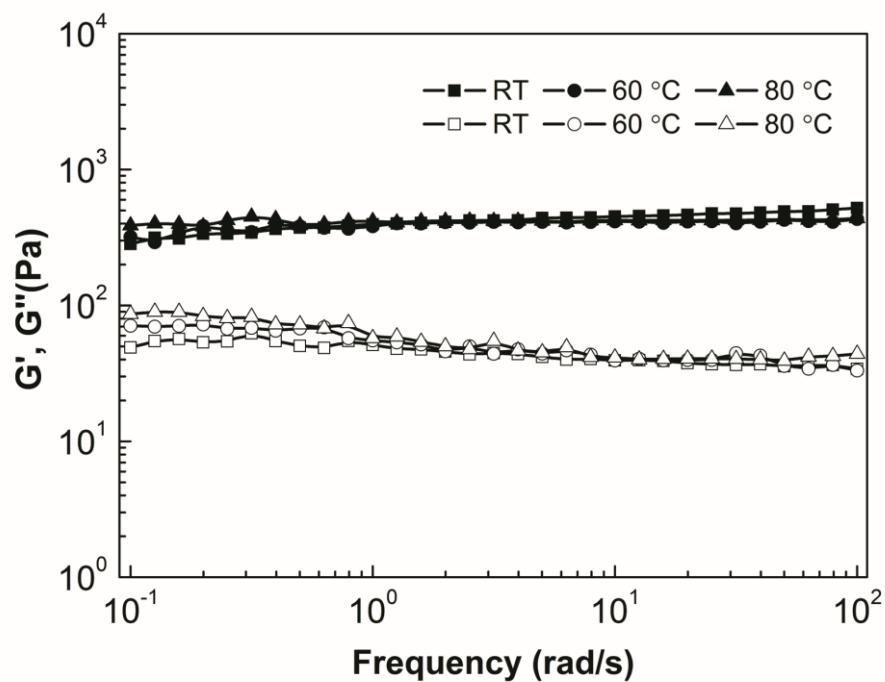


Fig. S7 Effect of temperature on G' and G'' (black and white symbols were denoted as G' and G'' , respectively).

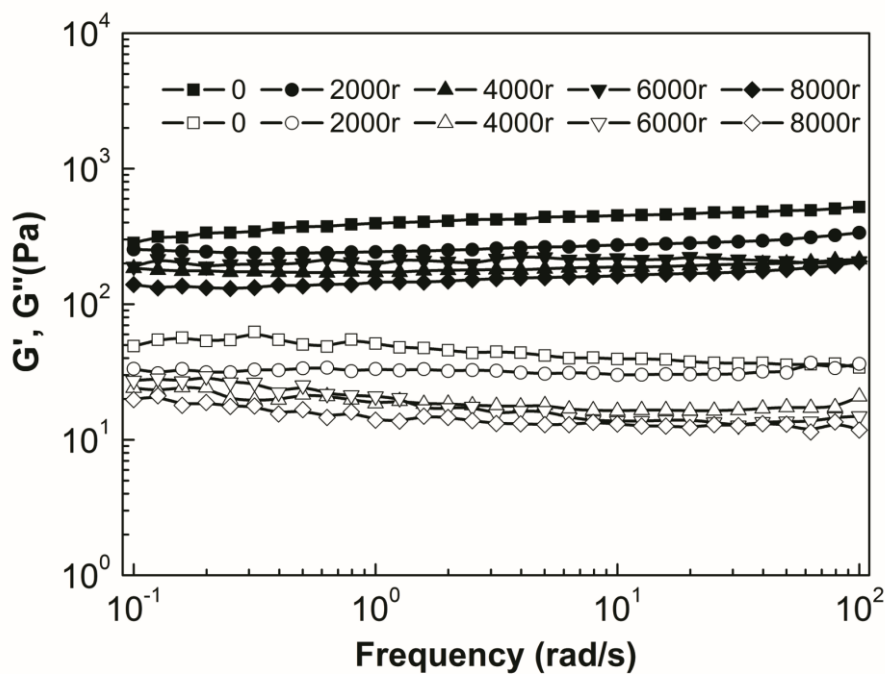


Fig. S8 Effect of centrifugation on G' and G'' (black and white symbols were denoted as G' and G'' , respectively).

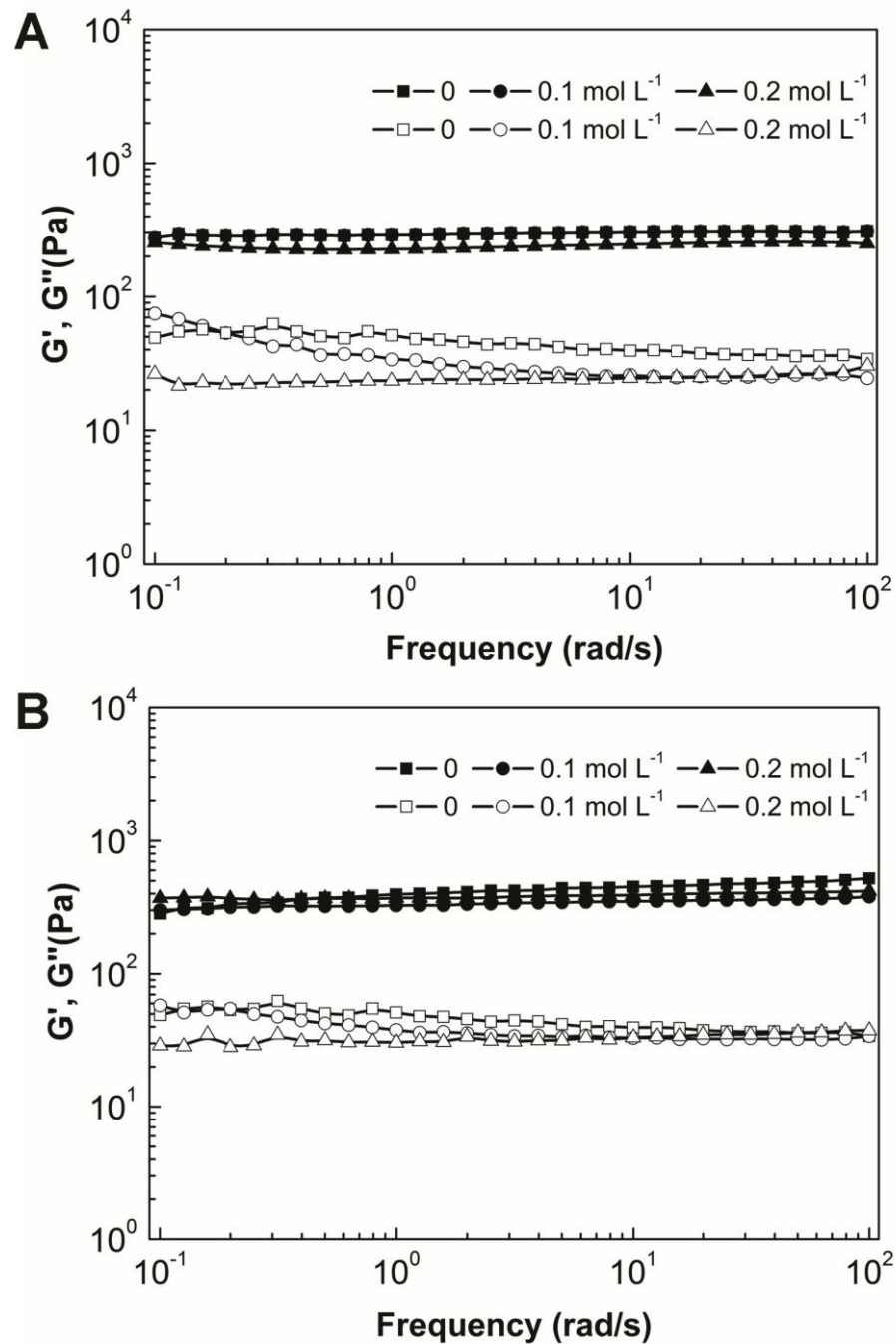


Fig. S9 Effect of (A) acid and (B) alkali treatment on G' and G'' (black and white symbols were denoted as G' and G'' , respectively).

Table S2 The relative weight swelling ratios of LDH-Ise hydrogels in water

g L^{-1}	20	30	40	50
m_1 (g)	2.04	2.06	2.08	2.10
m_2 (g)	2.05	2.07	2.09	2.12
$(m_2 - m_1)/m_1$	0.5%	0.5%	0.5%	1.0%