



Look What They've Done To My Shoes!

SCU_China Project 2016

Protocol
Hardware Team

Agar/PAAm double network (DN) hydrogel synthesis protocol

1) Materials.

All chemicals and solvents purchased were of the highest available purity, and unless otherwise stated they were used as received.

agar was purchased from Sangon Shanghai Inc.

Acrylamide(AAm) was purchased from Chengdu Kelong Chemical Inc.

2-Hydroxy-4'-(2-hydroxyethoxy)-2-methylpropiophenone (Irgacure 2959,BASF) was obtained from Curease Chemical, China.

N,N'-Methylbisacrylamide (BIS) was purchased BioDev, Inc.

2)Synthesis of DN gels

Agar/PAAM DN gels were synthesized by a one-pot method. Take the A5a17 DN gel as an example: 30ml Degas water, 10.662g AAm , 0.51g agar, 0.34g Irgacure 2959, 1.3ml BIS(0.5g/100ml). All reactants were added into a bottle, and the bottle was sealed under N₂ protection after three degassing cycles and then gradually heated up to 97°C in an air oven to dissolve the reactants in the water. The resulting solution was injected into a glass bottle (22*52mm) or a mold and cooled at 4 °C for 60 min to form an agar gel first. The photopolymerization reaction was carried out to form an Agar/PAAM DN gel under UV light (λ = 365 nm wavelength, intensity of 20W, distance of 30cm) for 2~3 h. After polymerization, gels were removed from the molds and used for further tests.

Agar/PAAm/SA triple network (TN) hydrogel synthesis protocol

1) Materials.

All chemicals and solvents purchased were of the highest available purity, and unless otherwise stated they were used as received.

agar was purchased from Sangon Shanghai Inc.

Acrylamide(AAm) was purchased from Chengdu Kelong Chemical Inc.

Sodium alginate (SA) was purchased from Chengdu Kelong Chemical Inc

2-Hydroxy-4'-(2-hydroxyethoxy)-2-methylpropiophenone (Irgacure 2959,BASF) was obtained from Curease Chemical, China.

N,N'-Methylbisacrylamide (BIS) was purchased BioDev, Inc.

3)Synthesis of DN gels

Agar/PAAM DN gels were synthesized by a one-pot method. Take the A5a17S0.2 TN gel as an example: 30ml degassed water, 10.662g AAm, 0.51g agar, 0.02g SA, 0.34g Irgacure 2959, 1.3ml BIS(0.5g/100ml). First, 0.02g SA was dispersed into 30ml degassed water to form a uniform solution. And then all other reactants were added into the bottle, and the bottle was sealed

under N₂ protection after three degassing cycles and then gradually heated up to 97°C in an air oven to dissolve the reactants in the water. The resulting solution was injected into a glass bottle (22*52mm) or a mold and cooled at 4°C for 60 min to form an agar gel first. The photopolymerization reaction was carried out to form an Agar/PAAM/SA TN gel under UV light ($\lambda = 365$ nm wavelength, intensity of 20W, distance of 30cm) for 2~3 h. After polymerization, gels were removed from the molds and used for further tests.

PAMPS/PAAm DN hydrogel synthesis protocol

1) Materials

All chemicals and solvents purchased were of the highest available purity, and unless otherwise stated they were used as received.

Acrylamide (AAm) was purchased from Chengdu Kelong Chemical Inc.

N,N'-Methylbisacrylamide (BIS) was purchased BioDev, Inc.

Postassium persulfate ($K_2S_2O_8$) was purchased from Chengdu Kelong Chemical Inc.

2-Acrylamido-2-methylpropanesulfonic acid (AMPS) was purchased from Xiya, Chemical, Inc.

2) Synthesis of PAMPS/PAAm DN gel

PAMPS/PAAm DN hydrogels were synthesized by a two-step sequential network formation technique. For example, the first network of the PAMPS-1-4/PAAM-2-0.1 DN gel was synthesized from an aqueous solution of 1M 2-Acrylamido-2-methylpropanesulfonic acid (AMPS) containing 4mol% crossing agent, N,N'-Methylbisacrylamide (BIS), and 0.1mol% initiator, Postassium persulfate ($K_2S_2O_8$). After degassing, the premix solution was then poured into glass bottle (22*52mm). And then to put in the 50°C water for about 15h to form the first network. This gel (first network) was then immersing in an aqueous solution of 2M Acrylamide (AAm), containing 0.1mol% BIS and 0.1 mol% KPS at 4°C, for one day until equilibrium was reached. And then the gel was bathed in 50°C water for another 15h. The second network was subsequently synthesized in the presence of the first network.

Laponite/PAAm Nanocomposite (NC) gel synthesis protocol

1) Materials

All chemicals and solvents purchased were of the highest available purity, and unless otherwise stated they were used as received.

Acrylamide (AAm) was purchased from Chengdu Kelong Chemical Inc.

Postassium persulfate ($K_2S_2O_8$) was purchased from Chengdu Kelong Chemical Inc.

Laponite XLG (Rockwood) was obtained from Hualing, Inc.

2) Laponite/PAAm NC gel synthesis

In this work Laponite/PAAm NC gel was designated as NCX-Y, where NC stood for the name of Nanocomposite, X stood for the mass of the Laponite of 100ml water, Y stood for the monomer mass of 100ml water.

DPC gels were synthesized through in situ copolymerization of AM(varied) in the aqueous suspension of LAPONITE XLG (varied) , initiated by KPS (0.0025 wt% relative to the mass of AM). For example, the experimental procedure used for NC2-40 was as follows: 1 g of LAPONITE XLG was first dispersed in deionized water (50 g) under stirring for at least 4 h until a homogeneous suspension was achieved. Then, monomer AM (20 g) was added into the LAPONITE XLG suspension under stirring. The mixture was stirred for another 24 h to make a uniform solution. Finally, the aqueous solution of initiator KPS 0.05 g (0.5g/100ml) was added to the former solution. The solution was put into an glass bottle and then sealed after being bubbled with N₂ for 10 min to eliminate oxygen, the polymerization was allowed to proceed at 50°C for 13h, and then NC gels samples were obtained.

In this work, DPC gels were designated as DPCn, where stood for the mass fraction of LAPONITE® (varying from 0.5 to 1.5) relative to the total mass of initial reaction solution.

Mechanical strength testing

Tensile Test.

Uniaxial tensile tests of as-prepared gels were carried out using a universal tensile tester equipped (Model 5576, Instron Instruments, U.S.A.) with a 1 kN load cell at a crosshead speed of 200 mm/min. The gels were cut into double-bone shape with a gauge length of 25 mm, a width of 3 mm, and a thickness of 3 mm. The fracture strain (ε_f) was determined as

$\varepsilon_f = \Delta l / l_0$ (mm/mm), where Δl is the difference of the elongation length (l) and the initial length

(l_0). The fracture stress (σ_f) was defined as $\sigma_f = F / A_0$ where F is the load force and A_0 is

the original specimen crosssectional area. The elastic modulus (E) of gels was calculated by fitting the initial linear regime of stress-strain curve and reported as mean and standard deviation ($n = 3$).

Compressive test

The compressive stress-strain measurements were performed on water-swollen gels using a universal tensile-compressive tester equipped (Model 5576, Instron Instruments, U.S.A.) with a 30kN load cell. The cylindrical gel sample of 2.2 mm diameter and 25mm thickness was set on the lower plate and compressed by upper plate, which was connected to the 30KN load cel, at a speed of 1mm/min.