



# Fabrication of mechanically tough and self-recoverable nanocomposite hydrogels from polyacrylamide grafted cellulose nanocrystal and poly (acrylic acid)



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## ABSTRACT

We present a facile strategy for the fabrication of mechanically tough and self-recoverable nanocomposite hydrogels reinforced by surface-modified cellulose nanocrystals. Polyacrylamide grafted cellulose nanocrystal (CNC-g-PAM) was first synthesized by ceric salt initiated surface graft polymerization of acrylamide onto CNC, then incorporated into chemically crosslinked poly(acrylic acid) (PAA) networks to obtain dual-crosslinked CNC-g-PAM/PAA nanocomposite hydrogels. CNC-g-PAM acted as both interfacial nanofillers and physical crosslinkers through reversible hydrogen bonds between PAA and PAM on the surface of CNC. FTIR analysis confirmed the formation of above hydrogen bonds. Scanning electron microscopy observations revealed good interfacial compatibility between CNC and PAA matrix. The nanocomposite hydrogels exhibited decreasing swelling ratio with increasing CNC-g-PAM content. Uniaxial tensile tests and tensile loading-unloading tests showed that elastic modulus, breaking strength and elongation at break of the nanocomposite hydrogels were significantly increased compared to PAA hydrogel, and that the nanocomposite hydrogels exhibited good self-recovery ability after large deformation.

## 1. Introduction

Conventional synthetic hydrogels usually do not have enough mechanical strength to meet the requirements of practical industrial and biomedical applications, due to the intrinsic structural inhomogeneity and lacking effective energy dissipation mechanism (Gong, 2010; Zhao, 2014). To broaden the applications of hydrogels, many efforts have been devoted by designing distinctive structure or introducing energy dissipation mechanism to fabricate tough hydrogels, such as slide-ring hydrogels (Okumura & Ito, 2001), tetra-arm PEG hydrogels (Sakai et al., 2008), double-network hydrogels (Gong, Katsuyama, Kurokawa, & Osada, 2003), nanocomposite hydrogels (Haraguchi & Takehisa, 2002), macromolecular microsphere composite hydrogels (Huang et al., 2007), and so on. However, most of these hydrogels need sophisticated synthesis procedures. In contrast, nanocomposite hydrogels are relatively simpler to fabricate. Various nanoparticles, e.g. clay (Huang et al., 2007), silica (Zhong et al., 2015) and graphene oxide (Liu et al., 2012) have been employed to fabricate mechanically tough nanocomposite hydrogels.

Cellulose nanocrystals (CNCs) are rod-like nanoparticles obtained by acid hydrolysis of cellulose fibers, usually have dimensions of

5–20 nm in diameter and 100–1000 nm in length, depending on the source of cellulose and the acid hydrolysis conditions (Beck-Candanedo, Roman, & Gray, 2005). CNCs are attractive as reinforcing fillers due to their renewability, high surface area, ultrahigh elastic modulus and tunable surface chemistry (Habibi, Lucia, & Rojas, 2010). Particularly, considering the good hydrophilicity and biocompatibility, CNCs have been widely applied as nanofillers to fabricate nanocomposite hydrogels. Just recently, the preparation, properties, and applications of hydrogels incorporating CNCs were summarized by De France, Hoare, and Cranston (2017). By straightforward physical incorporation of CNCs as filler/reinforcing agents within various polymeric hydrogel networks, the mechanical strength could be enhanced but the enhancement were usually moderate due to the weak physical interactions between CNCs and hydrogel matrix. By improving the physical interactions between CNCs and hydrogel matrix (De France et al., 2017) or introducing covalent bonds between CNCs and hydrogel matrix (Yang et al., 2015; Yang, Han, Duan, Ma et al., 2013), mechanically tough and elastic CNCs reinforced nanocomposite hydrogels could be obtained by improving the physical interactions between CNCs and hydrogel matrix (De France, Chan, Cranston, & Hoare, 2016)

Except for ultrahigh strength, good self-recovery property of a

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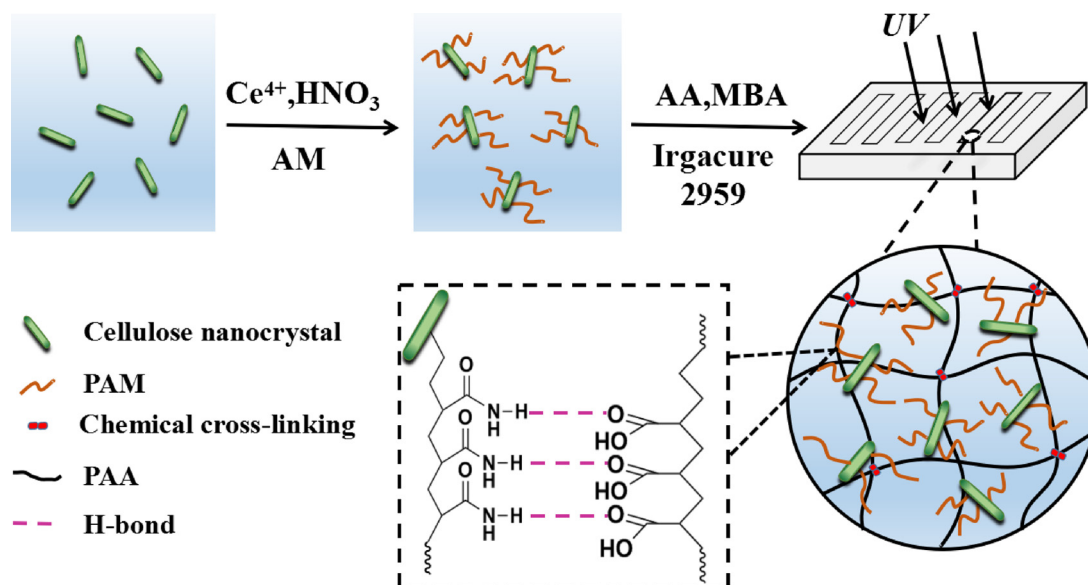


Fig. 1. Schematic illustration of fabrication process and network microscopic structure of CNC-g-PAM/PAA nanocomposite hydrogels.

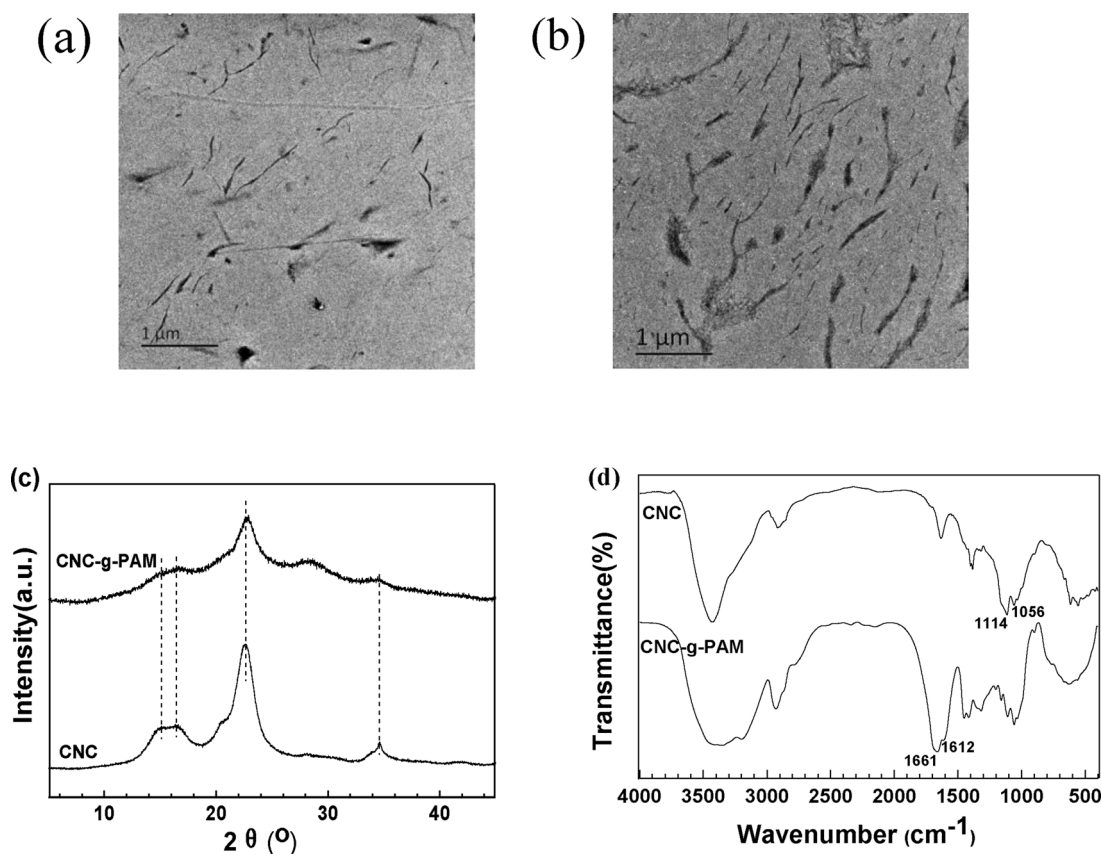


Fig. 2. Characterization of CNC and CNC-g-PAM: TEM images of CNC (a) and CNC-g-PAM (b), X-ray diffraction patterns of CNC and CNC-g-PAM (c), FT-IR spectra of CNC and CNC-g-PAM (d).

hydrogel at room temperature is also important for fatigue resistance and extending its service life. To address this problem, the reversible crosslinking mechanism has been used to fabricate hybrid crosslinked hydrogels (Lin, Ma, Wang, & Zhou, 2015) or dual physically crosslinked hydrogels (Hu et al., 2016). The reversible physical crosslinkings commonly used in hydrogels include ionic interactions (Hu et al., 2016; Lin et al., 2015), hydrogen bonds (Hu et al., 2016) and others, which allowed hydrogels to recover after large deformation. Recently, several

CNC-reinforced nanocomposite hydrogels with high strength and self-recovery ability were fabricated by introducing reversible physical crosslinkings between CNCs and hydrogel matrix. For instance, Yang, Han, Zhang, Xu, and Sun (2014) reported dual-crosslinked nanocomposite hydrogels prepared by introducing TEMPO oxidized CNCs into chemically crosslinked poly(acrylamide) (PAM) networks. TEMPO oxidized CNCs acted as both nanofillers and physical crosslinkers through hydrogen bonds between CNCs and PAM matrix. The covalent

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