RESEARCH ARTICLE

A new method for synthesis of modified cellulose-graft-poly(acrylic acid) copolymer using 4-cyano-4-[(phenylcarbothioyl)solfanyl]pentanoic acid to serve as RAFT agent

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Abstract

In this study, cellulose was first treated with 4-cyano-4-((phenyl carbonothioyl)solfanyl) pentanoic acid to serve as the reversible addition-fragmentation chain transfer polymerization (RAFT) agent, then the controlled grafting polymerization of acrylic acid was successfully performed. A well-defined, cellulose-graftacrylic acid copolymer (Cell-g-PAA), has been prepared by RAFT polymerization technique using three different approaches: RAFT agent was prepared by substitution of dithiobenzoate magnesium bromide with 4,4'-azobis(4-cyanopentanoic acid) in ethyl acetate as a solvent, mediated cellulose (Cell) block as the macromolecular Cell-RAFT agent and (cellulose-co-acrylic acid) copolymer with alternating sequence. The resulting (Cell-RAFT) for "living" free radical polymerization was then heated in the adjacent acrylic acid monomer for the development of the controlled graft copolymer onto cellulose. The structures of the intermediate, graft copolymer were investigated by FT-IR, DSC, ¹H NMR, scanning electron microscopy, and thermo gravimetric. The results demonstrate that the preparation of graft copolymers was successfully confirmed. This approach would provide an extensive classification of molecular designs to obtain modern types of tailored hybrid materials derived from natural polysaccharides and synthetic polymers. Also, using a macro-initiator is an excellent method for synthesizing new materials.

KEYWORDS

acrylic acid, cellulose, graft copolymers, polymerization, reversible addition fragmentation chain transfer polymerization

1 | INTRODUCTION

Cellulose is the most abundant natural bio-polymer that has many remarkable physical and chemical properties and a main role in the paper industry. These roles can be summarized in the following approaches: coatings, membranes, films, drilling approaches, building materials, foodstuffs and pharmaceutics. ^{1,2} Since the discovery and introduction of

cellulose (Cell) in 1838 by Anselme Payne,³ graft of polymerization on cellulose improves physical properties, since the presentation of side chains leads to different structural characteristic in the native material. In this method natural cellulose based on materials can be achieved, that is, with mechanical properties formed compared with the conventional cellulose.⁴ Nevertheless, the poor insolubility and compatibility of cellulose in water and also in the most

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unusual organic solvents are derived from its extraordinary structure. This event states that chemical and/or physical structure modification of cellulose is essential before it can be employed. Cellulose modification and grafting it with other molecules or characteristic functional groups on its structure approves the material to have more varied properties, creating the way for many developed applications. The preparation of a cellulose-grafted polymer is an impressive way for modifying cellulose's properties and combining the benefits of synthetic polymers and natural cellulose. ^{5–9} Grafting copolymerization of cellulose and its derivatives could be commonly categorized into three main groups: (a) ionic and ring opening polymerization, (b) free radical polymerization, and (c) controlled/living radical polymerization (CRP). ²

Although the graft copolymerization of different monomers onto cellulose and materials derived from it has been discussed by various traditional polymerization approaches, such as ultraviolet light, ¹⁰ gamma rays, ¹¹ plasma ion beams, ¹² and ceric(IV) ion initiation ¹³ methods, the un- controlled chain scission causes it impossible to predetermine the length of graft chains on the cellulose backbone. Accordingly, the synchronous structure of homo- polymers and copolymers dominates the grafting copolymerization. It was demonstrated that grafting vinyl monomers from a mixture of natural and synthetic polymers is a desirable solution to prevailing the drawbacks mentioned above.

The CRP approaches, such as atom transfer radical polymerization (ATRP),¹⁴ singleelectron-transfer living radical polymerization (SET-LRP),^{15,16} Nitroxide mediated polymerization (NMP),¹⁷ and reversible addition-fragmentation chain transfer (RAFT) polymerization,^{18–21} have been applied to the graft of cellulose. These approaches represent the tailoring of the properties of the cellulose derived from grafting copolymers by tuning the synthetic graft length, the architecture, and also the chemical formation of the product.⁸ Among these approaches, RAFT polymerization might be carried out in more favorable mediums and easily represent his application. Furthermore, there are no metal pollutants in the resulting polymer. So far there has been various evidence detailing the successful synthesis of grafting copolymers by RAFT approach.

RAFT polymerization is another favorite living radical approach for preparing graft polymers with well-defined structures. The main aims of the RAFT method, compared with other living radical polymerization methods, include: the ability of polymerizing, in a controlled method, a great variety of vinyl monomers and the less-demanding reaction situations that are involved (Scheme 1). The other polymerization variables, such as the operating pressure or the temperature are constant, as long as polymerization in homogeneous media is concerned. The choice and exhibition of the reversible chain transfer agent is an essential factor. ^{26–28}

In this contribution, a novel method for the synthesis of modified Cell-g-PAA copolymer using the RAFT agent was examined.^{29,30} For this purpose, an RAFT agent was prepared by substitution of dithiobenzoate magnesium bromide with 4,4'-azobis(4-cyanopentanoic acid) in ethyl acetate. Afterward, cellulose and acrylic acid were copolymerized by RAFT polymerization approach to achieve Cellg-PAA graft copolymer at 75 °C. Acrylic acid (AA) was applied as the monomer for the grafting polymerization. Since the corresponding copolymer will have the stimulant response and pH-sensitiveness, it might be used as a degradable matrix for the application of drug delivery. Competitive removal of heavy ions from polluted water, adsorption of protein from human plasma, anti-salt superabsorbent, hydrogels, and agricultural soil are among the applications of Cell-q-PAA copolymer. 31-34 The structures of the copolymer and other compounds were discussed using ¹H NMR, FTIR, and SEM methods, and their thermal stability was investigated using DSC and TGA analyses.

2 | MATERIALS AND METHODS

2.1 | Material

Microcrystalline cellulose with a degree of polymerization (DP) 200 was bought from Trade Company (China) and Beijing Fengli Jinggiu Commerce, then dried in a vacuum at 50 °C for 24 h prior to use. RFAT agent was prepared in our laboratory. 2,2-Azobisisobutyronitrile (AIBN; Switzerland, Fluka) as an initiator was re-crystallized from ethanol at 50 °C prior to use. Toluene (Merck), tetrahydrofuran (THF) and dimethylformamide (DMF) after reflexing over sodium, filtered and purified by distillation under argon gas. Carbon disulfide and ethyl acetate were dried by magnesium sulfate and distilled at low pressure. Acrylic acid as a monomer was dried with sodium sulfate and calcium hydride then, distilled at low pressure. N,N-dicyclohexylcarbodiimide (DCC, 99%, Aldrich), 4-dimethylaminopyridine (DMAP, 99%, Aldrich), methanol (99%, Merck) was utilized in the synthesizing of functional cellulose. The rest of the materials were acquired from Merck Company and then were purified according to the standard method.

2.2 | Characterization methods

Fourier transform infrared (FT-IR) spectra of the specimens were collected on Shimadzu FTIR-8400S model between the frequency ranges of 4000–400 cm⁻¹, with an attenuated general reflections facility. The specimens were synthesized by grinding the dry powders with potassium bromide (KBr) and, then compressed the mixture into disks. The spectrum was recorded at room temperature. The ratio of acrylic acid and cellulose of the (Cell-b-PAA) was

Reinitiation

$$R^{0} \xrightarrow{M} R - M^{0} \xrightarrow{M} Pm^{0}$$

Chain equilibration / propagation

Terminitation

$$Pn^0 + Pm^0 \longrightarrow dead polymer$$

SCHEME 1 The general mechanism of RAFT.

characterized by ¹H NMR (Varian INOVA-500), using CDCl₃ as the solvent. The size distribution and morphology of the particles were achieved by the use of a scanning electron microscope (FE-SEM) type LEO 1430 VP. The thermogravimetric analysis (TGA) was performed using the device TGA/DSC (Pyris-1, Perkin-Elmer, at a heating rate of 10 °C/min in flowing high purity nitrogen gas with 20 mL/min).

2.3 | Pretreatment of cellulose

To break down the vast hydrogen grafting between the hydroxyl groups of cellulose and to open up the ordered spaces, firstly, cellulose (2.0 g, DP = 200) was washed with ethanol, following that plunged into an aqueous solution of NaOH (150 mL; 10 wt%). Following that, the mixture was stirred for about 24 h at 120 °C. After 24 h, the swollen cellulose specimen was frequently washed with ice ethanol until a neutral solution was afforded. After addition of the specimen into 200 mL dichloromethane, stirred for about 2 h, and then filtered. This cellulose specimen was immediately utilized for the termination step (esterification with RAFT agent) without any drying.

2.4 Preparation of (4-cyano-4-((phenylcarbonothioyl)solfanyl)pentanoic acid) cellulose (Cell-RAFT agent)

A RAFT agent was synthesized in the laboratory according to a previous work (Scheme 2). $^{22-25}$ A 50 mL round-bottom flask was plunged with Cell (0.02 g) dissolved in DCM (10 mL), (0.370 g, 1.0 mmol), DCC (0.4 g, 1.94 mmol), and DMAP (0.26 g, 2.13 mmol). The reaction was sealed and placed in a pre-heated oil bath at 50 $^{\circ}$ C. The reaction was left to run for 6 days. The reaction mixture was then precipitated with cool methanol, dissolved in chloroform, and precipitated in methanol again. This cycle was repeated several times to achieve a pure product of (Cell-RAFT agent).

2.5 | Synthesis of graft copolymers

A mixture of Cell–RAFT agent (0.0568 g) and dry DMF (10 mL) was stirred magnetically under an argon gasses. After dissolving completely, AIBN (0.0042 g, 0.04 mmol) and AA (2.0 g) were added.

The polymerization continued at 75 °C for about 8 h at the end of this time, the reaction mixture was precipitated

SCHEME 2 Synthesis route of RAFT agent.

in tenfold benzene, and then filtered. The crude copolymer was dried in a vacuum oven at 60 °C. Scheme 3 summarizes the preparation of graft copolymers.

The Cell-g-PAA specimens were extracted with acetone for 3 h in a Soxhlet extraction instrument, purified and dried in a vacuum for 24 h.

3 | RESULTS AND DISCUSSION

3.1 Characterization of the RAFT agent, Cell-RAFT agent, and Cell-g-PAA

The FTIR spectrum of neat cellulose, Cell-RAFT agent, and Cell-g-PAA are seen in Figure 1. The FTIR spectrum of neat cellulose Figure 1a, represents the characteristic absorption peaks owing to the stretch vibration of C—O at 1043 cm⁻¹, C—H stretch vibration at 2885 cm⁻¹, the asymmetric wagging and bending of the —CH₂ shows at 1500 cm⁻¹, and the rocking vibration of the —C—H at 897 cm⁻¹. In addition, the broad peak centered at 3445 cm⁻¹ corresponds to the stretch vibration of hydroxyl groups. The infrared spectrum of the Cell-RAFT agent Figure 1b represents a very strong band for the C=O group at 1780 cm⁻¹, which proves the successful formation of the cellulose ester. The characteristic absorption is owing to the stretch vibration of the

C—N group (2246 cm⁻¹), the C—S group stretch vibration (720 cm⁻¹), the C—S group stretch vibration (1087 cm⁻¹), and the stretch vibration of the OH group (3448 cm⁻¹). The C=C stretch bands for the aromatic ring are apparent at 1438–1625 cm⁻¹. Cell–RAFT agent (Figure 2b) demonstrated stronger absorption at 2800–3000 cm⁻¹ for C—H than neat cellulose (Figure 2a). Cell-g-PAA (Figure 1c), the characteristic bands at 3100–3345 cm⁻¹ should be related to the absorption of O—H of the carboxyl group and the characteristic peak was increased at 1775 cm⁻¹ related to the carbonyl stretch band (C=O), compared to Cell–RAFT agent (Figure 1(b)). Also, C=C aromatic ring represented at 1620 cm⁻¹. These documents further indicated the successful preparation of the graft copolymer See Supporting Information.

The grafting percentage (*G*) indicates the increase in weight of original cellulose subjected to grafting with an AA and is calculated generally by the following equation and it was 13%.

% grafting (G) =
$$\frac{m_1}{m_2} \times 100$$

where m_1 is the weight of poly (acrylic acid) grafted onto cellulose (in g) and m_2 is the weight of the original cellulose (in g).

SCHEME 3 Synthesis of Cell-g-PAA copolymer.

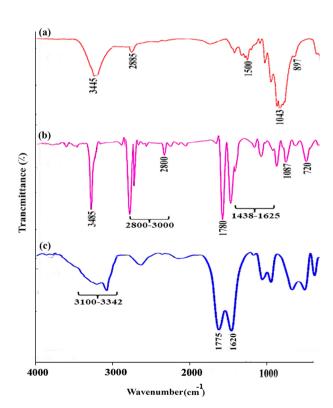


FIGURE 1 The FTIR spectra of neat cellulose (a) Cell-RAFT agent (b) and Cell-g- PAA(c).

Figure 2 represents the 1 H NMR spectrum of RAFT agent, Cell-RAFT agent and Cell-g-PAA. In the 1 H NMR spectrum of RAFT agent, Cell-RAFT agent and Cell-g-PAA, the resonances at about 7.98 ppm (d, 2 H, O–ArH), 7.60 (t, 2 H, m–Arh), 7.40 (t, 1 H, P-Arh) ppm), 2.70-2.00 ppm (m, 4 H, CH $_2$ CH $_2$), and 1.93 ppm (S, 3H, CH $_3$).

The thermal behaviors of neat cellulose and its Cell-g-PAA were demonstrated using DSC and TGA as shown in Figures 3 and 4. Figure 3 represents common TGA curves of weight loss as a function of temperature in the range of 50-600 °C for the cellulose and Cell-g-PAA. The TGA curve of neat cellulose (a) and AA chains connected to the backbone of cellulose, resulting in more flexibility in the formation of copolymer (b) cause to the degradation at lower temperatures. The TGA results represent a development in the thermal stability of the Cell-g-PAA copolymer compared to the neat cellulose. Thermal behavior and glass transition temperature (T_a) of the neat cellulose and Cell-g-PAA were examined using of differential scanning calorimetric analysis and results are represented in Figure 4. Figure 4a indicates an endothermic peak of nearly 40 °C attributed to the T_a of neat cellulose. In Figure 4b, thermal transition at around 20 °C is related to evaporating the solvent and the moisture content of the copolymer. T_q at 70 °C corresponds to the (Cell-g-PAA) copolymer. The $ec{\mathcal{T}_g}$ of Cell-g-PAA agent is more than the neat cellulose. The development of the

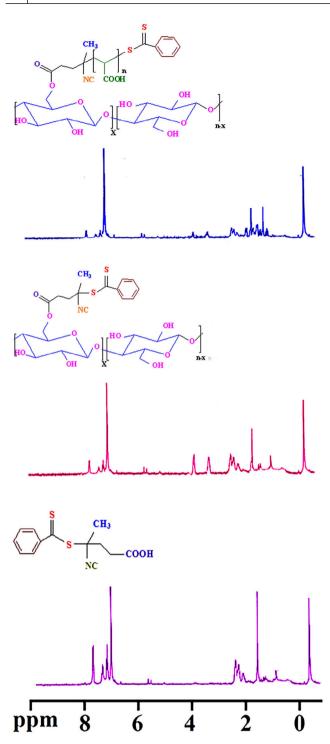


FIGURE 2 The ¹H NMR spectra of (4-cyano-4-[(phenylcarbothioyl)solfanyl]pentatonic acid) (a) Cell-RAFT agent (b), and Cell-g-PAA(c).

 T_g was winged to the polymerization degree and polymer chemical structure.

Figure 5 represents the electron micrographs of neat cellulose grafted with AA, respectively. As seen in SEM image of the neat cellulose (Figure 5a), it indicates smooth surfaces with low roughness. In contrast, after grafting of PAA to the surface morphologies of the Cell-g-PAA, the sam-

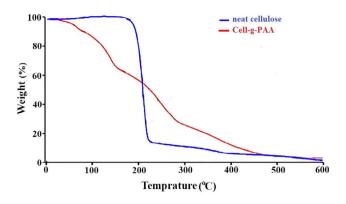


FIGURE 3 The TGA traces of the neat cellulose and Cell-g-PAA.

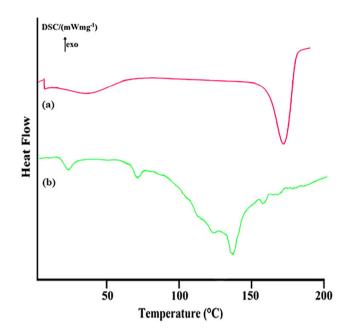


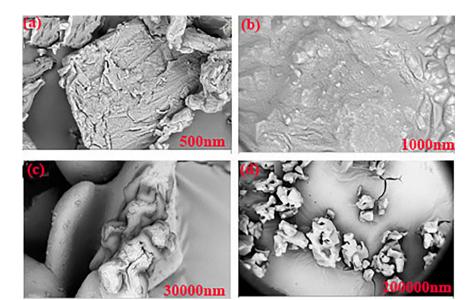
FIGURE 4 The DSC traces of (a) the neat cellulose and (b) Cell-q-PAA.

ple was changed in the topology of the grafted specimen. The increase of PAA segment onto cellulose eventuated to folded and wrinkled morphologies since the grafting occurs mostly at the cellulose surface.

4 | CONCLUSION

The main objective of this study is a simple and effective method to design polymer synthesis with well-defend structure. In summary, a new and efficient strategy for the synthesis of well-defined cellulosic copolymers modified with 4-cyano-4-((phenylcarbonothioyl)solfanyl) pentanoic acid to serve as RAFT agent and then the graft polymerization of acrylic acid was performed at 75 °C. ¹H NMR, FTIR, and SEM spectrum illustrate the successful synthesizing of the graft copolymer. TGA results demonstrate development thermal stability of the Cell-g-PAA copolymer compared to the neat cellulose. DSC results indicated the increased glass

FIGURE 5 The SEM image of (a) neat cellulose and (b–d) Cell-g-PAA with 1, 30, and 200 µm.



transmission temperature of PAA compared to neat polymers. This study provides a modern method to synthesize cellulose grafting copolymers with well-defined structures and controlled molecular weights.

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CONFLICT OF INTEREST STATEMENT

The authors declare no conflicts of interest.

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SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

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