



Synthesis and characterization of novel polyaspartic acid/urea graft copolymer with acylamino group and its scale inhibition performance



Ying Zhang^a, Hongquan Yin^{a,*}, Qingshan Zhang^a, Yunzheng Li^b, Pengjun Yao^a

^a School of Chemical Engineering and Environment, Beijing Institute of Technology, Beijing 100081, China

^b Anhui Sealong Biotechnology Co., Ltd, Bengbu 233316, China

HIGHLIGHTS

- A novel green scale inhibitor, polyaspartic acid/urea graft copolymer (PASP/Urea) with acylamino group, was synthesized.
- Inhibition efficiency against CaCO₃ is 93% when the concentration is 10 mg/L.
- Inhibition efficiency against CaSO₄ is 97% when the concentration is 4 mg/L.
- Inhibition efficiency against Ca₃(PO₄)₂ is 100% when the dosage is 12 mg/L.
- The scale crystals became irregular and distorted, when the PASP/Urea was added.

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ABSTRACT

A novel green scale inhibitor, polyaspartic acid/urea graft copolymer (PASP/Urea), was synthesized by polysuccinimide (PSI) and urea. PASP/Urea was a non-toxic, non-phosphorus, biodegradable acylamino copolymer. The structure of PASP/Urea was characterized by Fourier transform infrared spectroscopy (IR), ¹H nuclear magnetic resonance (¹H NMR) and gel permeation chromatography (GPC) analysis. Its scale inhibition performance was estimated with static scale inhibition methods. The influence of PASP/Urea on formation of CaCO₃, CaSO₄ and Ca₃(PO₄)₂ was investigated using scanning electronic microscopy (SEM). The PASP/Urea exhibited more excellent scale inhibition property against CaCO₃, CaSO₄ and Ca₃(PO₄)₂ than the PASP and the comprehensive scale inhibition property of PASP/Urea is much better than the property of PASP. The scale inhibition efficiency of PASP/Urea against CaCO₃ was 93% when the dosage of PASP/Urea was 10 mg/L. The inhibition efficiency against CaSO₄ increased to 97% in the PASP/Urea concentration of 4 mg/L. The inhibition efficiency against Ca₃(PO₄)₂ reached up to 100% in the concentration of 12 mg/L. The results of SEM indicated that the scale crystals became irregular and distorted and the formation of scales was inhibited when the PASP/Urea was added.

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1. Introduction

Nowadays, with the increasing of worldwide population, expansion of modern society and the depletion of industrial water resource, the water resource crisis is aggravated. To save water and alleviate the water resource shortage, circulating cooling water system is widely utilized in industrial processes. However, scale deposition is one of the major problems in circulating cooling water system because scale hinders heat transfer and reduces the cooling effect. So scale inhibitor is widely used in circulating cooling water system to avoid formation of scale [1–3]. Scale inhibitors mainly include natural inhibitors, phosphorous inhibitors, polycarboxylic acid inhibitors and green degradable polymer inhibitors. Natural inhibitors such as tannin, lignin and starch,

are unstable and liable to decomposition under high temperature condition and their scale inhibition performance is not prominent [4,5]. Phosphorous inhibitors such as 1-hydroxyethylidene-1,1-diphosphonic acid and 2-phosphomobutane-1,2,4-tricarboxylic acid, nourish bacteria and algae easily, which results in water pollution [6–8]. Polycarboxylic acid inhibitors such as polyacrylic acid inhibitor and polymaleic acid inhibitor, cannot be degraded into the final product (CO₂ and water), which exerts adverse effect on the environment [9–12]. Green degradable inhibitors such as polyaspartic acid and polyepoxysuccinic acid, not only possess super scale inhibition property, but also are degradable and environmentally friendly [13,14]. Because of the above disadvantages of other scale inhibitors and the enhancement of awareness of environmental protection, the production of green degradable inhibitors has become an important topic in water treatment agents.

As a typical green degradable scale inhibitor, PASP presents a good prospect because of its non-toxicity, non-phosphorus and good

* Corresponding author.

E-mail address: hqyin77@bit.edu.cn (H. Yin).

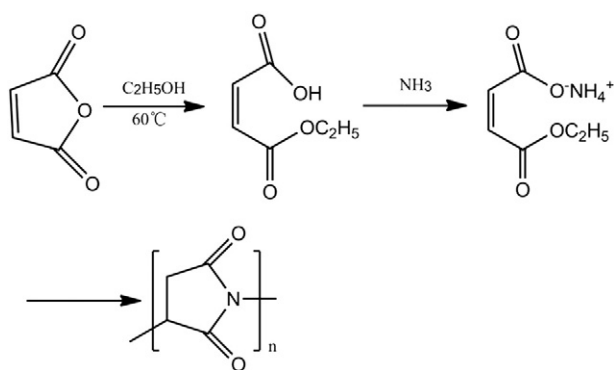


Fig. 1. Synthesis routes of PSI.

biodegradability [15–17]. Meanwhile, PASP molecule contains carboxylic group with fine chelating ability [18–25] and dispersibility [26–28], which is associated with scale inhibition property. However, PASP only possesses free carboxylic group and its comprehensive scale inhibition performance is not outstanding. In order to enhance the scale inhibition performance of PASP, it is essential to modify PASP by introducing new functional groups into the side chain of PASP. Studies on modified PASP have been carried out and several modified PASP scale inhibitors have been reported [21–23].

The carboxylic group in the polymer is the main functional group inhibiting the formation of calcium carbonate and calcium sulfate scale, while the hydroxylic group and acylamino group are the main functional groups inhibiting the formation of calcium phosphate [29]. Urea which contains acylamino group is a low-cost degradable organic compound. Therefore, to find novel biodegradable and super comprehensive scale inhibitor, we tried to introduce acylamino group to synthesize PASP/Urea graft copolymer by using urea and PSI as the reactant. PSI was produced by thermal polycondensation of maleic anhydride, ethanol and ammonia. This article presents the synthesis, structural characterization, and the scale inhibition performance of novel polyaspartic acid/urea graft copolymer with acylamino group.

2. Materials and methods

2.1. Reagents and instruments

The chemicals were used as received without further purification and were of analytical reagent grade. Maleic anhydride, ethanol,

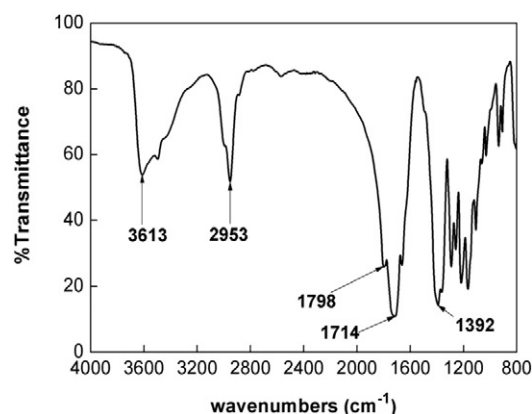


Fig. 3. IR spectrum of PSI.

ammonia, calcium chloride, sodium sulfate and sodium phosphate were purchased from Beijing Chemical Plant (China). *N*-methyl pyrrolidone, sulfolane, urea, sodium ethylate and sodium hydrate were obtained from Sinopharm Chemical Reagent Co., Ltd. (China).

The structure of PASP/Urea copolymer was characterized by IR, ¹H NMR and GPC. SEM was used to view the scales. IR spectra were recorded on a PerkinElmer FTIR spectrometer and NMR spectra were obtained on an ARX 400 MHz spectrometer. The absorbance of PO₄³⁻ was obtained on a 722 grating spectrophotometer and SEM images were performed by using a FEI Quanta FEG 250 scanning electron microscopy. GPC data were done by using a Waters 1525 gel permeation chromatography (Ultrahydrogel 250 7.8 × 300 mm column with 0.1 M NaNO₃ solution at a flow rate of 0.5 mL/min on a 2414 RI-detector).

2.2. Synthesis of polysuccinimide (PSI)

19.6 g (0.2 mol) maleic anhydride and 13.8 g (0.3 mol) ethanol were added into a three-neck round flask to produce monoethyl maleate in oil bath at 60 °C for 2.5 h under stirring and ammonia was then added to the monoethyl maleate mixture solution to gain ammonium salt. Then, *N*-methyl pyrrolidone and sulfolane were added to the solution in the three-neck flask equipped with a stirrer, a decantor and a device for injecting N₂, and the reaction mixture was condensed into polysuccinimide in oil bath at 180 °C for 6 h. Meanwhile, water brought in the reaction was removed through the decantor. Last, the resultant solution

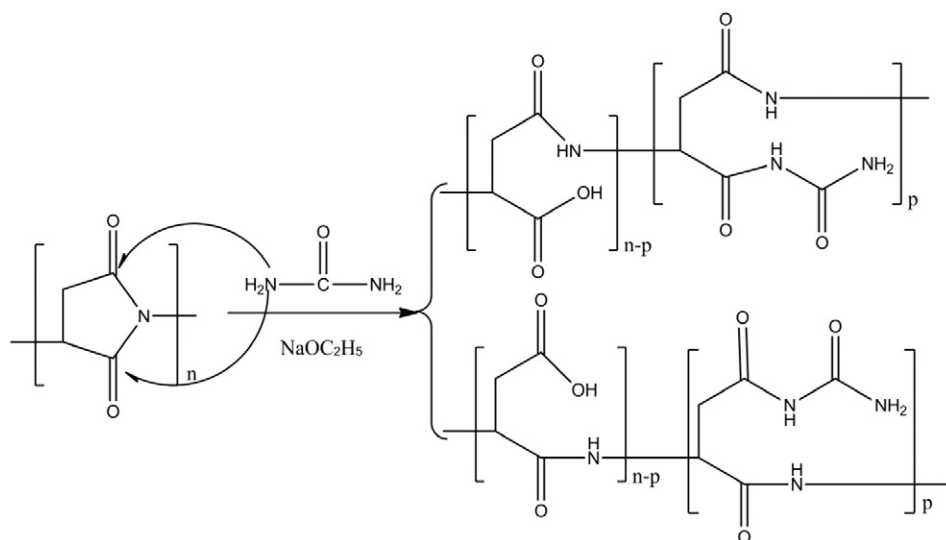


Fig. 2. Synthesis route of PASP/Urea copolymer.

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