



## Sag control of waterborne acrylic latex with regenerated nanocellulose suspension



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### ABSTRACT

Waterborne acrylic latex has been widely used for its decent performance and less emission of volatile organic compounds. In the present study, regenerated nanocellulose suspension from microcrystalline cellulose (MCC) and wood pulp toward improving waterborne polyacrylate (PA) latex sag resistance was evaluated. Rheology behavior of PA latex-regenerated nanocellulose (RC) was investigated. PA latex-RC displayed a “gel-like” behavior and had a desirable shear thinning property. Moreover, sag resistance of PA latex was evaluated by creep behavior. Results showed that deformation of PA latex at constant stress obviously decreased with an increase of the RC concentration. Meanwhile, PA latex showed high recovery of strain (> 90%) with 2% RC concentration after removal of stress, which may be related to the reinforcement from the RC entanglements and their interaction. In addition, the properties of dry PA film with RC were also evaluated. The tensile strain, tensile stress and thermal stability of the films were slightly improved, while optical transparency slightly compromised with 2% RC concentration. Overall, the results suggested that RC is a promising biomass material for improving the sag resistance of waterborne acrylic latex.

### 1. Introduction

Waterborne latexes are widely used in many industrial applications such as adhesives [1,2], coatings [3,4] and paintings [5–7]. An important requirement for waterborne latexes products is to have a prospective appearance after application. The final appearance of latex is determined by many factors. Rheology, substrate geometry and process parameters play complex and highly interdependent roles in this development, and failure to appropriately control these factors can have dramatic consequences on the ultimate latexes appearance [8]. A defect such as sag is an example of the possible consequences of poor latexes control.

Sag is a common phenomenon of latexes characterized by gravity-driven flow after deposition [8,9]. In practice, all processes that utilize liquid applied latexes on non-horizontal surfaces are susceptible to sag and excessive amount of sag can lead to final product defects [8,10], such as drips, in the final dried film [11]. It is well established in literature that the sag resistance ability of latex is governed by its rheological properties, surface tension and gravity [10]. Controlling the rheology properties of latex during processing is critically important for

sag resistance. Conventionally, cellulosic and associative thickeners are the most widely used rheology modifiers in waterborne latex to tune the in-can stability and flow-levelling and sag resistance [10,12,13].

Nanocellulose is derived from cellulose, a renewable, biodegradable and the most abundant biopolymer on the earth [14,15]. Nanocellulose has already been reported widely as reinforcing components for waterborne latex [16–19]. Using nanocellulose is attractive because of their beneficial properties: excellent mechanical performance at low weight, biocompatible, transparent and reactive due to the hydroxyl groups on their large fiber surfaces [16,20–23]. Apart from the mechanical performance of nanocellulose, rheological properties were also widely studied and nanocellulose have been used as rheology modifier in waterborne latex [24–27]. Ruiz et al. [28] described a waterborne epoxy polymer emulsion with cellulose nanocrystal (CNC) designated for coatings. The CNC significantly increased the viscosity compared to the neat epoxy emulsion at low shear rates. At higher shear rates (100/ s) the viscosity was nearly the same for the neat polymer emulsion. Franziska et al. [26] prepared acrylate coating containing nanofibril cellulose (NFC). The viscosity of acrylate-NFC suspension was precisely adjustable and predictable in the considered shear range by NFC

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addition. In comparison with other nanocellulose products, regenerated cellulose is a promising nanocellulose material [29,30] with high yield (> 80%) and reasonable production cost. Besides, the rheology properties of RC have been studied in the pioneering works by Jia et al. [31], such as high zero-shear viscosity, marked shear thinning properties at higher shear rates and significant viscoelasticity.

In this work, RC was used as a rheology modifier to obtain good sag resistance of waterborne PA latex. Creep recovery of PA latex with RC was tested and the results showed that the deformation of PA latex can be efficiently reduced and deformation had high recovery ability. In addition, the properties of PA latex films with RC were also evaluated. The tensile strain, tensile stress and thermal stability were either slightly improved or remained as good upon incorporation of RC, while the optical transparency was slightly impaired. All results showed that RC could be a potential biomass material for improving the sag resistance of waterborne acrylic latex.

## 2. Materials and methods

### 2.1. Materials

Microcrystalline cellulose (MCC, particle size of 25  $\mu\text{m}$ ) was purchased from Macklin biochemical Co., Ltd. Wood Pulp was obtained from Xinxiang Natural chemical Co., Ltd. 85 wt% Phosphoric acid ( $\text{H}_3\text{PO}_4$ ), Butyl acrylate (BA), Methyl methacrylate (MMA) and Styrene (ST), Sodium dodecylbenzenesulfonate (SDBS), Potassium persulfate (KPS), Sodium bicarbonate ( $\text{NaHCO}_3$ ), Ammonia solution ( $\text{NH}_3\cdot\text{H}_2\text{O}$ ) were supplied by Sinopharm Chemical Reagent Co. Ltd., China. Acetoacetoxyethyl methacrylate (AAEM) was purchased from 9 Ding Chemistry Co. Ltd. The chemicals above were all of chemically pure grade. Heterogeneous 13 alcohol polyoxyethylene ether (1309) was provided by the Mobil Reagent Co. Ltd. The emulsifier mixture was composed of SDBS as anionic emulsifier and 1309 as nonionic emulsifier (weight ratio 2:1). KPS is as initiator.  $\text{NaHCO}_3$  is as buffering reagent.  $\text{NH}_3\cdot\text{H}_2\text{O}$  is as pH regulator. All chemicals were used as received and the aqueous solutions were prepared using deionized water.

### 2.2. Preparation of regenerated cellulose suspension

RC suspension was prepared according to our previous report [29,32]. Generally, 2 g of MCC or wood pulp was wetted with 6 mL of deionized water and mixed with 100 mL and 200 mL respectively of 85% aqueous phosphoric acid to reach a homogenous state at 0 °C for 24 h. Then, the cellulose solution was added to 500 mL of deionized water to obtain a milky dispersion, which was centrifuged at 12,000 rpm (Thermo, USA) until a constant pH was obtained. The precipitated RC was collected and then treated with a high pressure homogenizer (APV2000, Germany) at 100 bar. The final solid content of RC was 3% (w/w). The stability of MCC, wood pulp and regenerated cellulose suspension was visually checked after storage for 7 days at room temperature.

### 2.3. Synthesis of PA latex

The PA latex was synthesized as a binder in textile pigment in our laboratory [33].

The pre-emulsification of seed was prepared as followed: Deionized water (100 g), compound emulsifier (1.35 g), and  $\text{NaHCO}_3$  buffering agent (0.3 wt.%), followed by the addition of 10 g BA monomer, high-speed dispersion for 20 min (using a homogenizer at 10,000 rpm, in an ice bath). The pre-emulsification of core was prepared as followed: Deionized water (30 g), compound emulsifier (0.9 g), followed by the addition of 35 g BA monomer, 11.25 g MMA monomer, and 3.75 ST monomer, high-speed dispersion for 20 min (using a homogenizer at 10,000 rpm, in an ice bath). The pre-emulsification of shell was prepared as followed: Deionized water (30 g), compound emulsifier

(0.45 g), followed by the addition of 15 g BA monomer, 10 g MMA monomer, 5 ST monomer, and 5.23 g AAEM monomer, high-speed dispersion for 20 min (using a homogenizer at 10,000 rpm, in an ice bath).

The pre-emulsion of the seed was added to the four-necked flask, after mixing in nitrogen at  $75 \pm 2$  °C, adding 0.15 g of the potassium per-sulfate (KPS) as the initiators. When the appearance of the emulsion changed from gray to slightly bluish, the remaining core monomer mixture and the aqueous solution of initiators (0.15 g of the KPS dissolved in 15 g deionized water) were added dropwise into the stirred reactor at 48 mL/h and 10 mL/h, respectively. After the addition was completed, the reaction was maintained for 2–3 h at  $75 \pm 2$  °C, the stirring rate was controlled at 200 rpm.

The pre-emulsion of shell monomer and the aqueous solution of initiators (0.15 g KPS dissolved in 15 g deionized water) were added to the core emulsion for about 1.5–2 h and 0.5 h, respectively. When the shell emulsion remained 1/3 of the total volume, AAEM was added as a crosslinking comonomer. The emulsion polymerization was carried out at  $78 \pm 2$  °C for 2–3 h in a nitrogen atmosphere after all of the monomers were added completely. At last, the synthesized emulsion was cooled to 40 °C, and then 30% ammonia solution was dropped into the synthesized emulsion to control the pH value of the final latex in the range of 8–9. The solid content of PA latex was 30% (w/w).

### 2.4. Preparation of PA latex–RC suspensions

For all suspensions, a constant PA latex solid content of 15% (w/w) was used. RC concentration was varied from 0.1 over 0.3 to 0.75% (w/w) in the suspension to achieve concentration of 1, 2, and 5% of dry RC referred to solid polymer. The neat PA latex film and composite were prepared by simply casting at room temperature until complete evaporation of water.

### 2.5. Rheological measurements

Rheology analyses were performed on a ThermoHaake-Mars 60 rheometer with parallel plate (35 mm in diameter). Creep recovery tests for PA latex-RC suspension were carried out by subjecting the samples to a constant stress (1 Pa, below the yield stress) for 3 min followed by 5 min of recovery by decreasing the stress to zero. The frequency sweeps (0.1–10 Hz) were done to study the viscoelastic parameters at a fixed strain of 1%. All these rheological measurements were carried out at 25 °C.

### 2.6. TEM

The morphology of RC was characterized using transmission electron microscopy (TEM, JEM-2100, JEOL, Japan). Samples were prepared by dropping 30  $\mu\text{L}$  of an aqueous RC dispersion (RC content 0.1 mg/mL) onto a carbon support copper grid. The sample was subsequently dried in an oven at 50 °C for 2 h. After drying, the samples were observed at 200 kV.

### 2.7. DP determination

Degree of polymerization (DP) of original MCC, wood pulp and regenerated celluloses from MCC or wood pulp was determined by measuring viscosity of cellulose solution in cupric ethylenediamine solution according to ISO 5351 standard [34]. Each measurement was repeated for three times and the averaged data was reported.

### 2.8. Tensile properties

Tensile properties of the neat PA latex film and composite materials were measured using a universal mechanical testing machine (H5K-S, Hounsfield). The specimen gage length was 30 mm and the testing

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