



# Synthesis and application of cationic spherical polyelectrolyte brushes as retention and drainage aid in bleached eucalyptus kraft pulp



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## ARTICLE INFO

### Article history:

Received 15 May 2015

Received in revised form 27 June 2015

Accepted 5 July 2015

Available online 13 July 2015

### Keywords:

Cationic spherical polyelectrolyte brushes

Flocculation

Retention

Drainage

Mechanism

Papermaking

## ABSTRACT

In the present study, a series of cationic spherical polyelectrolyte brushes (CSPB), consisting of a silica core and a shell of copolymer of acrylamide (AM) and [2-(methacryloyloxy)ethyl] trimethylammonium chloride (METAC), were synthesized. Then a dual-component system which was composed of CSPB and anionic polyacrylamide (APAM) was used to improve the retention and drainage properties of bleached eucalyptus kraft pulp and precipitated calcium carbonate (PCC). Comparative researches about retention and drainage properties between CSPB/APAM system and cationic starch/APAM system were undertaken as well. Results showed that further improvement in first-pass retention (FPR) of pulp, FPR of PCC and drainage time could be achieved by CSPB/APAM system. It was also found that increase of shear intensity led to a decrease in retention efficiency, while it had no significant influence on drainage time of pulp. However, due to the symmetrical or quasi-symmetrical spherical brush structure, CSPB still showed better retention efficiency than cationic starch under different turbulent conditions. Furthermore, the flocculation mechanism of the CSPB/APAM dual-component system was proposed in the paper.

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## Introduction

In order to retain fiber fines and fillers, reduce a treatment load on wastewater and improve runnability of paper machine, a variety of retention and drainage aids have been developed [1–3]. Cationic polymers, such as cationic polyacrylamide (CPAM), poly (2-methacryloyloxyethyl-trimethylammonium chloride) (PME-TAC), poly (diallyldimethylammonium chloride) (PDADMAC) and cationic starch, are one of the most commonly used wet-end additives in papermaking. These cationic polymers can interact with fibers and fillers, flocculating pulp slurry [4]. However, little improvement in retention and drainage is found when cationic additives are used alone. Hence, a series of dual-component systems are developed to further improve the retention and drainage efficiency [5–7]. A typical dual retention and drainage system usually consists of cationic components with high charge density (e.g., CPAM, PMETAC and cationic starch) and anionic components with low charge density

[e.g., anionic polyacrylamide (APAM)]. The cationic components are first adsorbed onto the substrate surface to form patches, followed by the bridging formation of anionic components between these cationic patches [8–10]. Significant improvement in retention and drainage can be achieved by conjugation of cationic and anionic components.

However, most additives used in wet-end of papermaking are linear polymers. As the operating speed of papermaking machines increases constantly, there is an obvious disadvantage in shear resistance for the systems based on traditional linear polymers, which is adverse to the improvement of production efficiency of papermaking [11,12]. To meet the needs of high-speed papermaking, we develop a kind of cationic retention and drainage aid, namely cationic spherical polyelectrolyte brushes (CSPB), which consists of a nanosilica core and a shell of copolymer of acrylamide (AM) and [2-(methacryloyloxy)ethyl] trimethylammonium chloride (METAC). The symmetrical or quasi-symmetrical spherical brush structure of CSPB makes it less sensitive to shear. Thus better retention efficiency can be obtained, especially when CSPB is combined with a second anionic additive.

In this paper, we first describe the preparation and characterization of CSPB. Then flocculation, retention and drainage of pulp

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suspension by CSPB/APAM dual-component system are examined. The flocculation mechanism of CSPB/APAM dual-component system is also discussed.

## Experimental

### Materials

Silica nanoparticles with an average particle size of 90 nm were obtained from Chosen-chem New Material Technology Co., Ltd., (Jiangsu, China) and used as received. Toluene, methanol, triethylamine, acetone, ethylenediaminetetraacetic acid (EDTA), triethanolamine, Eriochrome Black T (EBT), HCl, ammonia water and acrylamide (AM) were purchased from Sinopharm Chemical Reagent Co., Ltd., (Shanghai, China). Toluene was refluxed over sodium and distilled prior to use. [2-(methacryloyloxy)ethyl] trimethylammonium chloride (METAC, 75 wt% in water) from Aladdin Reagent Co., Ltd., (Shanghai, China), was precipitated in acetone and washed several times with acetone. All other reagents could be used without further purification. The azo initiator with monochlorosilane end-group was prepared according to literatures [13,14].

Precipitated calcium carbonate (PCC) with an average particle size of 2.41  $\mu\text{m}$  and specific surface area of 11  $\text{m}^2/\text{g}$  was provided by Yueyang Forest and Paper Co., Ltd., (Hunan, China). Anionic polyacrylamide (APAM) and cationic starch with degree of substitution of 2% were obtained from Maxleaf Paper Co., Ltd., (Hubei, China). The cationic starch was thermally gelatinized for 20 min under stirring and cooled to room temperature.

In this experiment, bleached eucalyptus kraft pulp was used. The pulp suspension was prepared by mixing bleached fibers (35°SR) with 20 wt% PCC. Then the concentration of this pulp suspension was adjusted to 0.2 wt%.

### Synthesis of CSPB

The synthesis of CSPB is divided into two steps as described in Scheme 1. The azo initiator was firstly immobilized on the surface of silica through coupling reaction of monochlorosilane end-group of the initiator with surface hydroxyl groups. 0.83 g of azo initiator and 4 ml triethylamine were added into 100 ml dry toluene containing 0.45 g of silica. The mixture was stirred overnight at room temperature. The products were centrifuged and washed three times with toluene and methanol respectively, and then dried overnight under vacuum.

Then CSPB was synthesized through surface-initiated polymerization. The above azo initiator-immobilized silica was dispersed in 10 ml distilled water, followed by the addition of different amount of AM and METAC. And the system was carefully degassed to remove the oxygen and polymerized at 60 °C for 6 h under nitrogen atmosphere. The products were then purified and dried in vacuum at 60 °C for 12 h. The pertinent parameters of different CSPB were summarized in Table 1.

**Table 1**  
The molecular parameters of CSPB.

Samples	Molecular weight (g/mol)	Charge density $\mu\text{eq/g}$
CSPB-1	207,200	252.5
CSPB-2	291,200	260
CSPB-3	289,700	427.5

### Structure of CSPB

The structure of CSPB was characterized by Fourier-transform infrared spectroscopy (FTIR, Nicolet AVATAR 360, USA). The FTIR spectrum was recorded in the 4000  $\text{cm}^{-1}$ –500  $\text{cm}^{-1}$  region using KBr pellet technique.

### Molecular weight of brushes

The molecular weight of brushes and their distribution were measured by gel permeation chromatography (GPC, Spectra SERIES P100, USA).

### Determination of charge density

The charge density of samples was determined via colloid titration using a Particle Charge Detector (Mütek PCD 03, Germany). All the samples were titrated with 0.001 M sodium-polyethylenesulphate (PES-Na) to the end point.

### Characterization of morphology

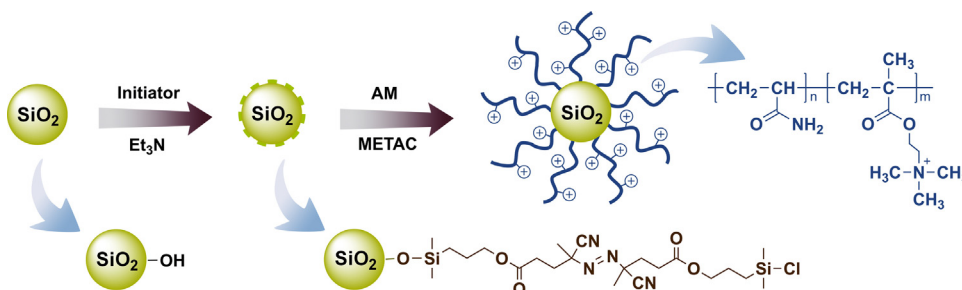
The morphology of samples was observed by transmission electron microscopy (TEM, JEM-2100, Japan) and field emission scanning electron microscopy (FESEM, Sigma, Germany).

### Determination of zeta potential

The zeta potential of pulp suspension before and after addition of additives was analyzed by System Zeta Potential Tester (Mütek SZP 06, Germany) at 25 °C.

### Flocculation of pulp suspension

The flocculation of pulp suspension was determined as follow. Required amount of CSPB was firstly added into a tube containing 20 ml pulp suspension, and the mixture was immediately shaken for 5 s. 0.03 wt% of APAM was added 2 min later. Then the system was allowed to keep for 30 min at room temperature. 4 ml of the sample was carefully collected from the top of pulp suspension, and transmittance of samples was measured using a UV-vis spectrometer (Shimadzu UV-3600, Japan) at 550 nm [10]. The relative turbidity,  $\tau/\tau_0$  was used to evaluate the flocculation ability, where  $\tau$  and  $\tau_0$  are the turbidities of the suspensions with and without additives respectively.



**Scheme 1.** Synthesis process of the cationic spherical polymer brushes.

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