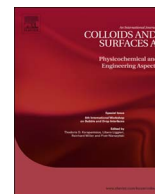




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Preparation of silica/polyelectrolyte complexes for textile strengthening applied to painting canvas restoration

Krzysztof Kolman^{a,*}, Oleksandr Nechyporchuk^{a,1}, Michael Persson^{a,b}, Krister Holmberg^a, Romain Bordes^{a,*}

^a Department of Chemistry and Chemical Engineering, Chalmers University of Technology, Göteborg, Sweden

^b AkzoNobel Pulp and Performance Chemicals, Bohus, Sweden

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ABSTRACT

We here report three different approaches to prepare silica-polyelectrolyte complexes for mechanical strengthening of cotton fibers. In the first approach, polyvinylpyrrolidone (PVP) was used as a stabilizing polymer to delay the adsorption of a poly(quaternary ammonium) species, PQA (a copolymer of dimethylamine and epichlorohydrin), on the surface of silica. In the second approach cationic starch (CS), which is a branched polyelectrolyte, was used and the adsorption of CS resulted in formulations with good colloidal stability. The third approach was based on reduction of the charge density of silica to prevent PQA adsorption. Lowering the pH reduced the surface charge of the silica and enabled control of the adsorption. As a result, the aggregation was prevented and only a thin layer of polymer adsorbed. For all formulations a second polyelectrolyte, carboxymethyl cellulose (CMC) was subsequently adsorbed on the cationic polyelectrolyte layer. The silica/polyelectrolyte formulations were evaluated by dynamic light scattering (DLS). The obtained formulations were applied on model surfaces of degraded painting canvas. The performance of the silica particles coated either with one cationic polyelectrolyte and or with a layer of cationic polyelectrolyte followed by a layer of anionic polyelectrolyte were assessed by tensile testing and the morphology of the treated samples was investigated with SEM. The particles coated with a single cationic layer increased the maximum load at break by 29% at the cost of a reduction in strain. The particles coated with a double layer increased the maximum load to a lesser extent; however, higher values of strain were recorded. For all systems the mass uptake was limited to around 5 wt%.

1. Introduction

Ageing of natural cellulose fibers originates from polymer chain degradation due to acid hydrolysis and oxidation [1]. It is manifested by color change, accompanied by brittleness and loss of mechanical resistance. For many applications, and especially for valuable artefacts of cultural heritage, e.g. painting canvases, it is important to prevent or limit the problems related to ageing [2,3]. Since cellulosic materials are sensitive to acidity, not only a mechanical reinforcement of the fibers has to be achieved [4], an alkaline reserve also needs to be applied to prevent further degradation [5,6]. Silica is suitable for the purpose because apart from being alkaline in nature it has been proven to provide enhanced mechanical properties to textiles when applied as a colloid [7]. Furthermore, the growing trend in art restoration towards minimum intervention promotes the utilization of nanoparticles, which due to small size and a high surface area work efficiently, even at low

concentration. Nowadays, nanoparticles are commonly used in art restoration for different purposes [8]. Calcium and magnesium hydroxide/carbonate nanoparticles are known to be excellent deacidification agents [6,9]. Titanium dioxide and zinc oxide nanoparticles have been tested for protection against soiling, fungi growth and UV-induced degradation [10,11]. Wood stabilization and strengthening, as well as protection against fire, UV light, microorganisms and insects have been achieved by treatment with functionalized silica sols [12].

Good colloidal stability is essential and constitutes a prerequisite for a surface treatment based on nanoparticles. This is especially true when the nanoparticles are used for mechanical reinforcement of fibrous material. Aggregation of the nanoparticles, triggered for instance by changes in pH or ionic strength or by adsorption at a solid surface [13–15], would lead to reduced penetration into the material to be treated and would also give a less homogeneous treatment. Another aspect to consider is the nanomaterial's compatibility with the surface

* Corresponding authors.

E-mail addresses: kolman@chalmers.se, krzysztof.kolman@gmail.com (K. Kolman), bordes@chalmers.se (R. Bordes).

¹ Present address: Swerea IVF, SE-431 22 Mölndal, Sweden.

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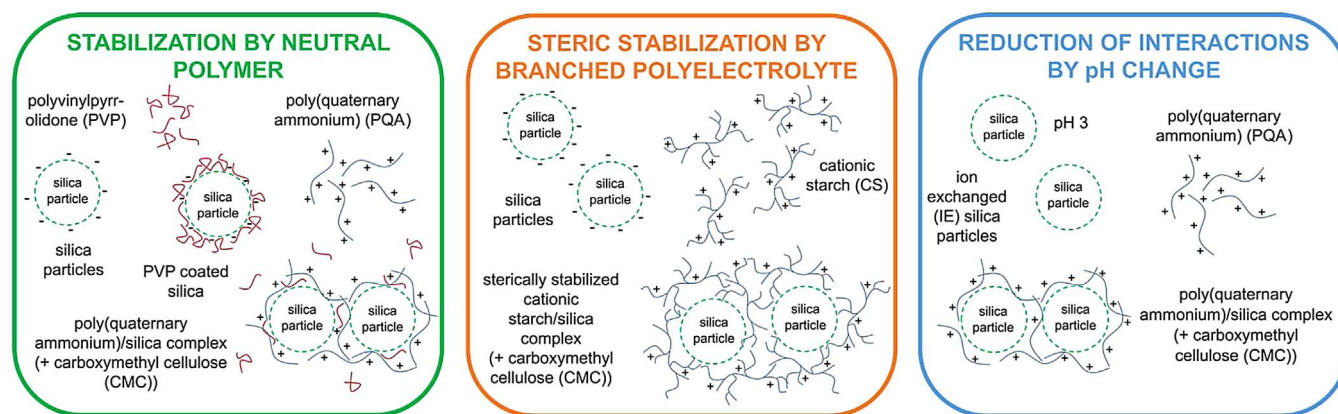


Fig. 1. Schematic illustration of different strategies to adsorb polyelectrolytes on silica particles.

to be treated. A lack of compatibility will lead to a less efficient treatment and will also increase the risks of release of the nanomaterial into the environment. Improved stability and chemical compatibility can often be achieved by adsorption of an oppositely charged polyelectrolyte [16]. However, this may lead to flocculation due to the strong electrostatic interactions between the particles and the polymer chains [17,18]. After the adsorption, the particle is neutralized and lacks electrostatic repulsion which leads to aggregation [19].

In this work, we present the preparation of particle/polyelectrolyte formulations consisting of colloidal silica, copolymer of dimethylamine and epichlorohydrin, cationic starch and sodium carboxymethyl cellulose. The three different strategies proposed to reduce or arrest aggregation in the preparation process of the silica/polyelectrolyte complexes are displayed in Fig. 1: (i) stabilization by neutral polymer, (ii) adsorption of polyelectrolyte with branched structure and (iii) silica surface charge reduction by pH change. In the second part of the work we performed preliminary tests regarding the application of the formulations on model surface of degraded cotton painting canvas. The treated samples were characterized by scanning electron microscopy and by assessment of the tensile strength.

2. Materials and methods

2.1. Materials

Dispersion of silica nanoparticles Levasil CS50-28 (former Bindzil 50/80) was supplied by AkzoNobel Pulp and Performance Chemicals, Bohus, Sweden. The surface area of these particles was $80 \text{ m}^2/\text{g}$ which corresponds to a spherical diameter of 35 nm [20]. The average particle size obtained with DLS was close to 110 nm. The suspension had a pH of 9 (sodium ion stabilized), a concentration of 50 wt% silica, and a density of 1.4 g/ml.

The polyelectrolytes, sodium carboxymethyl cellulose (CMC), Akucell AF0305, and a copolymer of dimethylamine and epichlorohydrin (poly(quaternary ammonium) – PQA), EkaFix 41, were supplied by AkzoNobel Pulp and Performance Chemicals. The degree of substitution of CMC was 0.77 and the viscosity of 1 wt% solution was 12 mPa·s. The weight average molecular weight of CMC was 650 000 g/mol (determined by size exclusion chromatography). PQA was provided as a 50 wt% solution with a density of 1.15 g/ml and a pH of 5.5. The weight average molecular weight of PQA was 50 000 g/mol. Potato cationic starch (CS), Avecat 10, was supplied by Avebe Paper, the Netherlands. The degree of substitution of the CS was 0.02. The viscosity of a 15 wt% solution of the CS was 300 mPa·s. Polyvinylpyrrolidone (PVP) with weight average molecular weight of 10 000 g/mol was purchased from Sigma-Aldrich. For ion exchange and pH adjustments, resin Dowex Marathon C (H^+ form), resin Marathon A (OH^- form) and reagent grade sodium hydroxide and hydrochloric acid

were used. All these chemical were purchased from Sigma-Aldrich. The Dowex Marathon C was washed thoroughly with ethanol and dried before use, the other chemicals were used as received.

Solutions of 0.001 N poly(diallyldimethylammonium chloride) (polyDADMAC) and 0.001 N sodium poly(ethylene sulfonate) (PES-Na) were used as titrants for particle charge density measurements.

Plain-weave cotton canvas was purchased from BarnaArts (Barcelona, Spain). Before treatment, the canvases were first washed in a washing machine at 60°C for 40 min (no detergent added) and then washed at 85°C for 1 h in a glass reactor with mechanical stirring.

The chemicals used for the accelerated canvas degradation, hydrogen peroxide (35 wt%) and sulfuric acid (95–97 wt%), were purchased from Fisher Scientific and Merck Chemicals, respectively.

2.2. Formulation preparation

Schematic illustrations of the strategies to prevent silica aggregation by polyelectrolyte adsorption are presented in Fig. 1. As a reference PQA was added to silica, which led to immediate irreversible aggregation followed by sedimentation within a few minutes. For each strategy, two formulations were prepared – with and without additional adsorption of the anionic polyelectrolyte CMC. In the first approach, a stabilizing polymer, PVP, was used. The formulation stabilized with PVP was prepared as follows: 1 ml of a 50 wt% silica dispersion was mixed with 10.9 ml of ultra-pure water (Milli-Q system) and 1.75 ml of 40 wt% PVP solution. In the next step polyelectrolytes were adsorbed. First, 0.185 ml of a 6 wt% PQA solution and then 0.6 ml of a 3 wt% CMC solution were added. The second approach was based on adsorption of CS which is a branched polyelectrolyte with low charge density. In order to prepare this formulation, 1 ml of a 50 wt% silica dispersion was mixed with 5.5 ml of ultra-pure water and 5.5 ml of a 8.75 wt% solution of CS. The adsorption of the second polyelectrolyte was performed by adding 0.41 ml of a 3 wt% CMC solution. In the last strategy, the interactions between polyelectrolyte and silica were reduced by changing the pH close to that of the isoelectric point of silica. First, 1 ml of a 50 wt% silica sol was ion exchanged (IE silica) by Dowex Marathon C resin until pH 3 was reached. 0.36 ml of a 6 wt% solution of PQA at pH 3 and 5 ml of ultra-pure water were then added. The pH of the formulation was changed to 8 using Marathon A resin and 0.1 M sodium hydroxide solution. The anionic polyelectrolyte was adsorbed by addition of 1.12 ml of a 3 wt% CMC solution. All formulations were mixed using a vortex mixer and their final pH was set to 8 using 0.1 M sodium hydroxide solution and Dowex Marathon A resin. The dry mass content and the composition of each formulation are shown in Table 1.

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