

Research Article

Combination of Natural and Thermosensitive Polymers in Flocculation of Fine Silica Dispersions

Angel Licea-Claverie,¹ Simona Schwarz,² Christine Steinbach,²
Sandra Montserrat Ponce-Vargas,¹ and Sabine Genest²

¹ Instituto Tecnológico de Tijuana, Centro de Graduados e Investigación, Apartado Postal 1166, 22000 Tijuana, BC, Mexico

² Leibniz-Institut für Polymerforschung Dresden e.V., Hohe Straße 6, 01069 Dresden, Germany

Correspondence should be addressed to Simona Schwarz; simsch@ipfdd.de

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A novel strategy for faster and better flocculation in solid-liquid separation processes is reported: the use of the natural polyelectrolyte chitosan (CH2500) in combination with the biocompatible thermosensitive polymer poly(*N*-vinylcaprolactam) (PNVCL). Silica dispersions (Aerosil OX50) were used as model and evaluated by means of analytical centrifuge, laser diffraction, and turbidimetry studies. Results show that the sedimentation velocity is doubled by addition of PNVCL and that at 45°C the density of the sediment is 33% higher, as compared to the use of CH2500 only. This results from the temperature sensitive behavior of PNVCL that phase-separate expelling water at temperatures higher than its LCST (32–34°C) leading to compaction of the flocs. By using this strategy the sediment is more compact, contains less water, and contains a very small amount of biodegradable CH2500 and biocompatible PNVCL.

1. Introduction

Solid-liquid separation through coagulation and flocculation is an important stage of many technological processes. Typical examples are waste water treatment, sludge dewatering, and pulp and paper production as well as the pharmaceutical, cosmetic, and metal working industry. The overwhelming majority of these processes use polyelectrolytes to regulate the stability and flocculation properties of dispersed systems. This resulted in a great variety of synthetic and natural flocculants, which are now commercially available to meet the specific demands of industrial fields, where an efficient solid-liquid separation is required [1, 2].

Application of polysaccharides like chitosan and starch as flocculants in solid/liquid and liquid/liquid separation is also an important field of high industrial relevance, as shown by numerous patents and publications [3–9].

Starch and chitin/chitosan are the most promising candidates to produce and apply natural flocculants on industrial scale [10, 11]. They are abundant and biodegradable polymers,

which can be obtained from renewable natural resources at relatively low costs. High content of hydroxyl (starch and chitosan) and amino/acetamido (chitosan) groups allows advanced chemical modification through cationization, hydrolysis, oxidation, enzymatic grafting or degradation, and so forth to yield polysaccharide derivatives with specific properties to particular fields of application.

In this work a novel strategy for a faster and better flocculation performance is reported: the use of natural polyelectrolytes like chitosan (CH2500) in combination with a biocompatible polymer that exhibit lower critical solution temperature (LCST) behavior in water, poly(*N*-vinylcaprolactam) (PNVCL). By this strategy we envision to speed up the flocculation process and to decrease the water content of the sediment by taking advantage of the hydrophilic to hydrophobic transition of a thermosensitive polymer by heating. We have recently shown that this strategy works well for clay dispersions [12]. Every percentage of water that can be recovered and reused means a saving in money; besides, using thermosensitive polymers, the sediment should be

more compact. The sediment will contain a small amount of the natural biodegradable polymer CH2500 and the biocompatible thermosensitive polymer PNVCL. Because we do not want to invest much energy in heating we chose PNVCL that exhibit a LCST at 32°C in water [13–15]. To the best of our knowledge there is only one report in the literature where PNVCL is used directly as flocculant in acidic conditions at 40°C, a temperature above its LCST [16].

However there are several reports about the use of another thermosensitive polymer in flocculation processes, poly(*N*-isopropylacrylamide) (PNIPAAm) [17–21]. In this account the flocculation mechanism of minerals with PNIPAAm is studied; for example, a detailed study on the interaction forces between bare silica surfaces and PNIPAAm in solutions as well as the influence of contact angle of the surfaces and molecular weight of PNIPAAm is reported [16]. It was found that the presence of PNIPAAm on silica surfaces significantly increases their hydrophobicity at a temperature above the LCST. A strong adhesion between PNIPAAm coated surfaces was also found, which is absent with no polymer in the solution. That means that the detected attractive force and subsequent adhesion resulted from hydrophobic attraction induced by PNIPAAm at temperatures above the LCST.

On the other hand, temperature and molecular weight of PNIPAAm play a significant role in the solid-liquid separation of silica and alumina mineral particles. O'Shea et al. [18] reported that increasing polymer molecular weight resulted in increased polymer adsorption at 25°C. Greater initial adsorbed amounts of polymer on the surface produced more nucleation sites for deposition of additional polymer as the temperature was increased from 25°C to above the LCST.

In our investigation we did not use PNIPAAm since it is not a biocompatible polymer; we used instead PNVCL but not directly as flocculant since the initial conditions for their usage are the natural pH of the solution at room temperature, conditions at which it may not act as flocculant. Silica dispersions (Aerosil OX50) were used as model systems assuring good comparability of the results obtained with various flocculants. Furthermore, the study of Aerosil OX50 dispersions in dependence of the addition of a combination of CH2500 and PNVCL is performed using the analytical centrifuge LUMiSizer which permits a centrifugal separation analysis. This novel centrifugal sedimentation method allows the direct calculation of stability parameters like sedimentation velocity and a prediction concerning shelf life. The flocculation performance using conventional polymeric flocculants is determined by substrate characteristics and stability measurements in dependence on the kind and amount of polymer used. The efficiency of this process may be enhanced by inclusion of thermosensitive polymers through the increase of temperature. Thus, remaining separation problems may now be successfully handled.

2. Experimental

2.1. Silica (Aerosil OX50). Silica Aerosil OX50 was used in all experiments as supplied. Silica dispersions in distilled

water solution (pH = 5.6 ± 0.1) were prepared with ultrasonic treatment for 15 min followed by vigorous stirring during 1 h. HCl was used to adjust the pH. Solid content in the dispersions was 0.1% (w/v).

2.2. Polymers. Chitin forms the exoskeleton of animal groups like insects, spiders, crustaceans (such as crabs), and lobsters and is also found in some fungi and bacteria. Through alkaline splitting-off of the *N*-acetylic group (deacetylation) of chitin the biopolymer chitosan is obtained (Figure 1(a)). Chitosan is a product with a degree of deacetylation (DA) above 40–50% and is soluble in organic acids.

This acidic environment leads to a protonation of the present amino groups which results in a natural, cationic polyelectrolyte. CH2500 with deacetylation degree of 85% and molecular weight of 2 500 000 g mol⁻¹ was obtained by Hepepe Biolog GmbH and used as received. PNVCL with molecular weight of 67 200 g mol⁻¹ was synthesized by free-radical polymerization in 1,4-dioxane at 70°C using 4,4'-azobis-(4-cyanopentanoic acid) as free radical initiator [22]. The structure is shown in Figure 1(b).

The flocculation behavior of CH2500 was investigated in silica dispersions at pH 6. Colloid titration, jar-test, and floc-size measurements by laser diffraction were used for characterization of the phase separation efficiency and elucidation of flocculation mechanism.

2.3. Flocculation. Flocculation tests were carried out in batch in a series of beakers containing 10 mL of Aerosil OX50 dispersion each. After addition of different amounts of the polyelectrolyte solution the mixtures were stirred for 15 min and allowed to settle for 20 min. After that, 2 mL of the supernatant was taken from the beaker and its turbidity measured using a turbidity meter 2100AN (HACH).

2.4. Floc Size Measurements. Dispersions for floc size characterization were prepared according to the procedure described above for flocculation tests. Floc sizes were measured at 2000 rpm rotation speed by means of the laser diffraction particle size analyzer Malvern-Sizer equipped with impeller mixer.

2.5. Turbidimetry. The Nephelometer 2100AN IS (HACH Company, Germany) was used to determine the turbidity *T* of the supernatant. The device is equipped with an IR-LED ($\lambda = 860$ nm) as light source. As the wavelength is out of the visible region, possible coloring of the samples do not perturb the results.

2.6. Analytical Centrifugation. The stability of particle dispersions was studied by an analytical photocentrifuge LUMiSizer (L.U.M. GmbH, Germany), which permits a centrifugal separation analysis and thus allows the direct calculation of stability parameters like sedimentation velocity and predictions concerning shelf life.

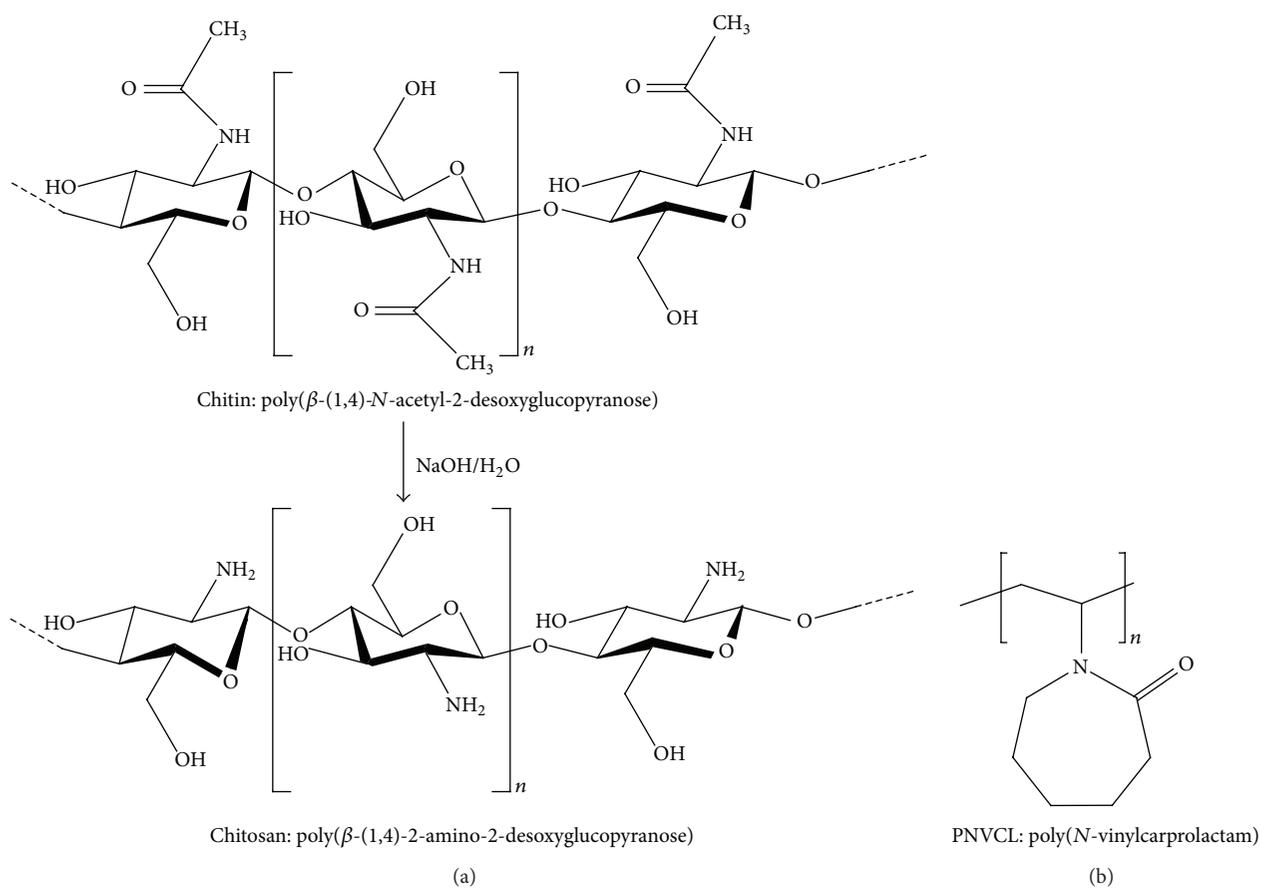
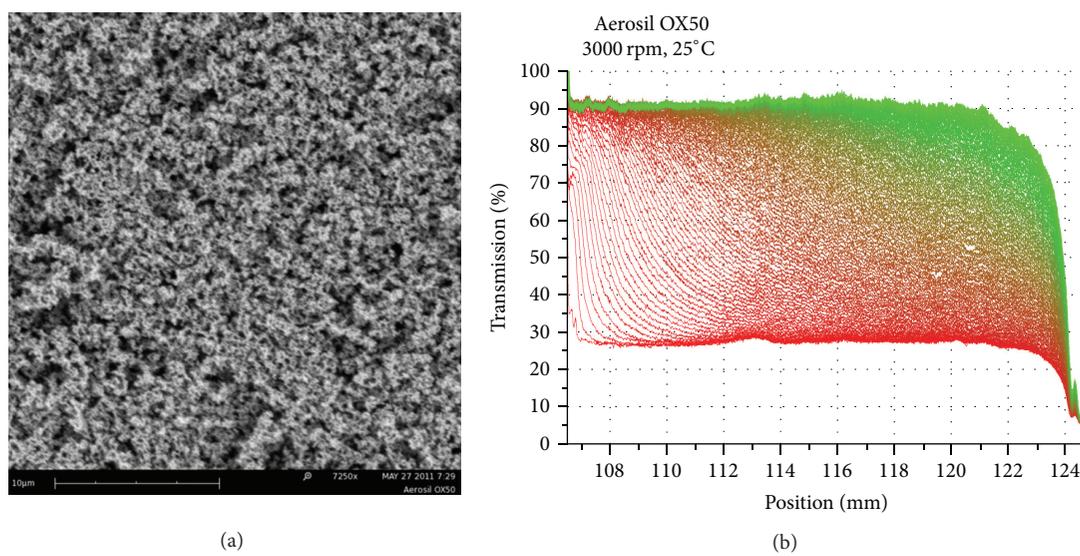
FIGURE 1: (a) Deacetylation of chitin to chitosan, (b) poly(*N*-vinylcaprolactam).

FIGURE 2: Aerosil OX50: (a) SEM picture and (b) transmission profile.

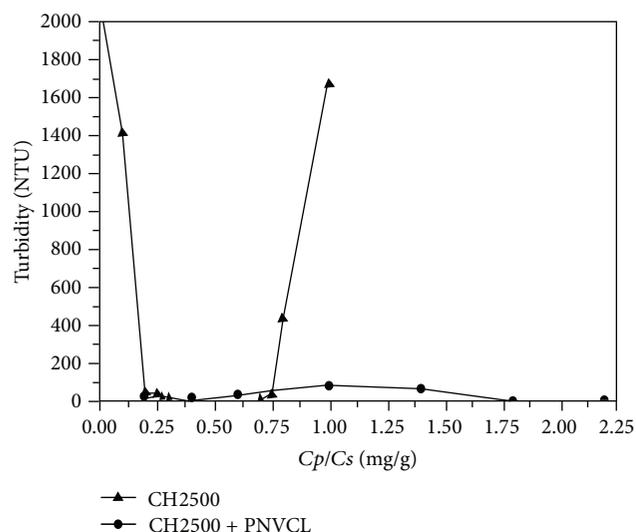


FIGURE 3: Turbidity behaviour of a silica dispersion through addition of different CH2500 amounts and addition of a combination of CH2500 and PNVCL at 25°C.

3. Results and Discussion

Fumed silica is a white fluffy powder consisting of spherically shaped primary particles. These particles produced by flame pyrolysis are very small. The primary particles tend to form aggregates, which have a large specific surface area. The average size of the aggregates in the used dispersion was 280 nm. A scanning electron microscopy (SEM) micrograph of Aerosil OX50 is shown in Figure 2(a). Dispersions of Aerosil OX50 particles in Millipore-water (pH around 6) were prepared by dispersing 5.0 g of powder into 500 mL of water using a high intensity ultrasonic processor (UP200s, Dr. Hielscher GmbH).

The LUMiSizer is very effective in characterizing the storage stability of dispersions and allows comparing the stability in dependence of the polymer amount. Figure 2(b) shows the transmission profiles of Aerosil OX50 at 3000 rpm and a temperature of 25°C. Every 10 s a new profile was measured. The first profile is on the left side and the last on the right side. The profiles show that the dispersion is more or less stable and the size distribution is relatively broad. This is in good agreement with the particle size distribution measured by laser diffraction (see Figure 4).

The solid-liquid separation process of those dispersed particles has been analyzed in dependence on the addition of different amounts of CH2500 (Figure 3). CH2500 as flocculant showed a very narrow range of flocculation. The optimum concentration of the flocculation window was between 0.2 and 0.75 mg/g. Cp/Cs is the ratio between concentration of polymer and concentration of substrate. At higher concentration than 0.75 mg/g redispersion took place very quickly.

The further addition of thermosensitive polymer PNVCL at a constant concentration of CH2500 (0.2 mg/g) in the optimum concentration range showed no influence on the turbidity; however, the sedimentation process was much

faster. In addition, no redispersion of silica took place when PNVCL was added, up to concentrations of up to 2.25 mg/g.

Figure 4 shows the particle size distribution of pure Aerosil OX50 dispersion and the dependence on the polymer concentration. The average size of Aerosil OX50 was 280 nm. The size distribution was very broad from 100 nm to 1000 nm. For applications of CH2500 as flocculant in Aerosil OX50 dispersions it was necessary to increase the polymer concentration. The particle size distribution of Aerosil OX50 dispersion was influenced by the polymer concentration. Increasing the concentration of the polymer, particle size became higher and the distribution was broader. Furthermore by increasing the amount of flocculant the percentage of small particles decreased while the percentage of bigger aggregates increased. The average aggregate size shifted to higher values of up to 7–8 μm .

The combination of CH2500 and PNVCL showed only a small influence in dependence on the polymer amount. The particle size distribution was bimodal with particles in the range of about 300 nm and 5 μm .

The stability of particle dispersions was analyzed by the analytical photocentrifuge LUMiSizer. This microprocessor controlled centrifuge is adapted for rapid classification of stability and separation of even concentrated dispersions [23]. It records the kinetics of transmission changes for 12 samples simultaneously, like a time lapse motion picture, up to 25,000 times faster than tests at gravity by naked eye. The LUMiSizer allows analyzing materials with up to 40% solid content.

The principle of operation of the LUMiSizer is described as follows and is based on the following steps: a light source sends out parallel NIR-light ($\lambda = 880 \text{ nm}$) which is passed through the sample cells lying on the rotor. The distribution of local transmission is recorded over the entire sample length by a CCD-line detector. Finally space and time resolved transmission profiles are obtained from which the integral value indicates the sedimentation progress with time [24–26].

Comparing the stabilization efficiency of CH2500 and combination of CH2500 and PNVCL, the transmission profiles showed differences (see Figure 5). The steps between the profiles were much faster in presence of PNVCL, which indicates that the sedimentation velocity was higher.

In regard to accruing transmission profiles during the sample centrifugation, one can summarize that the lower the integral transmission the more sedimentation will be observed. Thus, the slope of the curves indicates the sedimentation velocity which has its maximum in the flocculation optimum.

In Figure 2(b) the transmission profiles of pure Aerosil OX50 were obtained at 3000 rpm and pH 5.6. But the dispersion at this high speed shows only a very low sedimentation. The sedimentation velocity (v) was only 4.6%/h. Figure 5 shows the transmission profiles at only 300 rpm. The sedimentation was so fast at 3000 rpm that with the first profile the transmission reached about 90% already. The calculated velocity of sedimentation was 1331%/h in presence of CH2500 and 3391%/h for combinations of CH2500 and PNVCL.

Figure 6 shows the variation of integral transmission during sample centrifugation of Aerosil OX50 dispersions

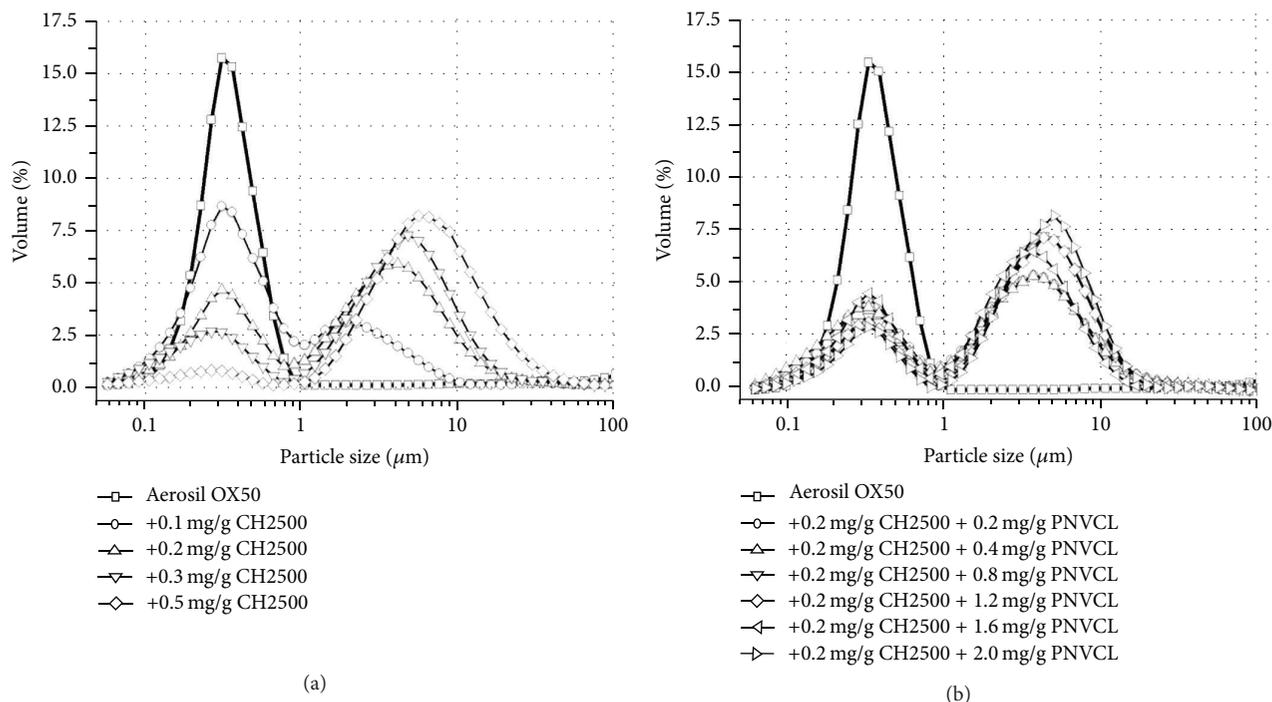


FIGURE 4: Particle size distribution in dependence on the polymer concentration: (a) flocculation with CH2500 and (b) flocculation with combination of CH2500 and PNVCL (pH 5.6, T 25°C).

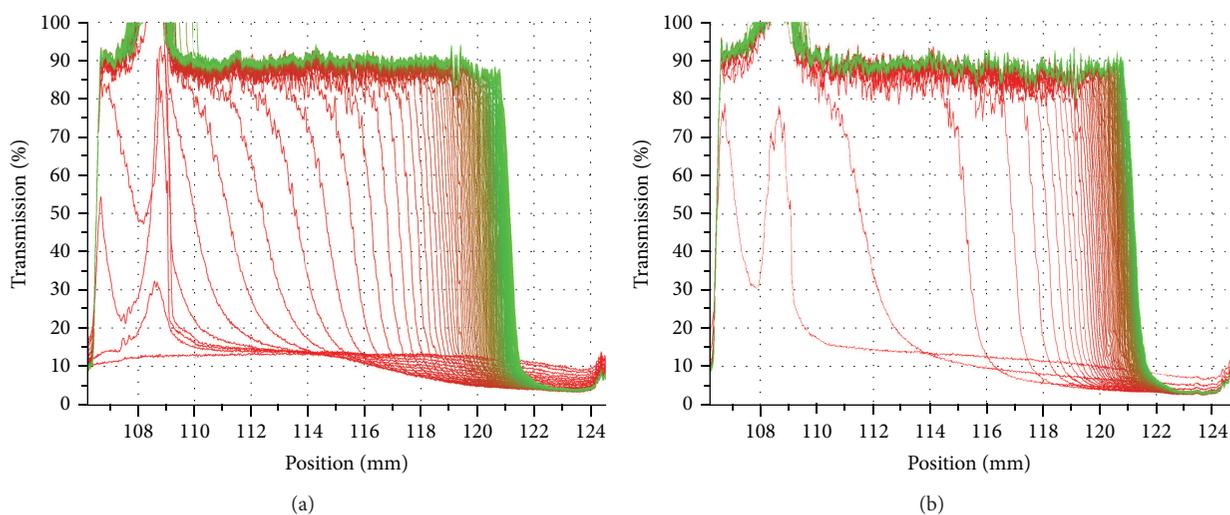


FIGURE 5: Transmission profiles of Aerosil OX50 at 300 rpm and T 25°C: (a) Aerosil OX50 + 0.2 mg/g CH2500 and (b) Aerosil OX50 + 0.2 mg/g CH2500 + 0.8 mg/g PNVCL.

at pH 2 in presence of CH2500 as flocculant and adding PNVCL. Solid lines correspond to the stability of the silica dispersion with CH2500 at 25 and 45°C. As can be seen, increasing temperature decreases the stability of dispersions. The slope was higher at 45°C. The calculated velocity was about 10 times higher (see Table 1).

In the case of combinations of CH2500 and PNVCL we found the same effect: the velocity increases with temperature, but the values in presence of PNVCL were two times that of using CH2500 alone.

In general, much better results were obtained at lower pH values because the charge density of CH2500 was higher and

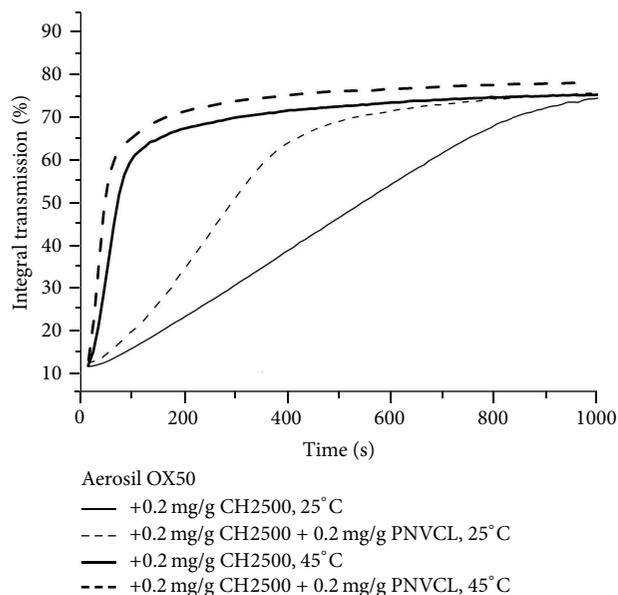


FIGURE 6: Integral transmission versus time of Aerosil OX50 in presence of CH2500 and combination of CH2500 and PNVL at pH 2, 300 rpm and T 25°C and 45°C.

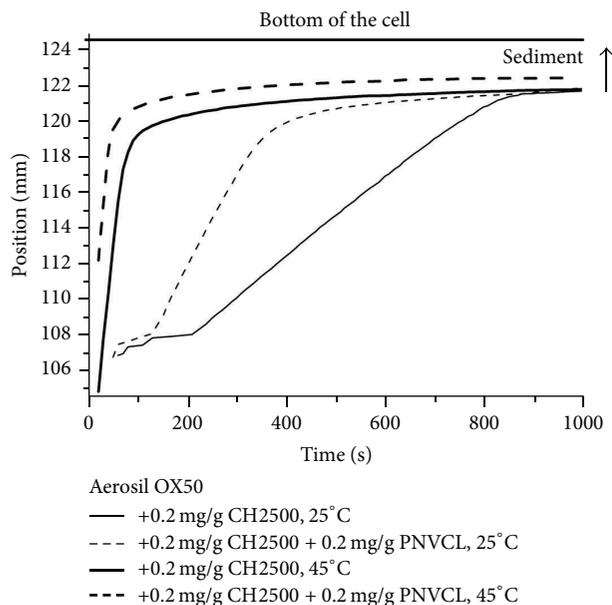


FIGURE 7: Determination of the density of the sediment. Position of the sediment versus time of Aerosil OX50 in presence of CH2500 and combination of CH2500 and PNVL at pH 2, 300 rpm and T 25°C and 45°C.

we had additional protonation of PNVL. The sedimentation process was much faster at lower pH values and in presence of PNVL at higher temperature (higher than the LCST temperature).

A very important aspect related to possible applications in the solid-liquid process is the density of the aggregates. For the storage or the burning of the sediment it is important to have a dense and compact aggregate structure. Because of the

TABLE 1: Calculated velocity at 300 rpm (pH 2).

Sample	v in %/h
Aerosil OX50 + 0.2 mg/g CH2500 25°C	257
Aerosil OX50 + 0.2 mg/g CH2500 45°C	2305
Aerosil OX50 + 0.2 mg/g CH2500 + 0.2 mg/g PNVL 25°C	524
Aerosil OX50 + 0.2 mg/g CH2500 + 0.2 mg/g PNVL 45°C	4032

possibility to build polymer coils during the heating process, we found a more compact sediment in the combination of CH2500 and PNVL. Furthermore, by tracking the position of the phase boundary over time, it is possible to evaluate the packing density of the sediment (see Figure 7).

Thermosensitive polymers like PNVL exhibit a lower critical solution temperature (LCST) behavior in water. That means that by heating an aqueous solution of thermosensitive polymer it can be observed that at temperatures above its LCST the solution becomes turbid, followed by the formation of flocs that finally precipitate.

At temperatures higher than the LCST it is significant that these flocs showed a positive influence of the flocculation behavior of inorganic particles in dispersion. The flocculation velocity was very high and the aggregates of flocs from inorganic particles, chitosan, and thermosensitive polymers were very dense. The position of the bottom in the measuring cell was at 124.7 mm. The plateau of the position was the same for the use of CH2500 (at 25 and at 45°C) and for the combination of CH2500 and PNVL at 25°C. In these cases, the height of the sediment was 3.2 mm but in the case of use of combination of CH2500 and PNVL at 45°C there was a more compact sediment of 2.3 mm. The observed behavior can be rationalized as a two-step process (Figure 8) and has been described by Sakohara et al. [26–28]. In the first step the silica dispersion (Aerosil OX50) bearing negative charges is flocculated by the cationic polyelectrolyte chitosan (CH2500), PNVL is absorbed onto the floc surface by bridging mechanism. In the second step PNVL goes through its LCST (at 45°C) spelling water out and compacting the flocs.

A lot of applications like paper production or dewatering of sludge takes place in the temperature range from 20 to 60°C so it is a big advantage to have a minimum of water in the sediment. In presence of PNVL the sediment showed up 27% lower water content than using a polyelectrolyte like CH2500 alone.

4. Conclusions

In this study, we compared the flocculation efficiency of chitosan and combinations of chitosan and poly(*N*-vinylcaprolactam), a thermosensitive polymer. We had studied the combination of CH2500 and PNVL as flocculants for Aerosil OX50 dispersions, as a function of concentration of the polymers and temperature. The biodegradable polyelectrolyte chitosan in combination with the temperature

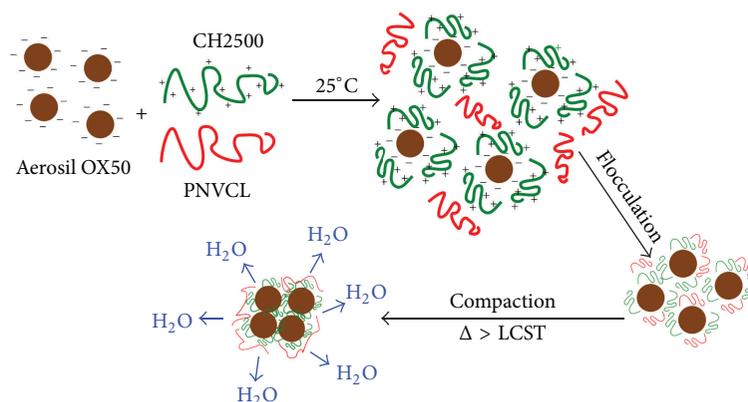


FIGURE 8: Proposed flocculation-compaction mechanism for the use of a combination of chitosan and PNVCCL for solid liquid separation of silica dispersions.

sensitive biocompatible PNVCCL gave a compact sediment at temperatures higher than the LCST temperature of 45°C. The density of the sediment is up to 1/3 higher and the sedimentation velocity is doubled as compared to use of CH2500 only.

The current developments in synthesis of polymers and copolymers allow the improvement of flocculant performance through variations of structure, branching degree, and hydrophobicity of polymers, a task that we would like to tag in the future.

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