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To Study The Flocculation Behaviour Of Poly (NIPAM) And Copolymer Acrylamide Microgel

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Abstract
The flocculation behaviour of poly(N-isopropyl acrylamide) (PNIPAM) copolymer Acrylamide microgel was studied as a function of 0.3 M NaCl. The critical flocculation temperature of P-NIPAM was determined by using DLS. The DLS graphs of CFT were plotted hydrodynamic diameter as a function of temperature. In the presence of an electrolyte, particles flocculate due to surface charge masking. But there was variation in hydrodynamic diameter upon variation in concentration of acrylamide and also what time that was added. The particle dispersion was stable at room temperature also with the addition of acrylamide and as the temperature was raised particles get aggregated.

Introduction

Microgels are made up of polymer atoms that can react to external stimulations like temperature, pH, and ionic strength. Poly-N-isopropyl acrylamide microgel has the ability to be swollen and de-swollen in the presence of a dissolver such as water. The microgels have intra cross-linked Structure. The poly (NIPAM) is synthesized mainly by the surfactant-free emulsion polymerization (SFEP) technique.[1,2,3]

According to Kawanguchi (2000), the CFT (Critical flocculation temperature) is the temperature in which the flocculation of the microgel particles occurred due to the changes in the steric stabilization. Thus, the variation in the CFT in the presence of electrolytes in various concentrations of microgels is may be due to disturbance in electrostatic repulsive force [4].

Keywords: PNIPAM Microgel, Acrylamide, Flocculation

Method

In this experiment, the Flocculation behavior of microgel particles was done by using DLS.

Preparation of Samples for DLS

Using NaCl: For the preparation of 0.5M NaCl, 2.922g of NaCl was poured in 100ml of distilled water. Then, for the DLS run, 0.3M NaCl concentration was required. Thus, for DLS, took the 3ml of 0.5M NaCl, added 1ml of microgel and 1ml of distilled water, and made the total volume 5ml (Ratio 3:1:1). Lastly, a sufficient quantity of sample has been poured into a glass cuvette for DLS analysis.

DLS (Dynamic light scattering) measurements were carried out by using Malvern Zetasizer Nano ZS with a measurement angle of 173ºC backscattering. The sample was diluted as described in the preparation of samples for DLS and poured into a glass cuvette. Three measurements were taken at each temperature, each consisting of 13 sub runs to get the average hydrodynamic diameter. The flocculation or aggregation of different ratios of Poly NIPAM and copolymer Acrylamide microgel study was carried out using NaCl runs in DLS.

Result and Discussion

According to Gracia (2007), the microgel particles showing flocculation because of the electrostatic forces are blocked by a high concentration of electrolyte in the dispersion. When the temperature gets increased to the CFT, the microgel particles aggregated and result in a higher hydrodynamic diameter. Flocculation can be indicated by the dramatic increase of the hydrodynamic diameter which occurs around the VPTT (Volume phase transition temperature). [6]

The hydrodynamic diameter of poly NIPAM (100%) microgel drastically increased about 20000 nm at around 45ºC and as temperature increased the hydrodynamic diameter decreased to 6000nm (Figure 1). The microgel particles get flocculate at 45^{O}C and at room temperature they were in a dispersed state.

The hydrodynamic diameter significantly increased around VPTT, due to the formation of flocs of microgel. These large flocs sediment after some time, which causes the curve to decline. The addition of an electrolyte diminishes the repulsive forces between the particles and tent to aggregation.

The addition of copolymer acrylamide in the P-NIPAM microgel did not affect the CFT. The same results were obtained for 90:10% and 80:20% P-NIPAM-Co-AAm microgels (Figure 2 and 3).

100% P-NIPAM MICROGEL

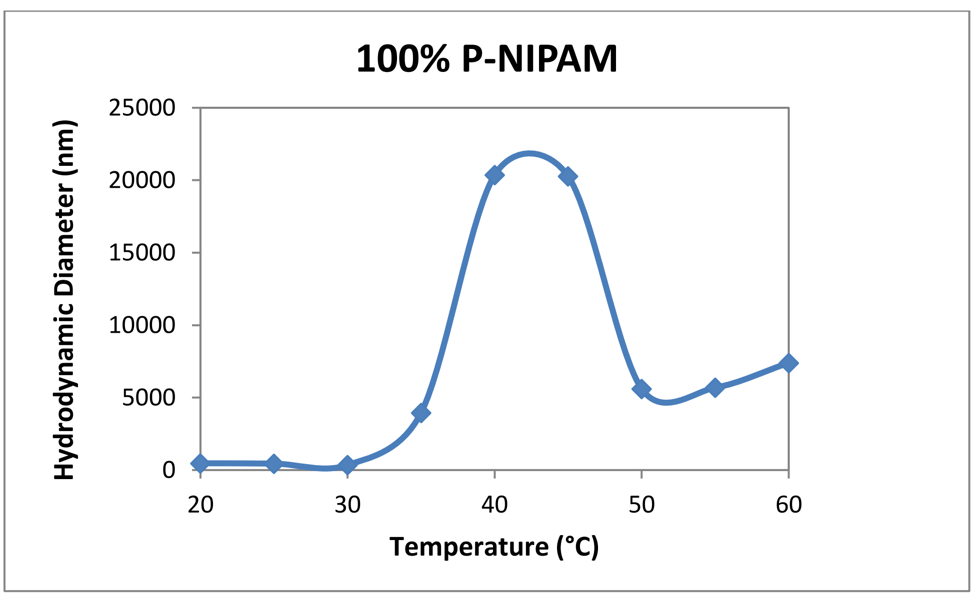

Figure 1: Hydrodynamic diameter of 100% P-NIPAM microgel as function of temperature for NaCl.

90:10% P-NIPAM Microgel and 80:20% P-NIPAM Microgel:

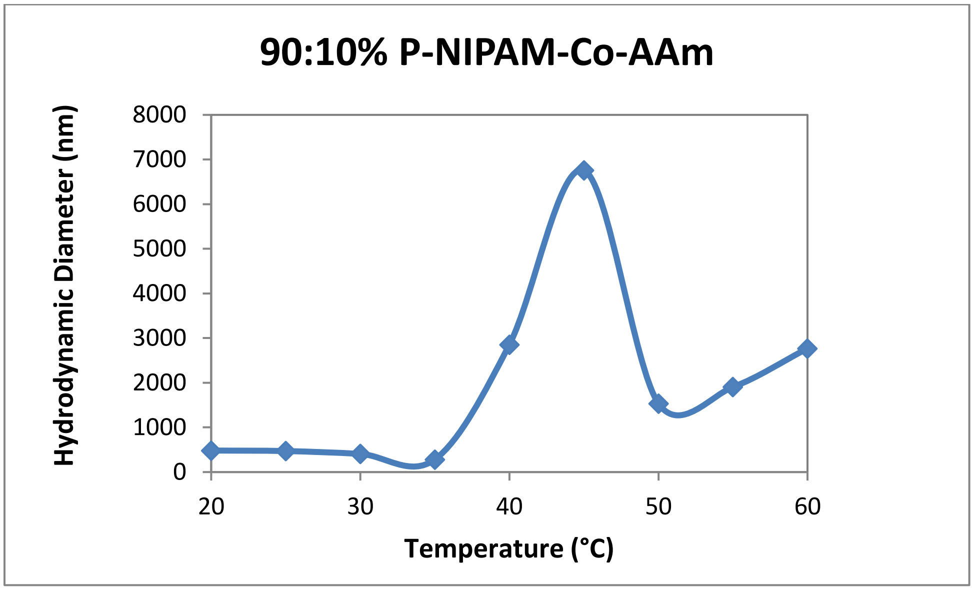

Figure 2: Hydrodynamic diameter of 90:10%P-NIPAM microgel as a function of temperature for NaCl.

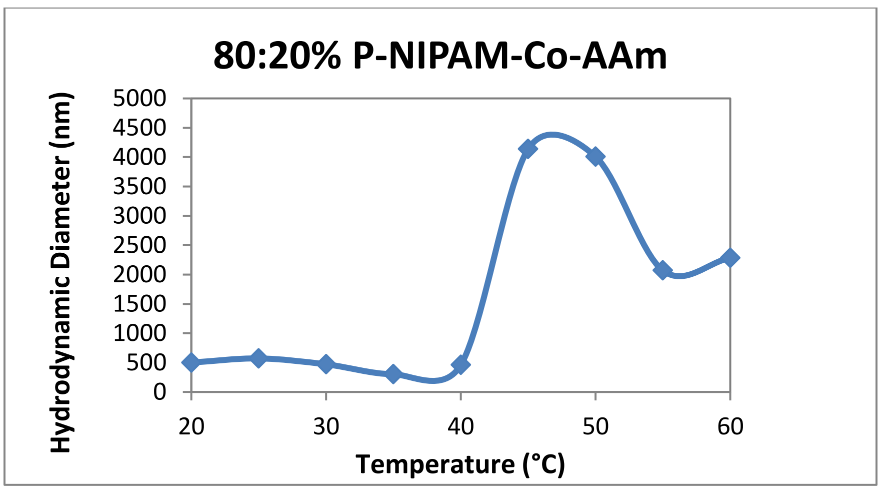

Figure 3: Hydrodynamic diameter of 80:20%P-NIPAM microgel as a function of temperature for NaCl.

For the injected acrylamide 85:15% P-NIPAM-Co-AAm, the CFT was the same as that of 100%, 90:10%, 80:20% P-NIPAM microgels (Figure 4). It was noticed at $45^{O}C$ and hydrodynamic diameter was 5479±5nm at that point. But for directly added copolymer acrylamide it was show different results. The flocculation temperature was seen at $50^{O}C$ and the hydrodynamic diameter was 3586±9nm at that point.

85:15% (Direct) P-NIPAM Microgel and 85:15 (Inject) % P-NIPAM Microgel:

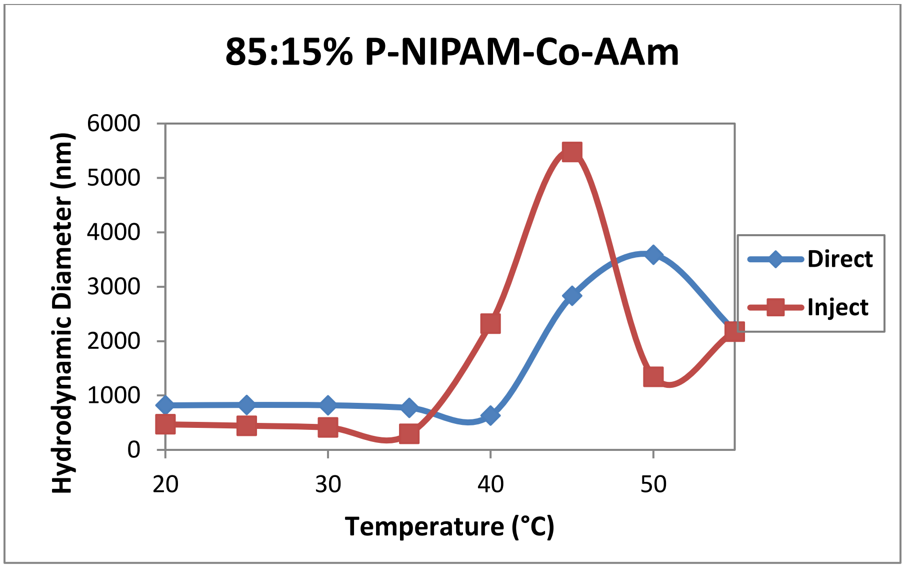

Figure 4: Hydrodynamic diameter of 85:15% (Direct) and 85:15% (Inject) P-NIPAM microgel as a function of temperature for NaCl.

The results for 70:30% A, B, C samples are similar to that of other microgels (Figure 5). As samples show CFT at 50^{O}C. But the particle size for samples A and B were the same 500±10nm at room temperature but that was double for sample C which was 1000±8nm. This was shown as a result of the addition of acrylamide. For sample C, acrylamide was added with NIPAM and BA. So due to reaction of both NIPAM and acrylamide with the initiator results in a significant increase in diameter.

70:30% (A, B, C) P-NIPAM MICROGEL:

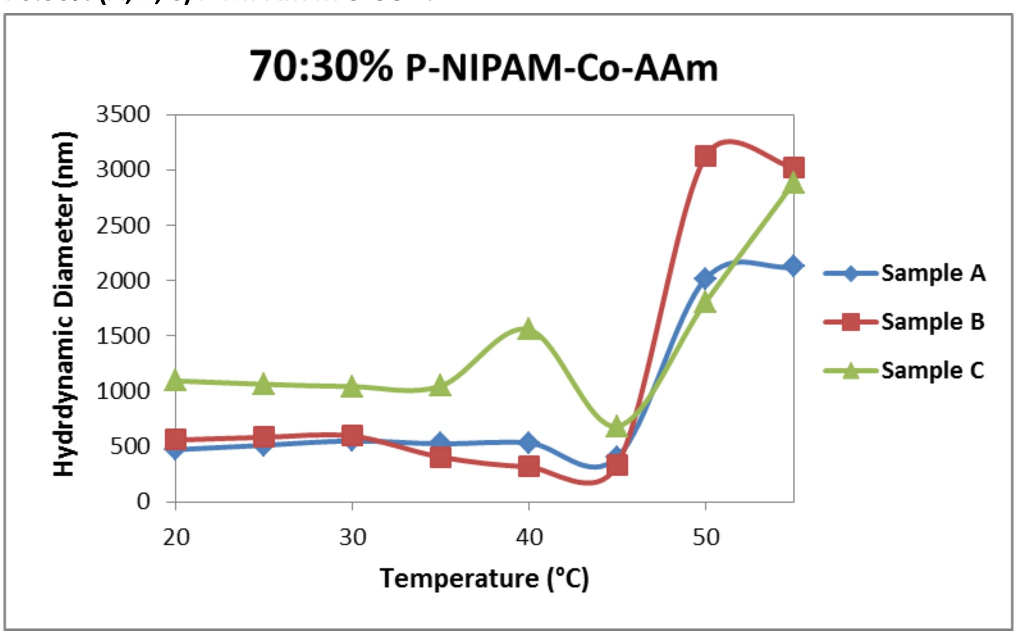

Figure 5: Hydrodynamic diameter of 70:30% (A, B, C) P-NIPAM microgel as a function of temperature for NaCl.

Conclusion

It was shown that the flocculation of P-NIPAM particles is consistent with the addition of copolymer in different concentrations. In the presence of an electrolyte, particles flocculate due to surface charge masking. But there was variation in hydrodynamic diameter upon variation in concentration of acrylamide and also what time that was added. The particle dispersion was stable at room temperature also with the addition of acrylamide and as the temperature was raised particles get aggregated.

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