

Potential for Desalination Using Lower Critical Solution Temperature Polymers: Concentration of Salt Solutions by Pluronic PE6200

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ABSTRACT: A commercially available lower critical solution temperature (LCST) polymer was demonstrated to preferentially adsorb water containing a reduced ion concentration, from CrCl_3 salt solutions and release some of this scavenged water at higher temperatures. The scavenging ability of the polymer was demonstrated to be dependent on the bulk salt concentration. The volume of water scavenged and the concentration of the remaining CrCl_3 solution increased linearly with the amount of LCST polymer (Pluronic PE 6200) used. Both the volume of water removed

and the amount by which the remaining CrCl_3 solution was concentrated decreased with increasing CrCl_3 concentration. Implications for research into a novel method for sea water desalination are addressed. We also show water extraction from dilute sodium chloride and sea water, and that these results are consistent with the CrCl_3 model system. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 113: 2346–2352, 2009

Key words: desalination; LCST; pluronic polymer; water structure

INTRODUCTION

Desalination is a well established technology. Some of the successful and novel methods developed in the last fifty years include multistage flash desalination (MFD), reverse osmosis (RO) desalination, and less successfully, ion exchange resin removal of salt (SIROTHERMTM) and clathrate hydrate crystallization. The most used is MFD which distills salt water through evaporation at elevated temperature and sequentially reduced pressures, in multiple stages.¹ The heat energy is often supplied through steam, however, to improve efficiency the heat released upon later exothermic water condensation (enthalpy of vaporization) is transferred to the incoming brine through heat exchangers. A competitive technology which is increasing in usage is RO membrane desalination,^{1,2} where sea water is forced under pressure through a semi-permeable membrane. The membrane allows almost exclusive passage of the water, but not the salt. In the SIROTHERM process, salt is selectively removed from brine by ion exchange resin beds and the resin is subsequently regenerated through heating,^{3–7} where the salts are desorbed at around 80°C. Another method is clathrate hydrate crystallization,⁸ this process attempts to profit from

the phenomenon that at low temperature and high pressure small alkanes (such as propane or methane) form hydrated crystals, such as those typically found in fossil fuel lines, but also in sea water under the correct conditions, high pressure, and low temperature. These crystals contain pure water, and so when they are collected and the pressure released, evaporating the gas, pure water remains. This technology required significant expenditure of energy for creating high pressures, and the crystals could not be satisfactorily separated from the sea water.⁸ This list is not exhaustive, for an extensive review of desalination processes both novel and practical see the following reviews.^{1,2}

The problem of removal of salt from water seems a simple one; however, the preferred method must be robust, economical, and efficient. An optimal process would only supply enough energy to overcome the enthalpic and entropic requirements to 'de-mix' the salt ions from the solvent, water. In practice significant losses are observed, such as those caused by the large pressures required to force water through a semi-permeable RO membrane; to apply pressure to the sea water/alkane mixtures to obtain the clathrate hydrate crystals; or the heat required to regenerate the ion exchange resin in the SIROTHERM process, to mention a few.

The work described here is based upon the technology of purifying salt solutions by the formation of ordered water surrounding uncharged organic molecules. Instead of forming solid crystals similar

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to clathrate hydrates, here we test whether the ordered water surrounding non-ionic lower critical solution temperature (LCST) polymers^{9,10} can remove water from the salt solution. The non-ionic polymers used here are soluble in water below the LCST and phase separate above it. Their solubility is believed to be caused by ordered water surrounding the polymers which allows them to remain in solution at low temperatures, and these are then destabilized at higher temperatures causing the polymers to precipitate.⁹⁻¹¹ A general advantage of selecting LCST polymers is that they can be designed to precipitate at a selected temperature through balancing hydrophobic and hydrophilic monomers¹² or crosslinked to form gels that release water with increasing temperature.¹³ Some evidence has previously been shown that poly(ethylene oxide) homopolymers, at low concentrations, can remove water from salt solutions at temperatures $>90^{\circ}\text{C}$.

This concept for LCST purification of water requires three stages:-

- (1) Formation of polymer-water complexes;
- (2) Mechanical separation of complexes from the bulk solution;
- (3) Release of the water from the polymer.

With access initially limited to pluronic polymer PE6200, kindly supplied by BASF, and simple homopolymers of *N*-isopropylacrylamide (NIPAM), we have focussed on the first of these steps, which is the key to the feasibility of such a process, and investigated the remaining steps only briefly. Mixing the LCST polymer and water, then heating to precipitate the polymer would effectively disrupt most or all of the structured water surrounding the pluronic, which we hoped would be the source of the pure water. This work became a preliminary investigation to justify development of new and novel polymers which might eventually lead to a cheap and low energy method of desalinating water with minimal infrastructure and ongoing costs.

In this publication, we have chosen to use CrCl_3 salt as a model for sea water. Because of its green color, its concentration can be measured spectrophotometrically, and the extinction coefficient is insensitive to temperature. In contrast, conductivity measurement is sensitive to temperature, and so all comparative measurements are prone to thermal variation. It is known that different ions can affect the LCST (or cloud point) behavior of pluronic polymers^{14,15} so for completeness a brief comparison with dilute sodium chloride and sea water is made.

The process explored here is not intended to compete with current (extremely successful) processes, but we wish to stimulate research into low energy, gradual output, extremely low cost solar powered

desalination without wasting energy by evaporating the water. This work is akin to the work done by the International Organization for Dew Collection¹⁶ (OPUR) who look at producing technologies to benefit remote communities in third world countries, supplying cheap water for sustenance using renewable processes.

EXPERIMENTAL

Materials

Water used was obtained from a RO purification system and used without further purification. Sea water came from Corio Bay, Geelong, Victoria, Australia and filtered through a porous glass frit to remove contaminants including sand and small biological organisms. The pluronic polymer used (PE6200) was a gift from BASF chemicals. It is a block copolymer with an approximate structure $(\text{EO})_6(\text{PO})_{37}(\text{EO})_6$ of ethylene oxide (EO) and propylene oxide (PO) units, and a cloud point of $\sim 33^{\circ}\text{C}$ for a 1% w/v solution. The cloud point is the temperature at which the ordered water surrounding the EO segments are disrupted, the polymeric surfactant becomes insoluble, and the solution becomes turbid, in the same way the LCST polymers precipitate at their LCST. The chromium (III) chloride ($\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$) was from AJAX chemicals, Sydney, Australia and was used without further purification. The sodium chloride was A.R. Grade 99.9% from BDH Chemicals, and was used without further purification.

Water extraction from chromium (III) chloride solution: Variations in concentration and volume

Ten milliliter solutions of aqueous CrCl_3 , along with 0, 2, 4, 6, 8, and 10 g respectively of the BASF pluronic PE6200 was added and shaken to form clear solutions in measuring cylinders. The solutions were then heated to $\sim 70\text{--}75^{\circ}\text{C}$, which is significantly above the cloud point, hence separation of the pluronic occurred forming clear polymer and salt solution layers. Complete separation of the layers was allowed to occur overnight (greater than 18 h). The volume of the upper water layer was measured and then a sample of this aqueous layer was carefully obtained without disturbing the lower viscous polymer layer. The UV-visible spectra were obtained using a Cary 300 instrument (Varian) with CrCl_3 solutions greater than 50 mM being diluted 1/10 due to their very high absorbance. An absorbance maximum was observed around 575 nm, this was used to monitor the concentration of $\text{Cr}(3+)$ in solution. At most concentrations the pluronic polymer was denser than the salt solution, however, as salt

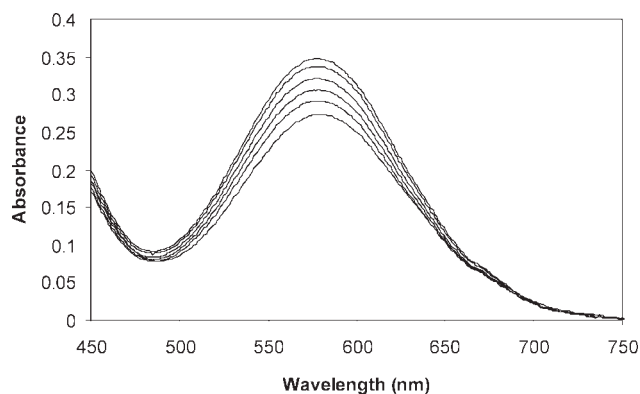


Figure 1 Absorption spectra of CrCl_3 solutions treated with varying amounts of pluronic PE6200. The curves from bottom to top were treated with 0, 2, 4, 6, 8, and 10 g of pluronic per 10 mL of 200 mM CrCl_3 . The spectra were taken of the solutions diluted 1/10.

concentration increases so too will the density of the aqueous phase. At a concentration of 200 mM, the densities of the CrCl_3 solution and pluronic polymers were nearly matched, however, if heated to 72°C expansion of the pluronic phase resulted in its density decreasing slightly and the densities of the two phases would then invert. This inversion of densities appears very suddenly between 70 and 75°C, where a slightly higher temperature would result in the salt solution at the bottom, and a slightly lower temperature (ca. 1–2°C) would result in the pluronic at the bottom. Minimal cooling of the sample results in the pluronic polymer becoming denser and falling to the bottom, making measurement at this concentration difficult. Some inaccuracy of the measurements at this concentration was also caused by inversion of the meniscus of the salt solution layer against the pluronic layer.

Water extraction from $\text{NaCl}(\text{aq})$ and sea water. Variation in salt concentration by conductivity

Treatments of 50 mM $\text{NaCl}(\text{aq})$ and sea water with pluronic PE6200 were the same as described earlier. The 50 mM NaCl solutions were diluted 1/10, prior to conductivity measurement. Sea water obtained after each separation was then diluted 1/25 and the conductivity compared to an equivalent dilution of sea water. The conductivity was measured using a Metrohm Herisau Konduktometer E527, which measures conductivity between two platinized plates (surface area $\sim 0.5 \text{ cm}^2$ and $\sim 15 \text{ mm}$ separation). Solutions were measured at room temperature (23°C) and equilibrated and measured in series to reduce thermal drift. The solution where 10 g of the pluronic polymer was mixed with seawater resulted in near density matching of the two phases and therefore good separation and accurate volume measurements was not possible.

Water reclamation from pluronic polymer

Solutions containing 10 g pluronic polymer and 10 mL of aqueous CrCl_3 were mixed and thermally separated (at $\sim 70^\circ\text{C}$). The pluronic polymer was carefully removed so not to contaminate the sample with the salt solution, and then heated to $\sim 80^\circ\text{C}$ and $\sim 90^\circ\text{C}$. Upon each temperature increase the clear pluronic became cloudy and a small amount of salt solution rose to the top. This was carefully collected (solution remained transparent), and was again diluted 1/10 (for 200 mM CrCl_3) and 1/5 (for 100 mM CrCl_3) and the concentration measured spectrophotometrically.

RESULTS AND DISCUSSION

Water extraction from chromium (III) chloride solution. Variations in salt concentration

Aqueous 200 mM CrCl_3 was collected after treatment with various concentrations of PE6200 and the absorbance spectra of solution diluted 1/10 are presented in Figure 1. Here, the absorbance maximum around 575 nm increases with the increased amount of PE6200 used. These measurements were repeated for 10, 20, 50, and 100 mM CrCl_3 and the profiles normalized with initial concentration are presented in Figure 2. A linear relationship between the initial amount of PE6200 dissolved and the final salt concentration of the CrCl_3 solution was observed for each initial concentration. The increase in salt concentration is explained by there being some water retained in the pluronic polymer after it separates out forming a lyotropic liquid crystal polymer phase.^{11,17} These pluronic polymers are well known to form a range of crystalline phases at high polymer concentrations and high temperature,^{11,17–20} however, this mesoscopic behavior is not discussed here. The gradients obtained in Figure 2 are presented in Figure 3, the error bars correspond to the 95%

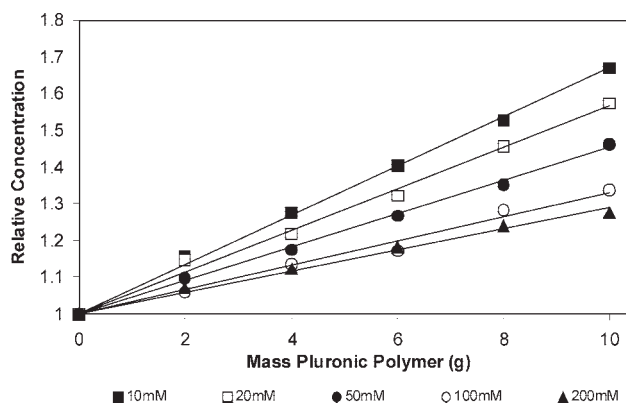


Figure 2 Plot of relative concentration, normalized to initial concentration, of CrCl_3 as a function of mass of pluronic PE6200 applied.

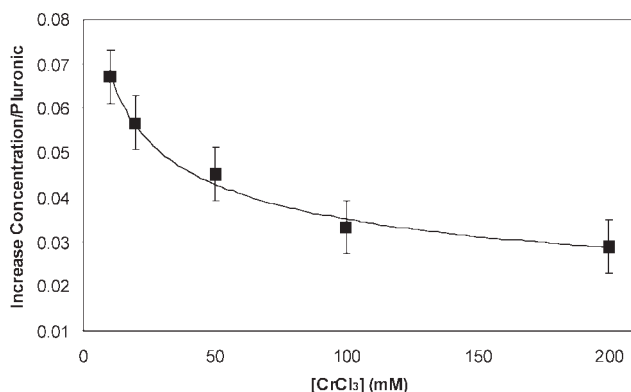


Figure 3 Plot of the gradients obtained in Figure 2 as a function of the initial CrCl₃ concentration, solid line merely to guide the eye.

confidence limits of the linear regression fits and the solid line is merely to guide the eye. The gradient plotted is an indication of the ability of the pluronic polymer to extract water from the CrCl₃(aq) phase. From the data presented in Figure 3, this ability is reduced by increasing the CrCl₃ concentration.

Water extraction from chromium (III) chloride solution. Variations in volume

To better understand the removal of water, the corresponding changes in volume were measured and the reduction in volume of CrCl₃ solutions plotted as a function of mass of pluronic, in Figure 4. A linear increase in the amount of water removed with mass of pluronic polymer used, is observed. As issues arise from interfacial effects (such as unclear menisci) between the lower pluronic and upper CrCl₃ solution, these values should only be viewed as indicative. The gradients of the linear plots in Figure 4 are presented in Figure 5 as a function of CrCl₃ concentration, the gradient being a measure of the amount of water captured within the pluronic

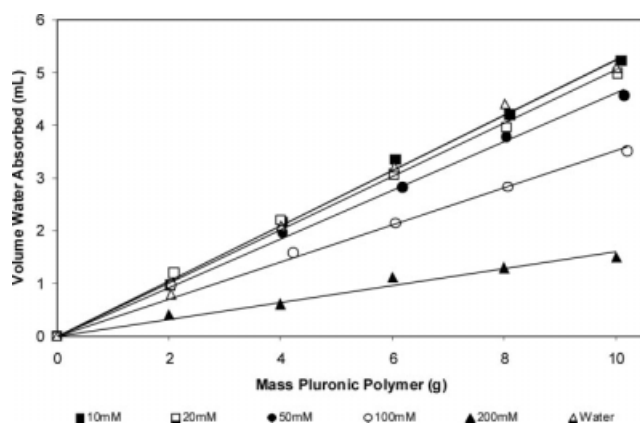


Figure 4 Plot of the volume of water absorbed from 10 mL of aqueous CrCl₃ as a function of the mass of pluronic polymer PE6200 applied.

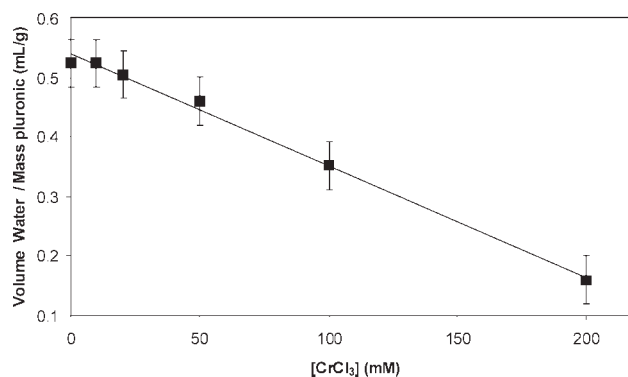


Figure 5 Plot of the gradient obtained in Figure 4 as a function of the initial CrCl₃ concentration.

polymer phase. It is clear that the volume of water absorbed by the pluronic polymer is sensitive to the CrCl₃ concentration, decreasing as the salt concentration increases. This is explained by the water associated to the polymer having a lower salt concentration than that of the bulk aqueous solution, therefore there will be an osmotic gradient driving water from the polymer phase to the bulk water. The higher the salt concentration, the stronger this driving force becomes, resulting in a reduced uptake of water into the polymer phase.

Concentration of chromium (III) chloride in pluronic polymer phase

The data in Figures 1–4 show that water is captured by the pluronic polymer even after thermal separation from the bulk aqueous phase. It must also be considered that some CrCl₃ will be associated to this water, so using the change in volume and concentration of the CrCl₃ solution (presented in Figures 2 and 4) we can get an indication of the concentration of CrCl₃ in the pluronic phase. From a mass balance, we can derive an expression for the concentration of chromium (III) chloride in the water captured in the pluronic, *c(pluronic)*, eq. (1):

$$c(pluronic) = \frac{V(o)c(o) - V(f)c(f)}{V(o) - V(f)} \quad (1)$$

where *c(o)* and *c(f)* are the initial and final salt concentrations and *V(o)* and *V(f)* are the initial and final volume of water in the bulk aqueous solution. The equation is explained simply by the numerator equating to the change in mass of CrCl₃ in the aqueous phase before mixing and after separation, divided by the change in volume. On further examination of the data presented above, no explanation of the linearity of the profiles in Figures 2 and 4 is possible at this stage. Using the volume and concentration values from the measurements obtained for 10 g of pluronic in 10 mL of CrCl₃

TABLE I
Composition of Solution Captured by Pluronic Polymer
Calculated Using Equation (1)

c(o) (mM)	[CrCl ₃] (mM) in pluronic	Relative concentration (%)
10	3.0 ± 0.4	30 ± 4
20	7.6 ± 0.6	38 ± 3
50	24 ± 3	48 ± 6
100	40 ± 10	40 ± 10

solution, approximate concentrations are calculated for the concentration of CrCl₃ in the water associated to the separated pluronic polymer phase using eq. (1) and are presented in Table I. The data from 200 mM CrCl₃ did not give meaningful values; however, the numbers for the lower concentration are all between 30 and 50% the values of the bulk, which is a significant decrease in concentration. Repeat measurement of the volumes presented, showed that a variation in volume of approximately ±0.4 mL is found, which is the largest source of error. This error is incorporated into the calculated values from eq. (1) and the approximate error for the values in Table I are also presented. Although these results are not comparable with current successful desalination methods, however, some efficacy is clearly demonstrated. Similarly to our findings, Kjellander and Florin found that when PEO polymers were exposed to salt water and then thermally separated, a more concentrated saline solution was produced.⁹ They used this result to deduce the amount of water associated to the PEO, but did not discuss the possibility that some salt could be associated with any entrapped water, and only investigated very low PEO concentrations.

Water extraction from NaCl solutions. Variation in concentration and volume

In this study, we are using Cr(3+) as a model for Na⁺, however, cations from different positions within the Hofmeister series are known to affect pluronic polymer phase behavior.^{14,15,21} Therefore, in order to test whether a similar process is possible in sea water, water removal from 50 mM NaCl and from sea water solution were studied and compared with the above results for CrCl₃.

Once again salt solution and pluronic polymer are mixed to produce a homogeneous solution and this is then heated resulting in separation of upper aqueous and lower polymer phases. A sample of the upper salt solution is removed and the variation in conductivity of this is measured to monitor the NaCl concentration. Any ions present in the pluronic polymer which may be transferred into the bulk phase would be a source of error. An investigation using

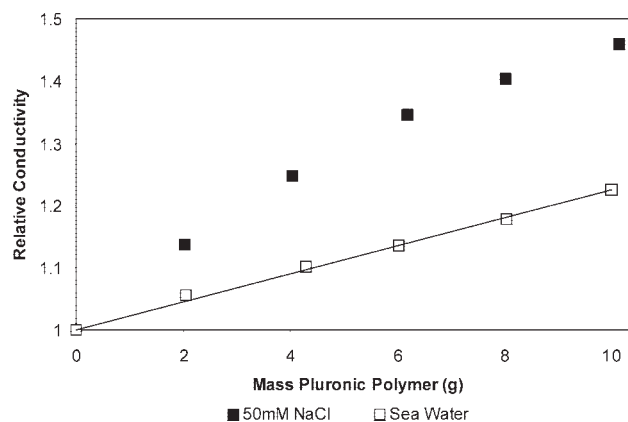


Figure 6 Plot of relative conductivity, normalized to initial conductivity, of 50 mM NaCl and sea water as a function of mass of pluronic PE6200 applied.

pure water found that the contribution of conductive species in the pluronic polymer is insignificant compared to that of the salt in the aqueous phase. The conductivity varies linearly with salt concentration over these ranges, and so changes in conductivity presented here are taken to represent changes in NaCl concentration. We thus are able to plot the relative conductivity normalized with initial conductivity versus mass of pluronic polymer used in Figure 6. The relationship is not quite linear for 50 mM NaCl, but the NaCl concentration increases to a similar extent as the profiles measured for CrCl₃ solutions, as seen in Figure 2. The increase in salt concentration of sea water was found to be quite linear over the same concentrations of pluronic polymer. The volume change observed when 50 mM NaCl and sea water were exposed to pluronic are presented in Figure 7. Once again linear profiles are observed, the increases are consistent with those measured for CrCl₃, see Figure 4. The highest pluronic polymer concentration treatment of sea water yielded two phases, but water droplets (up to 2 mL)

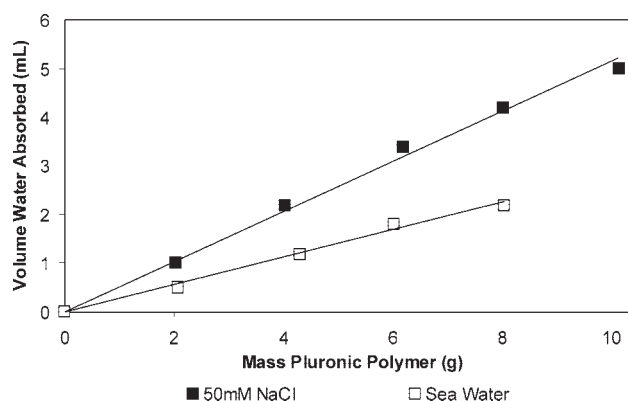


Figure 7 Plot of the volume of water absorbed from 10 mL of 50 mM NaCl and sea water as a function of mass of pluronic PE6200 applied.

TABLE II
Composition of Solution Released by Pluronic Polymer

T ($^{\circ}\text{C}$)	100 mM [CrCl ₃] (mM)	Relative concentration	200 mM [CrCl ₃] (mM)	Relative concentration
Control	100	100%	200	100%
~80	80	80%	212	106%
~90	38	38%	114	57%

remained near the bottom of the pluronic phase, and therefore a volume could not be measured accurately.

Using the measured volume change, the concentration of the remaining aqueous solution and eq. (1), we are again able to calculate the approximate net concentration of salt in the water absorbed into the pluronic phase. Approximately 5 mL of water has moved from the bulk into the 10 g of pluronic polymer, and this has a calculated concentration of $\sim 27 \pm 3$ mM NaCl. Compared to the original solution (50 mM) this represents 52% of the original concentration. This is also of the same order as that observed with 50 mM CrCl₃ (23.8 mM) indicating Cr(+3) is not an unreasonable model for Na⁺. Likewise, sea water is roughly 0.6M salt (primarily NaCl, amongst other ions) and the water volume extracted at 8 g/10 mL pluronic polymer was 2.2 mL (cf 1.3 mL in 200 mM CrCl₃). The concentration of the salt in water removed was calculated to be around (37 ± 10)% that of the bulk.

Water reclamation from pluronic polymer

To experimentally confirm the composition of the water remaining in the pluronic after phase separation at 70°C, the pluronic polymer phases with associated water (after treatment with 100 and 200 mM CrCl₃ respectively) were removed and heated further. According to Alexandris et al. the interfacial area per ethylene oxide unit in LCST polymers is affected as temperature is increased²⁰ above the cloud point and so the separated pluronic polymer may yield more aqueous solution upon further heating. Increasing the temperature of the isolated pluronic polymer phase overnight was found to produce an upper aqueous layer, and this layer could be carefully removed and the CrCl₃ concentration measured, as is described in the Experimental Section. The volumes obtained however, were small at both 80°C and 90°C, around 0.4 and 0.2 mL respectively for the 200 mM CrCl₃ and around 1 mL and 0.5 mL for 100 mM CrCl₃. To ensure no contamination from the bulk pluronic, only small (transparent) samples were taken. The CrCl₃ concentrations were measured and presented in Table II.

In Table II the solution removed from the pluronic phase at temperatures 80, and 90°C are compared, and it was found that the aqueous solutions obtained at higher temperatures had lower Cr(+3) concentrations with the small volume obtained at 90°C having significantly lower concentration than the initial solution. The solution removed from the 100 mM CrCl₃ at 90°C is very close to that calculated above (in Table I). The fact that different concentrations of CrCl₃ are extracted decreasing with increasing temperature shows that the bound water still contains some CrCl₃, importantly there is evidence that some desalination occurs. In practicality, the volumes collected were extremely low, the energy expenditure very high, and the rate of separation (overnight) extremely slow owing to the polymer viscosity. This system is somewhat impractical and so far merely an interesting model.

Our work suggests that desalination processes might be possible using LCST polymers. However, it must be considered that most LCST polymers have very abrupt thermal transitions. Other authors¹³ have already published work on crosslinked poly(-NIPAM/bis-acrylamide) used to dewater industrial waste. Further work by our group will look into designing LCST polymers that can absorb water and release the water by heating, not at a specific 'cloud point' but gradually with increase in temperature, in the range of 20–50°C. We intend to combined different monomers to vary LCST behavior^{12,22,23} and include a crosslinker¹³ to produce polymers with controllable water sorption/desorption properties as a function of temperature and do not dissolve. The PE 6200 pluronic polymer absorbed between 1 and 5 mL of water per 10 g of polymer, this is low and will result in inefficiency and expense, so we will investigate whether other polymers are able to absorb more water, yet yield low salt uptake. Once these polymers are designed and characterized, we will be able to test their applicability to desalination.

Desalination technology using water structuring around LCST polymers will always have the disadvantage of being a slower process than current successful RO membrane desalination. However, a potential niche for any future success could be in agriculture, for example fruit or grain farming where the water need not reach potable standards, merely a

sufficiently low salt concentration. In principle such a technology could benefit dry/arid regions, as they have a steady supply of sunlight, and can utilize brackish water from bores or sea water piped from the ocean. This is also a way that food supplies could in future be stabilized and become insensitive to annual fluctuations in rainfall including droughts. In practice, 0.2–0.3%w/v dissolved solids ($\sim 2\text{--}3$ g/L) is acceptable for raising health livestock²⁴ and an upper limit of 0.35%w/v dissolved solids is the limit for watering plants.²⁵ Thus taking sea water from $\sim 3.5\%$ w/v dissolved solids down to between 6 and 9% its initial value (to 0.20–0.32%w/v dissolved solids) via two successive 20–30% efficient but very low energy steps would allow successful treatment of brackish water or sea water, for use in a range of industrial and agricultural processes. Quite clearly new materials would be required for such an ambitious goal.

CONCLUSIONS

The commercially available polymer pluronic PE6200 was demonstrated to preferentially adsorb water with reduced salt concentration from salt solutions and release some of this water at higher temperatures. This non-ionic polymer was found to extract a salt poor fraction from CrCl_3 solution linearly with amount of polymer used. Similarly, the pluronic polymer was found to extract water from dilute NaCl solution and sea water. Consistent with the observation that the pluronic concentrated the CrCl_3 solutions, it was shown that a small amount of partially desalinated water can be reclaimed by further heating the separated pluronic phase. We feel that this study warrants further research by our group and others in to this area of science.

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