

Adsorptive Removal of Ni(II) from Water Using Alginate-Fixed Water Hyacinth: Effect of Organic Substances

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ABSTRACT

The potential of water hyacinth—*Eichhornia crassipes* biomass immobilized in calcium alginate for the adsorption of Ni(II) from water was studied using the Langmuir and Freundlich adsorption models. The equilibrium adsorption data were obtained at different initial Ni(II) concentrations = 0.05 - 2 mg/L, temperature = 30°C ± 0.2°C, agitation rate = 150 rpm, equilibration time = 3 hr, biosorbent dose = 4 g/L, and pH range = 4 - 7.4. Langmuir isotherms gave monolayer sorption capacities (q_m) of 26.5, 44.3 and 53.0 mg/g for the removal of Ni(II) in the presence of methanol, without added organic substance, and in the presence of acetonitrile, respectively. These findings were also confirmed by the trend in the Freundlich isotherm parameter ($1/n < 1$). Interpretation of the sorption data in terms of the separation factor, S_F , suggested that the removal of Ni(II) from water mainly occurred through a chemisorption mechanism. Desorption experiments to recover Ni(II) from the adsorbent showed that highest amounts of the metal could be removed from the adsorbent when previously adsorbed in the presence of 1-(2-Thiazolylazo)-2-naphthol (TAN) (>90%). The results from these studies indicated that *E. crassipes* fixed on calcium alginate beads is a viable metal enrichment media that can be used freely immersed in solution to achieve very high adsorption capacities and possible preconcentration of Ni(II) in aquatic environments.

Keywords: *Eichhornia crassipes*; Adsorption; Langmuir Model; Freundlich Model; Nickel

1. Introduction

Biosorption of heavy metals, which is the passive uptake of the metals by dead/inactive biological materials or by materials derived from biological sources, has attracted significant attention during the past few decades as an alternative to the conventional methods of heavy metal removal from aquatic systems [1-5]. Initial incentives of the process include low cost of the biosorbents, great efficiency for metal removal at low concentration, potential for biosorbent regeneration and metal recovery, high velocity of sorption and desorption, limited generation of secondary residues and more environmentally friendly life cycle of the material (e.g. easy to eliminate compared to conventional resins) [4,6].

In many cases, biosorption studies have been carried out using native biomasses and products obtained poly-

saccharides and glycoproteins. Even though they have several advantages, such as high rapid steady-state attainment, high biosorption capacity, less process cost and good particle mass transfer, they often suffer several drawbacks. Major limitations include low density, poor mechanical strength, little rigidity, solid-liquid separation problems, possible biomass swelling, inability to regenerate/reuse and development of high pressure drop in the column mode [3]. Several established techniques have been used to make biosorbents suitable for continuous-flow applications. Among these, immobilisation techniques such as entrapment and cross-linking have been found to be practical for biosorption [7]. Several advantages of biomass fixation in polymeric material have been realised when applied in packed or fluidised bed reactors. These include the control of particle size, regeneration and reuse of the biomass, easy separation of biomass and effluent, high biomass loading and minimal

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clogging under continuous-flow [8]. The immobilisation concept has been applied using several types of biosorbents in an effort to improve the mechanical properties of the biosorbents for practical applications, whilst maintaining favourable sorption equilibria [9-12].

Matrices that have been commonly used in biosorbent immobilization include sodium alginate [13], polysulphone [14], and polyurethane [8]. For environmental application, the choice of the polymeric matrix is important in that it determines the mechanical strength and chemical resistance to successive sorption-regeneration cycles.

Among plants that have been studied for their uptake of heavy metals from aquatic systems, *E. crassipes* has been shown to possess the ability for the sorption of several heavy metals [15-17]. Schneider *et al.* [15], reported that *E. crassipes* roots accumulated very high levels of several divalent metal ions in the living plant. More recently, *E. crassipes* immobilized in calcium alginate has been shown to remove high levels of Cu(II) [13].

Removal of metal ions in the presence of other metals (competitive adsorption) has been investigated by several authors [17-20]. In many cases, metal interactions have been shown to be antagonistic, *i.e.*, sorption of one metal decreases in the presence of the other. Although metal interference effects have been significantly studied, there is no evidence for the study of the effect of organic substances during metal biosorption. In particular, the effect of organic substances on the removal of metal ions by *E. crassipes* has not been documented. The present work has therefore been addressed to the study of the effect of selected organic substances: acetonitrile, methanol and 1-(2-Thiazolylazo)-2-naphthol (TAN) on the sorption dynamics of Ni(II) ions onto *E. crassipes* fixed on alginate.

2. Experimental

2.1. Instrument

Metal ion concentration was determined using a Varian 55B Series Atomic Absorption Spectrometer (Australia). The pHs were measured using a Jenway 3510 pH meter (UK), equipped with a combined glass electrode, which was calibrated regularly with buffer solutions (pH = 4.0 and 7.0). Agitation was achieved using a Griffin multi-functional shaker (Germany) and temperature was controlled using a KDB temperature bath supplied by Memmert Holdings, Pvt Ltd (India).

2.2. Chemicals and Reagents

All chemicals used in the experiments: $(\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O})$, 1-(2-Thiazolylazo)-2-naphthol, supplied by Sigma-Aldrich, Germany; NaOH, HCl, HNO_3 , $\text{KHC}_8\text{H}_4\text{O}_4$, KH_2PO_4 , Na_2HPO_4 , acetonitrile and methanol supplied by Sky Labs (Lenasia, South Africa), were of analytical reagent

grade. Deionised water of conductivity $0.001 \text{ S} \cdot \text{m}^{-1}$ (supplied by Trojan Nickel Mine, Bindura) was used for all dilutions. Buffer solutions of pHs 4, 6.8 and 7.4 were prepared by dissolving definite weights (singly or mixed) of $\text{KHC}_8\text{H}_4\text{O}_4$, KH_2PO_4 , Na_2HPO_4 and dissolving in 1000 ml of distilled water at 25°C .

2.3. Procedure

2.3.1. Alginate-*E. crassipes* Bead Fabrication

Mature *E. crassipes* samples were collected from Lake Chivero, Harare, and treated as previously reported [14]. The samples were washed three times on sight and carefully packed in polythene bags previously cleaned by soaking in 10% nitric acid. The plants were then placed in a fishpond filled with tap water at Bindura University of Science Education and allowed to grow. After 4 weeks the parent plants were harvested, the pond refilled with fresh water and the *E. crassipes* replaced by daughter plants reproduced during the period. Five grams of NH_4NO_3 fertiliser was added in the pond to promote shoot development. Plants were harvested after 4 weeks, and roots were separated and washed three times with running tap water and twice with deionised water. They were then sun-dried for 2 days before being oven-dried at 65°C for 12 h. Dried samples were crushed, ground with a mortar and pestle and sieved (size fraction of 2.5 mm). The samples were then acid-treated with 0.1 M HNO_3 for 8 h followed by washing with deionised water until a pH of 7 was attained. The biomass was then oven-dried at 65°C for 24 h, and the dried powder was stored in polythene bags until used. To prepare alginate-*E. crassipes* beads, biomass powder (2 g) was blended with 100 mL of 2% w/v sodium alginate solution and extruded dropwise into a 0.1 M calcium chloride solution through a pipette tip. The formed beads were then hardened by placing them in 2% calcium chloride solution for 12 h. Those that floated on the surface were discarded. The remaining beads were washed in deionised water by agitation at 100 rpm in 250-mL conical flasks for 30 min, discarding the solution and then repeating the process 5 times. After removing residual water with paper towels, the beads were dried gradually at room temperature to constant weight and then used for further experiments.

2.3.2. Adsorption of Ni(II) onto Alginate-*E. crassipes* Beads

The adsorption experiments were carried out using the batch equilibrium technique at 30°C , varying pH (4, 6.8 and 7.4), biomass of $4 \text{ g} \cdot \text{L}^{-1}$ and varying initial Ni(II) concentration ($0.03 - 2.00 \text{ mg} \cdot \text{L}^{-1}$). In a given experiment, alginate-*E. crassipes* beads (400 mg) were weighed into a 250 ml volumetric flask. Ni(II) solution (100 ml) (in the range $0.03 \text{ mg/L} - 2 \text{ mg/L}$) and 5 ml of buffer solution was added to the flask. The mixture was shaken

mechanically for 3 hr and the solution filtered and the concentration of Ni(II) in the filtrate was determined by flame atomic absorption spectrometry (FAAS). To determine the effect of TAN, 5 ml of 0.12% TAN and 5 ml of buffer solution were added to the Ni(II) solution to give a total volume of 100 ml. For acetonitrile and methanol, 5 ml of the organic solvents and 5 ml of buffer solutions were added to the Ni(II) solution as described for TAN. Biosorption experiments were performed as described for the sorption in the absence of organic substances. For any subsequent experiments to optimise metal uptake, this sorption procedure was repeated. For desorption, the alginate-*E. crassipes* beads were placed in 5 ml of 0.1 M HNO₃. The mixture was then incubated at 25°C for 30 min in a water bath with shaking at 150 rpm and filtered through 0.45 µm membrane filter. Five millilitres of the filtrate were diluted to 50 ml with deionised water. The metal concentration was then determined by FAAS.

3. Results and Discussion

3.1. Adsorption Isotherms

Analysis of equilibrium data on a specific adsorption isotherm is of significance for comparing different sorbents under different experimental conditions. It is therefore important to establish the most appropriate correlation for the equilibrium curves. The Langmuir and Freundlich isotherms, are among the two-parameter equations which have been commonly used to characterise sorption data. The Freundlich model suggests adsorbed monolayers, where the interactions among adjacent molecules that are adsorbed: the energy distribution is heterogeneous due to the diversity of the binding sites and the nature of the adsorbed metallic ions. The Langmuir model considers an adsorbed monolayer with homogeneous distribution of binding sites and adsorption energy, without interaction among the adsorbed molecules. One linearized form of the Langmuir model is shown in Equation (1):

$$C_e/q_e = 1/K_L q_m + C_e/q_m \quad (1)$$

where q_e (mg/g) is the equilibrium sorption capacity, q_m is the monolayer saturation at equilibrium, whereas K_L corresponds to the concentration at which the amount of Ni(II) ion, bound to the alginate-*E. crassipes* beads, is equal to $q_m/2$. This indicates the affinity of the ions to bind with the biomass.

The linear form of the Freundlich adsorption isotherm is shown in Equation (2):

$$\ln q_e = \ln K_F + 1/n \ln C_e \quad (2)$$

where K_F and $1/n$ indicate the adsorption capacity and adsorption intensity, respectively.

3.1.1. Langmuir

Typical Langmuir isotherm plots obtained by fitting the experimental data are shown in **Figure 1**. It can be seen, for example that the data fitted the model for Ni(II) removal in the absence of added interferent substances at pH 4, whereas the data did not fit the isotherm at higher pH (6.8) and in the presence of TAN. **Table 1** shows the Langmuir isotherm parameters for data the adequately fitted the the maximum adsorption capacities were 26.5, 53.0, and 44.3 mg/g for Ni(II) adsorption in the presence of methanol, acetonitrile and in the absence of organic substances, respectively. These values indicate that Ni(II) ions were effectively removed from water by the alginate-*E. crassipes* beads in the presence and absence methanol and acetone. High q_m values confirms stronger bonding affinity of alginate *E. crassipes* beads to Ni(II). The Langmuir parameter, K_L , showed that the biosorbent had a high ratio of Ni(II)-adsorbed to Ni(II)-desorbed, which reflected the high affinity of the sorbent for sorbate. However, the adsorption capacity appears to be suppressed in the presence of methanol, and enhanced in the presence of acetonitrile.

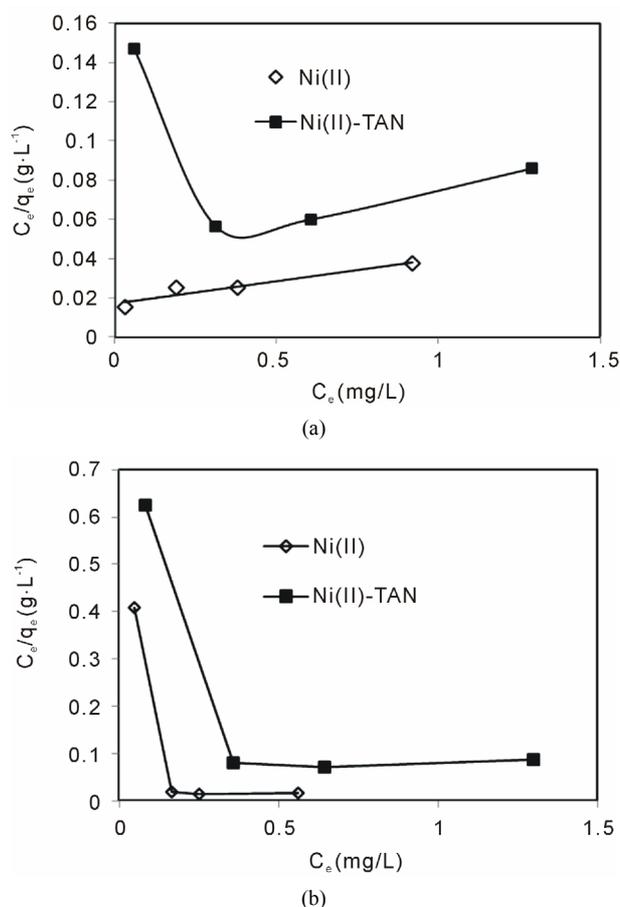


Figure 1. Langmuir adsorption isotherm plots for the adsorption of Ni(II) onto alginate-*E. crassipes* beads in the presence and absence of TAN. (a) pH 4, (b) pH 6.8.

Table 1. Langmuir and Freundlich isotherm parameters for the adsorption of Ni(II) onto alginate-*E. crassipes* beads.

Langmuir parameters			
Adsorbate	q_m	K_L	R^2
Ni(II)-acetonitrile	53.0	1.46	0.9997
Ni(II)	44.3	1.33	0.9258
Ni(II)-methanol	26.5	0.81	0.9687
Ni(II)-methanol-TAN	-	-	-
Ni(II)-acetonitrile-TAN	-	-	-
Ni(II)-TAN	-	-	-
Freundlich parameters			
Adsorbate	$1/n$	K_F	R^2
Ni(II)-acetonitrile	0.50	5.86	0.9987
Ni(II)	0.55	4.05	0.9105
Ni(II)-methanol	0.61	3.40	0.9988
Ni(II)-methanol-TAN	0.98	1.20	0.9825
Ni(II)-acetonitrile-TAN	2.37	0.25	0.9297
Ni(II)-TAN	2.96	0.07	0.9546

Further insight into the favourability or unfavourability of the adsorption was obtained by using an essential feature of the Langmuir isotherm called the separation factor, S_F , which is defined by the following equation:

$$S_F = \frac{1}{1 + K_L C_0} \quad (3)$$

where S_F is a dimensionless separation factor, C_0 is the initial metal concentration and K_L is the Langmuir constant. Various scenarios exist depending on the value of S_F :

$S_F > 1$: unfavourable interaction between adsorbate and adsorbent exists, and, in that case, adsorption may not be possible, $S_F = 1$: a linear relationship between adsorbate and adsorbent exists, and the mechanism characterizing the interactive process is a mixture of physisorption, and $S_F = 0$: there is an indefinite sticking between sorbate and adsorbent, and chemisorption predominates.

The data for separation factor plotted in **Figure 2** showed that the S_F values were < 1 at all concentrations, indicating that adsorption was favourable for the removal of Ni(II) by alginate-*E. crassipes* beads in the absence and in the presence of the organic substances. The results also confirm that the adsorption process became more favourable as the initial metal concentration increased, thus suggesting that high concentrations of Ni(II) in effluents may not be the limiting factor in their removal using *E. crassipes* fixed on alginate.

The effect of acetonitrile on Ni(II) removal from water by alginate-*E. crassipes* beads was further probed by

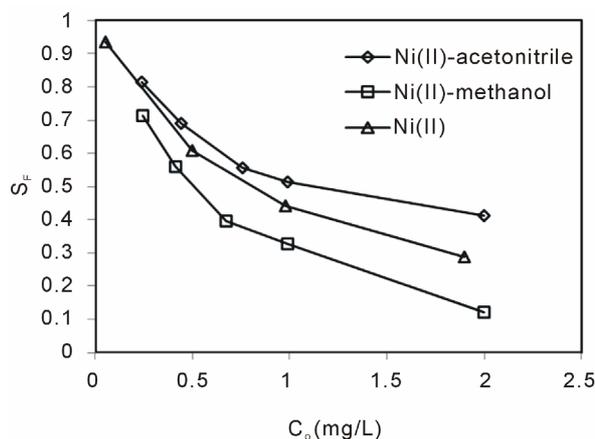


Figure 2. A plot of sorption favourability (S_F) vs initial metal ion concentration for the removal of Ni(II) ions by alginate-*E. crassipes* beads. pH 4.0, T = 30°C ± 0.2°C, agitation rate = 150 rpm, t = 3 hr, $C_0 = 0.05 - 2$ mg/L, biosorbent dose = 4 g/L.

computing the difference between the concentration of Ni(II) remaining in solution after adsorption in the absence and in the presence of acetonitrile

$$(\Delta C_{ads} = C_{ads, Ni(II)} - C_{ads, Ni(II)-acetonitrile}) \quad (4)$$

and the plots are shown in **Figure 3**.

There was a significant correlation between ΔC_{ads} for both solutions ($R^2 = 0.9922 - 0.9996$) and difference between the slopes for the plots was found to be constant at 1.05. These findings further point to a modification of the adsorption capacity of the adsorbent for the sorption of Ni(II) in the presence of acetonitrile in the pH range studied.

3.1.2. Freundlich

The Freundlich isotherm is convex for $1/n < 1$, linear for $1/n = 1$, and concave for $1/n > 1$. The lower the value of $1/n$, the higher will be the affinity and heterogeneity of the adsorbent sites. Typical Freundlich isotherm plots are shown in **Figure 4** and isotherm parameters given in **Table 1**. It can be seen that the $1/n$ parameter was less than unity for Ni(II) adsorption in the acetonitrile, methanol, methanol and TAN, and in the absence interferents. The trend in the Freundlich isotherm parameters, $1/n$ and K_F , confirms the trend observed for the Langmuir isotherm parameters, q_m and K_L , as the high q_m and K_L values observed for Ni(II)-acetonitrile (53.0 mg/g and 1.46 respectively) are consistent with the lower $1/n$ and higher K_F values (0.50 and 5.86, respectively), whilst the lower q_m and K_L values for Ni(II)-methanol (26.5 mg/g and 0.81, respectively) are also consistent with the higher value of $1/n$ (0.61) and lower value of K_F (3.40). Furthermore, it can be noted that although the sorption data obtained for Ni(II) in the presence of TAN, methanol-

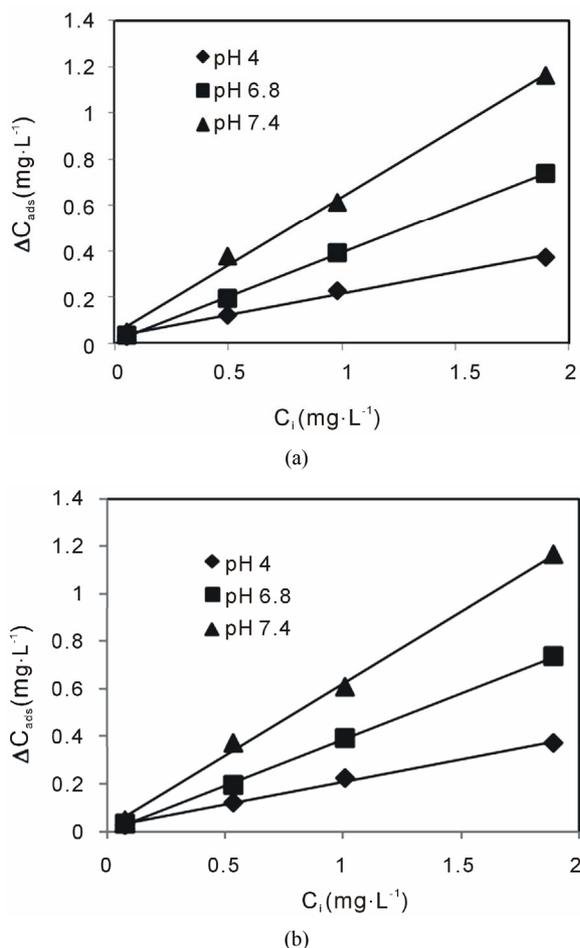


Figure 3. Plots of ΔC_{ads} against initial metal concentration for the adsorption of (a) Ni(II) and (b) Ni(II)-TAN onto alginate-*E. crassipes* beads. pH 4.0, $T = 30^{\circ}\text{C} \pm 0.2^{\circ}\text{C}$, agitation rate = 150 rpm, $t = 3$ hr, $C_0 = 0.05 - 2$ mg/L, biosorbent dose = 4 g/L.

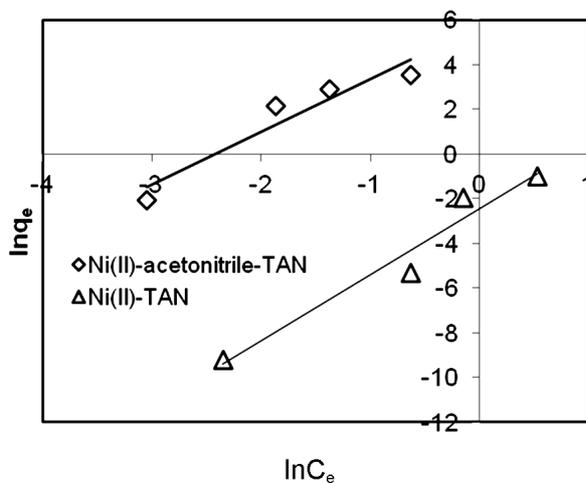


Figure 4. Freundlich adsorption isotherms for Ni(II)-TAN and Ni(II)-acetonitrile-TAN onto alginate-*E. crassipes* beads. pH 4.0, $T = 30^{\circ}\text{C} \pm 0.2^{\circ}\text{C}$, agitation rate = 150 rpm, $t = 3$ hr, $C_0 = 0.05 - 2$ mg/L, biosorbent dose = 4 g/L.

TAN, and acetonitrile-TAN did not fit the Langmuir model, the same data was adequately described by the Freundlich model, which indicates the predominance of multilayer adsorption. Therefore, the nature of Ni(II) adsorption was influenced by the presence of the organic substances and their respective combinations.

3.2. Ni(II) Desorption

Desorption is very much necessary when the biomass preparation/generation is costly. In that case, it is possible to minimise the process cost and also the dependency of the process on a continuous supply of the biosorbent. To achieve optimum desorption, it is necessary to have a proper selection of elutants, which strongly depends on the type of biosorbent and the mechanism of biosorption. In the present study, desorption experiments were performed to investigate the effect of pH on the recovery of Ni(II) after desorption using 2 M HNO_3 . The results shown in Figure 5 indicate that high recoveries were possible for the Ni(II) in the presence of TAN (>90%).

4. Conclusion

Novel, high sorption capacity alginate-*E. crassipes* beads were used to remove Ni(II) from aqueous solution. The current paper demonstrated how pH and the presence of methanol, acetonitrile and TAN affected the adsorption and desorption of Ni(II). In the end, it was shown that *E. crassipes* immobilized in calcium alginate can be successfully used for high removal of Ni(II) from water and hence offers a potential preconcentration of the metal from aqueous solution.

5. Acknowledgements

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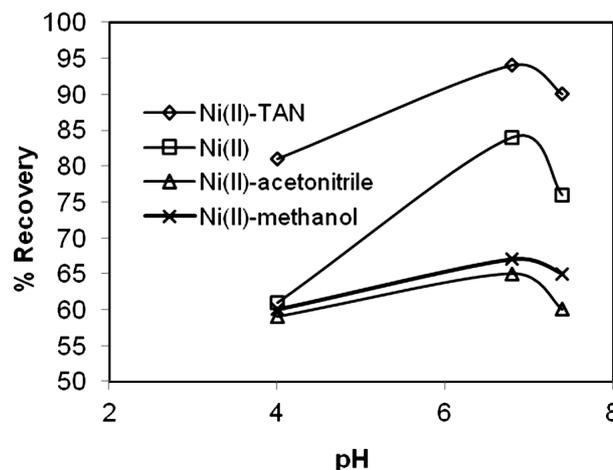


Figure 5. Effect of pH on the recovery of Ni(II) in the presence and absence of TAN, methanol and acetonitrile.

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