Full length article

Dewatering of super absorbent polymers: Alternatives to thermal desorption by liquid phase extraction using dimethyl ether

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ABSTRACT

The high saturation potential of highly hygroscopic desiccants makes their drying potential reuse an energetically unfavorable challenge. Perhaps there are other ways beyond dehydration the extract saturated water from these structures. That was the premise of the proposed experiments in which an existing method was redesigned in which condensed Dimethyl Ether (DME) was used aqueous fluids from commercial grade superabsorbent polymers (SAPs) similar to those found in commercial diapers. It was hypothesized that liquid extraction could be energetically less intensive than thermally dehydrating the SAP. By toggling between pressures above and below 0.6 MPa at room temperature, using gravity assistance, and nitrogen gas as driving forces, the DME condenses and interacts with the saturated SAP. The system successfully dewatered both DI water and saline saturated SAP. Roughly 90% of deionized water and ~80% of artificial urine are extractable from SAP when the chamber was pressurized 3 times. The salts in the artificial urine also conveyed in the aqueous extract and not concentrated in the dehydrated SAP. Energy assessments linked with each DME loading consumed 12.6 kJ or ~5 kJ/g of water extracted, more than required for thermal evaporation. The dehydrated superabsorbent polymer structure appears morphologically different by microscopy, but it can re-swell up to 35 times its dry mass in water and 15 times its mass in artificial urine. Overall, aqueous phase extraction using condensed DME dewaters saturated superabsorbent polymers allowing their reuse as other forms of desiccants but it is less efficient than by dehydration.

1. Introduction

Much consumer waste conveyed to landfills stems from absorbent hygiene products (AHPs), such as diapers, feminine pads, and adult incontinence products. Personal care products are not made from a single material, but are complex, integrated multilayered structures including many different materials. The materials include polypropylene and polyethylene layers as structural liners, perforated nonwoven structures for fluid wicking, tapes and adhesives for functional use, and binding functional layers together, and cotton for cushioning and comfort. For urine absorption, the major functional additive is a superabsorbent polymer (SAP) based on polymerized acrylic acid. SAP is produced in a particulate form and ensembles of particles are enmeshed into a pocket within the cloth. These personal care products are essentially non-recyclable given that they contain bodily fluids resulting in potential infection hazards.

Fractionating these integrated products into individually recyclable components has started and needs to be expanded ("Fatersmart: Commercial Diaper Recycling," 2020). The large fluid absorption capacity of the SAP makes these products a larger mass fraction of the whole waste stream. The US EPA notes that in 2018, 4.1 million US tons of diapers were generated and of that, 4.3% of the diaper mass was recycled and 77% of those were ultimately landfilled, the rest being incinerated (EPA, 2020). Diapers and other personal care products also present as a more prominent component of solid waste. Methods have been proposed to reduce diapers into smaller components and separate the different

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components based on specific gravity and to dehydrate the saturated superabsorbent polymer using calcium chloride and other salts to dewater the saturated desiccant (Kanda et al., 2010; Park et al., 2014; Cho et al., 2002; ;). Kanda et al. used condensed dimethyl ether extraction as an alternative drying method, but they processed complete diapers and some of the other polymer players were also soluble in DME. If the diaper can be deconstructed into material streams that can be co-recycled, similar to the Fater process ("Fatersmart: Commercial Diaper Recycling," 2020), the hydrophobic polyolefin layers could be separated and recyclable as sheets while allowing the SAP to be dehydrated and reused. If isolating and dehydrating saturated SAP isn't realistic using a low amount of energy, there might be a larger economic driving force to invest in recycling personal care products through their component isolation. Patents have already been granted for diaper recycling based on source separation and then an involved cleansing process mentioning dehydration, but with no energy estimates (Bartlett, 1992).

Simple fluid extraction strategies for water saturated products are energy intensive (40 kJ/mole of water, or equivalently, 2.26 kJ/g of water) which makes diaper recycling cost prohibitive. The numbers look even worse when most of the saturated desiccant mass is water. Perhaps other routes of extraction through liquid phase extraction is possible.

Dimethyl Ether (DME) is a viable solvent for liquid phase extraction. DME is a useful green solvent that can be easily stored and transported in a condensed liquid state at pressures higher than 0.6 MPa. Due to its chemical structure, DME is reasonably stable, although there is some reference to DME forming peroxides and other unstable, longer chain ethers (Ohrman and Pettersson, 2013). Moreover, DME does not emit toxic gasses, such as nitrates or sulfates during incineration (Kanda et al., 2008, 2011,). DME can also act as a potent extraction solvent regulated for multicomponent solids and gels. DME has been used as a solvent to extract oil components in *E. gracilis*, carotenoids and lipids from algae and water from porous coal(Ahmad and Liu, 2017; Kanda et al., 2015;) Further, DME has been designated as a Generally Recognized as Safe (GRAS) extraction solvent for food by the Food and Drug Administration (US FDA) in 2017 (Adams, 2017). It was also shown that the isolates of ferulic acid-conjugated lipids extractable by DME were beneficial for human health (Hara et al., 2016). Compared to traditional Soxhlet and extractions based on isopropyl alcohol, DME pressure-regulated aqueous extractions could potentially save energy time and cost.

The allure of dimethyl ether as a solvent is validated by the solubility of water in DME (~8%) as it condenses at 0.6 MPa. As a result, pressure condenses DME and raises its solvation capacity for water at ambient temperature. After allowing condensed DME to interact with saturated solids and separating the liquid and solid components, the DME evaporation will phase separate out the water fraction leaving a much drier solid substrate in its wake. DME boils at −24 °C at atmospheric pressure. Below that temperature, DME condenses and is known for its significant solubility for water at ambient pressure. Thus, by cycling between high and low pressure, the DME acts like a refrigerant gas in the system going through its condensation and evaporation cycles soaking up water with each pressurization cycle.

Refinements seemed very possible as did quantification to resolve the energy cost in recharging superabsorbent polymer. Using better seals, increasing the number of chambers in the system through more check valves and installing a collection reservoir offered the potential to capture vented DME and recycle it in batch mode. Other schemes like centrifugation, are effective but rather time consuming and with associated energy costs of upwards to 53,000 MJ/kg water or ~1.5 KW-hr/kg (Apaolaza et al., 2015). Considered here are the construction and use of a smaller scale DME extraction system based on a system from Kanda et al. and a focus only on exposing the SAP, not the entire diaper.

2. Materials and methods

2.1. Super absorbent polymer

The superabsorbent polymer used was an Aqualic CA type L700 provided by Procter and Gamble Corp (P&G) [13]. The SAP was a hygiene-grade particulate form, synthesized from cross-linked sodium acrylate copolymer, stored at room temperature before usage.

2.2. Dimethyl ether extraction system

A prototype Dimethyl Ether (DME) Extraction system was adapted at smaller scale based on a design from Kanda (Kanda et al., 2010). The conveyance system consisted of cylinders, valves, pressure gauges, and connectors capable of higher pressure, shown in Fig. 1. Experiments were limited to 1–2 gs of SAP, as opposed to the assessment of an entire diaper (Kanda et al., 2010) The threaded hardware was all made from stainless steel purchased from Swagelok. The gauges and valves offered more potential to separate the fluid extracts than the high-volume extractor from Kanda operating in the liters/minute range. Mesh filters (86 μm) housed the saturated superabsorbent polymer obtained from Ted Pella, Inc. The DME was obtained from Sigma Aldrich and deionized water was collected from an internal source.

Among independent variables were the number of compression cycles or chargings used, and two types of saturation solutions used for the superabsorbent polymer, deionized water and an artificial urine mimic that contained dissolved salts. The presence of ions reduced the saturation potential of SAP that has lower aqueous swelling capacity with increasing ionic strength. In a limited number of scout experiments following the 3 nitrogen chargings, a second DME injection was also done to gage further extraction potential.

2.3. Saturation solutions

The SAP was exposed to two different solvation solutions before being dehydrated with dimethyl ether. In addition to deionized water, a higher ionic strength aqueous solution was formulated based on recipes that represent artificial urine. Higher ionic strength solutions reduce the fluid saturation threshold of the SAP and that affects the energetics of the extraction and the throughput of SAP dehydration. The artificial urine mixture included 1 L of deionized water, 24.25 g of urea, 16 g of Sodium Chloride (NaCl), and 6.4 g of Tris sodium Phosphate Anhydrous
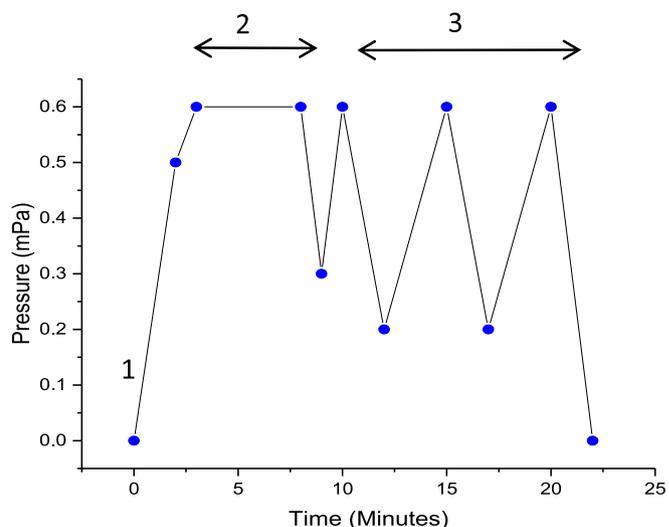

Fig. 1. Schematic of the DME extraction set up. The separation cylinder is aligned under the extraction column to maximize transport from the first column to the second column when the valve between them is opened.
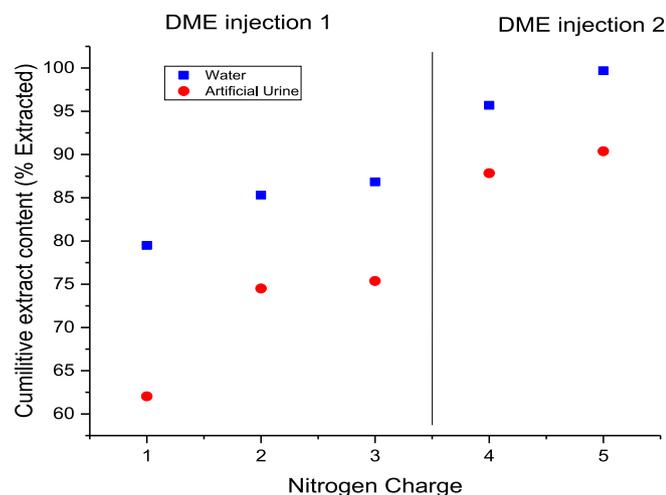
Fig. 2. Dynamic Pressure monitored during DME extracted. The pressure gage was mounted to the first cylinder. The two-step pressure rise initially is due to first introducing DME and then nitrogen in a first charging cycle. Each subsequent re-pressurization is another charging cycle that sounds very reasonable.

buffer, which were all obtained from Sigma Aldrich and based on a recipe from Burdick (Burdick, 2020). After all the ingredients were dissolved in the water, the pH was measured and then adjusted to 7.5 using Hydrochloric Acid (HCl) (Sigma Aldrich). The artificial urine was stored at 10 °C before its use. The electrical conductivity of the artificial urine and the extracted solutions were assessed using a Thermo Scientific Orion Versastar Pro-conductivity meter.

To expose the SAP to the saturation solutions, a nylon mesh was fashioned with a pocket to contain the SAP. The mesh was weighed initially and approximately 1 g of dry SAP was added into the mesh. The ends of the mesh were then sealed using iron ribbons to prevent any loss of SAP. The mesh was inserted into a stainless steel cylinder and the cylinder was immersed in 100 mL of either deionized water or the artificial urine. After 24 h, the fluid portion that was not absorbed into the mesh was decanted. The new mass of the swollen and saturated SAP in the mesh was recorded before extraction.

A traditional tea bag method was used to track SAP saturation. SAP was weighed (W_S) and folded into a sealed and weighed nylon mesh (W_N). The mesh was immersed fully into artificial urine for 24 h. The free liquid was decanted and the total mass was measured (W_T). The wetted mesh with saturated SAP was extracted and hung in the air for 10 min to allow the excess solvent to drip off. The mass difference was divided by the original weight of SAP. Both the tea bag method and measuring during extraction showed a consistent result of swelling ability.

$$\text{Swelling Ability (\%)} = \left(\frac{W_T - (w_N + W_S)}{W_S}\right) \times 100\% \quad (1)$$

2.4. Extraction protocol

The extraction system condenses DME at higher pressure (> 5 bar, 0.5 MPa). DME can be cycled between the gas and liquid phases and if condensed DME extracts water contained in desiccant, condensed DME will dry the SAP as it is routed through. An example experiment showing the dynamic pressure in the extraction cylinder is shown in

More precisely, saturated SAP /mesh samples were inserted into the extraction cylinder, sealed, and pressurized by both nitrogen (N$_2$)and 30 gs of dimethyl ether (DME) to perform each extraction. First, the DME reservoir was opened to the extraction column and allowed to remain open until the pressure was equilibrated to 5 bar (0.5 MPa). Immediately following DME injection, the DME valve was closed and the vessel was further pressurized to 6 bar (0.6 MPa) using N$_2$. The increased pressure

condenses DME into a liquid state at room temperature. The condensed DME and SAP were allowed to interact undisturbed for 5 min. The assembly of sample cylinders containing the DME and the mesh were then aligned vertically to allow gravity to help isolate the fluid contents in the upper chamber from the collection reservoir below. After 5 min, the valve from the upper (first) cylinder to the lower (second) cylinder was opened while adding more N$_2$ to push the contents down isolating the fluid from the extraction column (This step is referred to as a nitrogen or "N$_2$ charge"). The check valve between cylinders was then closed and the first cylinder was once again re-raised to 6 bar pressure by N$_2$. The second cylinder was opened to a third cylinder to collect the aqueous extracted at the bottom while the DME was evaporated using a release valve to vent it at the top. The "N$_2$ charge" step was performed as many as three times to achieve maximum extraction. The dehydrated specimens were also re-saturated to gage both further mass uptake and the reswelling capacity post-extraction. The fourth pressurization is linked with the third gaseous charging.

The overall period for each dehydration experiment was ~20 min with each valve release and recharge taking ~4 min, respectively. Once DME exposure was complete by up to three N$_2$ chargings, the apparatus was separated, and a residual mass of the mesh post-extraction was measured, after which the sample was placed in a 40 °C oven to remove any residual moisture and stored. One could also separately measure the isolated water fraction collected from the vaporization of the DME.

3. Results

3.1. Water extraction from SAP

Extraction was performed on superabsorbent polymers saturated with either deionized water or the artificial urine. After one injection of DME and three N$_2$ charges, ~ 87% of deionized water is extractable on average and about 75% of artificial urine is extractable. Most of the water is extracted after the first N$_2$ charging. Comparing the two saturation solutions, the artificial urine extractions had a similar electrical conductivity and acidity to the original solution shown in Table 1. Dissolved salts in the artificial urine were also conveyed during extraction since the conductivity of the post extraction fluid would have been less if the salts were not conveyed. The average water extraction efficiency per each N$_2$ charge using one injection of DME are also shown in Fig. 3. The first N$_2$ charge extracts by far the most water. Total extraction rates rise with each successive nitrogen cycle but with diminishing returns as less water is available for extraction. Obviously, the extraction cycle time would be faster with only one N$_2$ charge, but larger extraction rates require more than one cycle.

Comparing the amount of water extracted with other techniques, Park et al. evaluated the use of higher salinity solutions to drive water from saturated solutions using time scales ranging from 10 min to several hours and varying the salt concentration in solution(Park et al., 2014). They found similar efficiencies to DME liquid phase extraction. In general, ~70% water extraction was noted using 10 min of salt exposure and up to 90% extraction using equilibration times from an hour to hours. By using relatively inexpensive salts like calcium chloride, they achieved similar extraction potential although they were still using ovens to thermally desorb the rest of the water that was not expelled. The DME extraction system seems to equilibrate more quickly with a

Table 1
Summarized pH and conductivity measurements of artificial urine reservoir preextraction and in the extraction volume after the DME and nitrogen chargings. There is no difference in the solutions suggesting salt transfer during DME extraction.

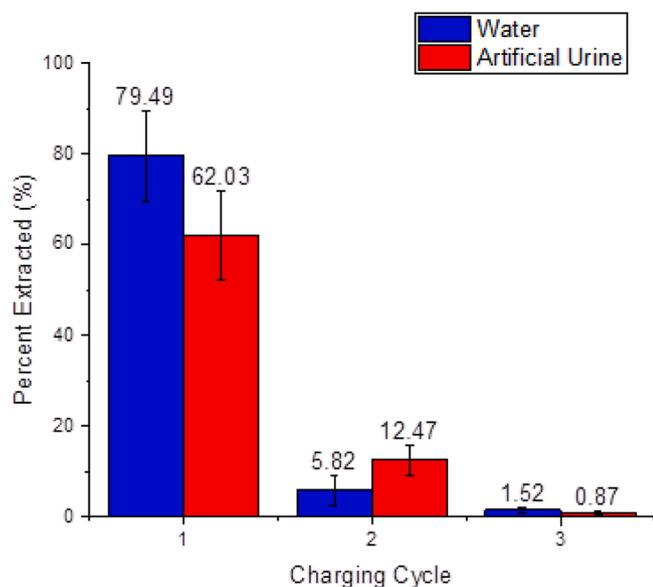| Artificial Urine	pH	Specific conductivity (mS/cm)
Before Extraction	7.6	23.1
After Extraction	7.7	25.8

Fig. 3. Average extraction efficiency shown for water and artificial urine with each Nitrogen charging cycle after one 30 g injection of DME. Error bars represent one standard deviation for $n = 30$ extractions.

similar extraction potential and could be processed faster, but it requires pressure.

SAP swells more in water than in the artificial urine solution with higher ionic strength. It was found that the DME dehydrated SAP after extraction still swelled to about 35 times its mass in water and to about 15 times its mass in artificial urine. Upon taking the DME-dried samples and re-equilibrating them in water and artificial urine, the desiccant continues to function through swelling with the expected response as shown in Fig. 4. The residual water contained in the superabsorbent polymer following the DME-based liquid phase extraction, raises the mass of the initial sample before reswelling and lowers the total water capacity when reswelling. It is also conceivable that DME is altering the water absorption capacity of the resin which would require more complete characterization.

The significant decrease in re-swelling capacity of the dehydrated SAP compared to the virgin material suggests that the SAP structure is being changed through the extraction.
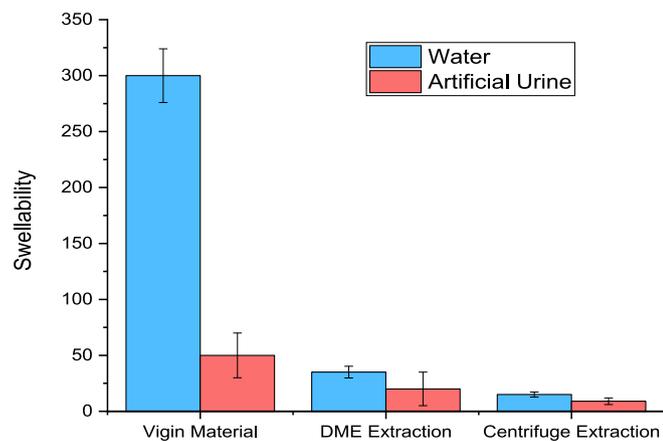

Fig. 4. Re-swellability of SAP after DME extraction. *Re*-swellability represents a percentage compared to the SAP dry mass in Eq. (1). Error bars represent one standard deviation for $n = 30$ experiments duly noted.

3.2. Energy estimation and effectivity of regeneration process

The hope for DME-aided SAP extraction is that it is less energy intensive and more rapid than water isolation by thermal dehydration (2.25 kJ/g of H_2O). To consider the overall energy cost, several factors were estimated. They included the heat of DME evaporation, the enthalpy for removing DME, the relative solubilities of water in DME and visa versa. The enthalpy of vaporization for DME reported in the literature is 0.42 kJ/g[5] In our exploratory studies, ~30 g of DME were injected, corresponding to ~12.6 kJ for evaporating DME in each extraction cycle. This is somewhat simplistic as it does not account for the energy losses on venting if DME is acting as a refrigerant. With ~80% of water extracted (~2.4 g from 3 gs swelling the SAP) from the first cycle (10 min), the energy expended is ~5 kJ/g which is more than the heat to vaporize water on its own.

In a more realistic set up, the energy required for extraction would correspond to the refrigeration cycle of DME, and that would be equivalent to the work of compression. In an ideal cycle this can be calculated with Eq. (2).

$$\text{Work of Compression (kJ)} = \dot{m}_F * (h_2 - h_1) \tag{2}$$

Where:

$\dot{m}_F$ is the mass of the fluid
h_2 is the enthalpy of the fluid at state 2
h_1 is the enthalpy of the fluid at state 1

For our case with ~ 30 g of DME, state 1 corresponds to the saturated vapor at 0.5 MPa with an enthalpy of approximately 620 kJ/kg, while state 2 corresponds to the saturated liquid at 0.6 MPa with an enthalpy of approximately 225 kJ/kg (Teng et al., 2004). This means the proposed system would require 11.85 kJ or 4.94 kJ/g H_2O removed. This energy cost is still significantly higher than the enthalpy of evaporation of water (2.26 kJ/g) while not considering the inefficiencies involved in a compressor.

Confirming other efforts on the energetics of extraction, work by Tallon et al. comparing the ternary system CO_2-DME-Water at high pressure is instructive. (Tallon and Fenton, 2010). At 313 K, they found the solubility of water in DME at ~19% mole%. Raising the temperature to 333 K increased the solubility to ~24% mole, albeit at higher pressures. For a mixture of 1 mole of DME and 1 mole of water enmeshed in the SAP, one cycle of condensation and evaporation creates two phases, a DME rich vapor carrying water away and a water rich phase with some DME contained within the SAP. At 19 mole% water saturation in DME, 3.5 gs of water (0.19 mol) should be extracted in the original 46 gs of condensed DME(Chapoy et al., 2011; Tallon and Fenton, 2010;) resulting in an extraction energy of 5.45 kJ/g based on solubility. Other losses are incurred as some DME is lost dissolved in water and would require recharging. (*Fundamental Aspects of Dimethyl Ether - Energy.gov, 1998*).

Focusing on the similar system developed by Kanda, they used ~87 L of DME to extract 3 kg of water embedded in several functional diapers. The energy cost expended for Kanda et al. for this large volume of DME is ~35 MJ, and extracting 3 kg of water, puts the energy cost at ~10.5 kJ/g (Kanda et al., 2010), about twice what we found in our system.

Further recharging of the system with N_2 condenses the residual fraction of DME in the extraction vessel, and the second cycle, only collects another 6–12% of the original water mass. Subsequent chargings increase the amount extracted but without an increase in overall efficiency.

In terms of mechanism, perhaps more water is extractable if the soft, swollen gels are also compressed. It is encouraging to see related data from Melandres et al. who showed a an 80% drop in pressure driven reduced water absorption in saline filled SAPs relative to free swelling, even at very low pressures (0.005 MPa) (Melandres et al., 2019).

Compression is probably more efficient than the hydrostatic loading here. There are other mass balance losses of DME into the water, which would need accounting in a completely recharging and recyclable model for dehydration, but based on energy expended, results are not encouraging for this to be an energetically favorable process.

4. Conclusion

A condensed DME-based extraction system leveraged hydrostatic pressure (0.1–0.6 MPa) to extract water and its dissolved salts from saturated superabsorbent acrylic acid-based polymers (SAPs). Close to 90% extraction rates with a deionized water-based extraction experiment and ~ 80% extraction with an artificial urine are achieved with a single 30 g injection of DME. Water is extracted more, both by mass and a percentage basis than artificial urine because there is a much higher volume of deionized water swollen in the SAP. Conductivity measurements show that salt in the artificial urine is also extractable. The system operates under ambient temperatures although there are energy losses to the surrounding environment by thermal radiation from the hardware. The DME-dehydrated superabsorbent polymers can undergo re-swelling. After being dried, the regenerated SAP may be re-deployable for some other form of desiccant usage. While drying is evident, it is not energetically favorable relative to simply evaporating the water out.

Declaration of Competing Interest

None.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.resconrec.2021.105641.

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