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Properties of Sugar, Polyol, and Polysaccharide Water−Ethanol Solutions

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The solubility and viscosity of sugars (glucose, lactose, leucrose, maltose, raffinose, sucrose, and trehalose), polyols (maltitol, mannitol, sorbitol, and xylitol), and polysaccharides (β-cyclodextrin, dextrans, and inulin) in water and water−ethanol mixtures was investigated at 310 K. The increase in ethanol fraction caused a decrease in solubility in all cases. The viscosity of a 10 % (w/w) solution of any of those sugars was (1 to 1.25) mPa s except dextran solutions, which reached 5 mPa s. The viscosity of saturated solutions varied strongly from one compound to another even though the solubilities were similar. The metastable zone widths of sucrose, maltose, and lactose precipitated with ethanol were significantly larger than the one measured for mannitol.

Introduction

Data on the solubility and viscosity of sucrose and lactose in aqueous solution are widely available in the literature.[1−5] However, little information is available for many other sugars, polyols, and polysaccharides.[6−9] Even though the effect of ethanol on a few of these solutions has been investigated,[10−20] the information available is fragmented and limited to the most common sugars. Still, some work on the prediction of the solubility of sugars in water−ethanol mixtures was done using the basic and modified UNIFAC[21−24] and UNIQUAC models,[10,13,25] or the Redlich−Kister expansion model,[20] but only for some common small sugars: sucrose, glucose, and lactose.

The addition of an antisolvent, such as ethanol, to an aqueous solution containing a solute poorly soluble in water would result in the precipitation of the solute by a process called antisolvent precipitation, dilution, salting-out, or drowning-out crystallization. Antisolvent precipitation investigation has been mainly devoted to kinetic studies under batch or continuous operations[26−29] or to the experimental and theoretical study of the influence of the process conditions on the particle size and shape.[29−36]

Also, other data on crystallization-related parameters are scarce. Compared with other crystallization processes, limited information is available on the metastable zone width caused by the addition of antisolvent[34,37,38] during antisolvent precipitation.

These physicochemical data are of interest in the fields of pharmaceutical formulation and food, where various sugars, polyols, and polysaccharides are commonly used. The properties measured in this study might then be required for the design of new processes. In this work, solubility, viscosity, and supersaturation measurements were conducted on various sugar, polyol, and polysaccharide solutions prepared with water−ethanol mixtures at 310 K.

Materials and Methods

Materials. D-Saccharose (crystalline, Riedel-de Haen), lactose anhydrous (crystalline, Fluka), and D-mannitol (crystalline, Fluka) of European Pharmacopoeia grade, dextrose anhydrous (crystalline, glucose, Sigma) of USP grade, D-trehalose dihydrate (crystalline, Sigma), D-leucrose (+98 %) (crystalline, Fluka), D-(+)-maltose monohydrate (crystalline, Riedel-de Haen), D-(+)-raffinose pentahydrate (+99 %) (crystalline, Fluka), maltitol (+98 %) (crystalline, Sigma), D-sorbitol (+98 %) (crystalline, Aldrich), xylitol (+99 %) (crystalline, Sigma), (2-hydroxypropyl)-β-cyclodextrin ($M_r \sim 1380$) (amorphous, Fluka), and dextran from *Leuconostoc* spp. ($M_r \sim 6000$) (amorphous, Fluka) and from *Leuconostoc mesenteroides*, strain B512 ($M_r \sim 68800$, clinical grade) (amorphous, Sigma) were used as received. Short-chain inulin (90 % to 95 %, 8 to 12 monomers, amorphous, Frutafit IQ) was supplied by Sensus, Rosendaal, The Netherlands. Technical grade ethanol (100 %) was purchased from Chemproha.

Solubility Measurement. The solubility of the solute was simply measured by gravimetry using a balance (AE 100 Mettler Toledo, ±0.0001 g). To prepare saturated solutions, the mixtures containing crystals were left for over 1 week in a thermostated shaking bath at 310 K. The Erlenmeyers used for the dissolution were sealed with Parafilm to avoid any changes in the solvent composition during the dissolution process. Viscous solutions (e.g., sucrose, cyclodextrin, etc.) were especially monitored by visual inspection of the concentration gradient to ensure that appropriate mixing was available to achieve saturation conditions throughout the solution. The mixtures were rapidly filtered through a fiberglass prefilter (glass fiber prefilters AP20, Millipore), and the known weights of solution were left to evaporate, first at ambient conditions and then in an oven heated at 60 °C until the measured mass was stable. The solubility in pure water and in 50 % ethanol was measured for all of the studied compounds, and the solubility in 95 % and 100 % ethanol was measured for most compounds. All solutions were prepared at least in triplicates. The standard deviation on the measurements was at most 0.02 $g \cdot g^{-1}$ solution.

Viscosity Measurement. The viscosity of the 10 % (w/w) solutions and saturated solutions prepared for the solubility measurements was measured. A Haake VT 550 viscometer (Karlsruhe, Germany) thermostated at 310 K and equipped with a cylinder geometry (NV, inner rotor cylinder, outer cup cylinder, 0.35 mm gap, 60 mm high) was used. The standard deviation of the viscosity measurements was 0.2 mPa s.

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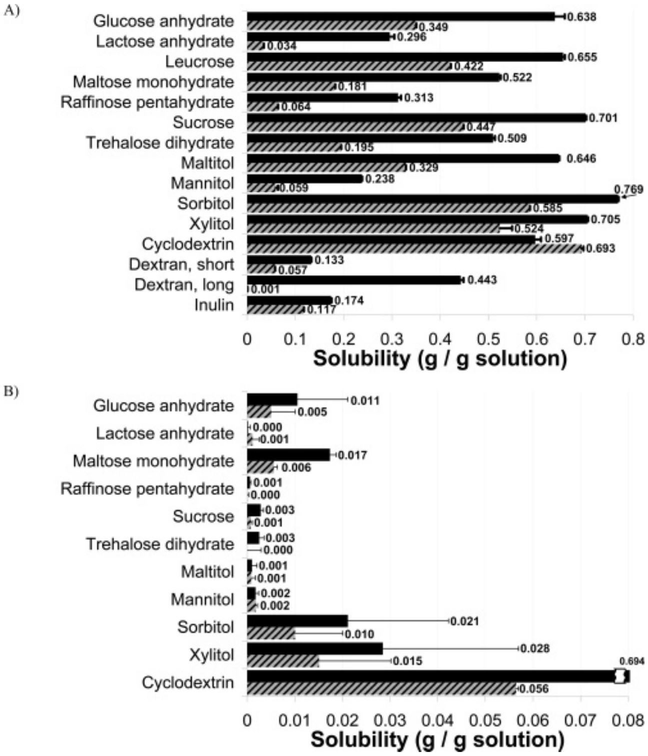

Figure 1. Solubility of sugars, polyols, and polysaccharides in (A) pure water (black bars) and in 50 % ethanol (hatched bars) and in (B) 95 % ethanol (black bars) and in 100 % ethanol (hatched bars) at 310 K. The standard deviation is illustrated with error bars.

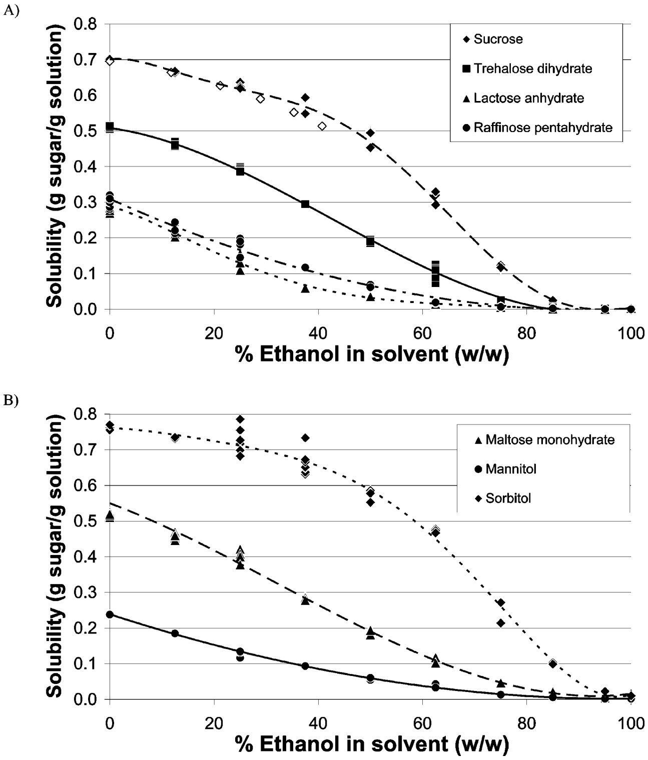

Figure 2. Solubility of sugars and polyols at 310 K in various water−ethanol mixtures: (A) sucrose (open diamonds correspond to interpolated literature data from ref 39), trehalose dihydrate, lactose anhydrate, and raffinose pentahydrate; (B) maltose monohydrate, mannitol, and sorbitol.

Maximum Supersaturation. Saturated solutions were prepared using a shaking bath set at 310 K and were allowed at least 1 week for dissolution (lactose had only 24 h). The solutions were filtered before use to remove undissolved crystals. About 400 mL of saturated solution were poured into the 1.5 L jacketed glass precipitation vessel. Stirring was performed by a two-blade impeller mounted above the vessel. The temperature was controlled by a heating bath. Ethanol was continuously added with a pump, and the amount of ethanol added was calculated with the difference in ethanol weight. During the addition of ethanol, solution samples were collected every minute and immediately analyzed offline by absorbance [350 nm, Perkin-Elmer Lambda 2 UV−vis spectrophotometer (Überlingen, Germany)].The concentration of the solutions was measured as described previously, and the density of the solution was estimated by weighing the volume of solution used in each experiment. The induction time to precipitation was identified from graphics of the absorbance as a function of the time, confirmed with the visual observation of the solutions.

Results and Discussion

Solubility. The solubility of all studied sugars, polyols, and polysaccharides in aqueous solution and in 50 % (w/w) ethanol at 310 K is depicted in Figure 1A, and the solubility of most of these compounds in 95 % and 100 % (w/w) ethanol at 310 K is also depicted in Figure 1B. The solubility was always higher in pure water than in ethanol mixtures, with the exception of cyclodextrin (Figure 1). The difference in solubility was large, from about (0.133 to 0.769) $g \cdot g^{-1}$ of solution in water, and extreme in 50 % ethanol (from (0.001 to 0.693) $g \cdot g^{-1}$ of solution) and in 95 % ethanol (from (0.001 to 0.694) $g \cdot g^{-1}$ of solution). The solubility was generally extremely low in 95 % or 100 % ethanol (Figure 1B) with the exception of cyclodextrin, which has comparable solubilities in 50 % and 95 % ethanol (∼0.69 $g \cdot g^{-1}$ of solution) and is relatively soluble in 100 % ethanol.

The measured values for sucrose solubilities are in accordance, within 1 % (w/w), with those available in the literature for at high water content and within 5 % (w/w) when the ethanol fraction in the solvent is increased (Figure 2).[39] The standard deviation on the measurements was at most 0.02 $g \cdot g^{-1}$ of solution (Figure 1; Table 1). The relative error in the concentration of glucose, sorbitol, and xylitol solutions prepared with 95 % and 100 % ethanol could reach 100 %. The very sharp transition between an undersaturated solution and a very thick slurry that could not be rapidly filtered hindered the measurement of more accurate values.

The solubilities of some of those sugars and polyols in more water−ethanol mixtures at 310 K were also determined (Figure 2; Table 1). Three families of curves are observed. (A) For the compounds with the lowest solubility in water [(0.25 to 0.3) $g \cdot g^{-1}$ of solution]—lactose anhydrate, raffinose pentahydrate, and mannitol—the solubility rapidly decreased as soon as ethanol was present in the solution. (B) The solubility of the moderately water soluble compounds maltose monohydrate and trehalose dihydrate (∼0.5 $g \cdot g^{-1}$ of solution in water) also decreased with the addition of ethanol, but the decrease was relatively much smaller. In fact, the solubility data could be best fitted with a quadratic equation, with an inflection point around 60 % water in solvent (w/w). (C) Highly soluble sucrose and sorbitol, with aqueous solubilities of (0.70 and 0.75) $g \cdot g^{-1}$ of solution, respectively, showed similar behaviors to what was observed with (B) curves, but the slow decrease in solubility was further extended to higher ethanol fractions. For these compounds, the inflection point was around 35 % water in solvent (w/w). On the basis of the shape of those curves, the antisolvent precipita-

Table 1. Solubility of Sugars and Polyols at 310 K in Various Water−Ethanol Mixtures (Grams per Gram of Solution)

% water (w/w)	% ethanol (w/w)	lactose anhydrate	maltose monohydrate	raffinose pentahydrate	sucrose	trehalose dihydrate	mannitol	sorbitol
100	0	0.29 ± 0.02	0.519 ± 0.005	0.30 ± 0.01	0.701 ± 0.002	0.508 ± 0.003	0.238 ± 0.001	0.762 ± 0.006
87.5	12.5	0.212 ± 0.007	0.46 ± 0.01	0.24 ± 0.01	0.663 ± 0.003	0.461 ± 0.004	0.185 ± 0.001	0.734 ± 0.003
75	25	0.13 ± 0.02	0.39 ± 0.02	0.18 ± 0.01	0.624 ± 0.006	0.394 ± 0.006	0.132 ± 0.006	0.72 ± 0.03
62.5	37.5	0.064 ± 0.002	0.284 ± 0.004	0.1169 ± 0.0009	0.56 ± 0.02	0.295 ± 0.001	0.093 ± 0.001	0.66 ± 0.03
50	50	0.035 ± 0.001	0.186 ± 0.007	0.063 ± 0.003	0.46 ± 0.02	0.190 ± 0.004	0.060 ± 0.002	0.57 ± 0.02
37.5	62.5	0.016 ± 0.001	0.107 ± 0.009	0.018 ± 0.001	0.31 ± 0.02	0.11 ± 0.02	0.034 ± 0.003	0.472 ± 0.004
25	75	0.0072 ± 0.0005	0.0470 ± 0.0006	0.007 ± 0.001	0.119 ± 0.004	0.024 ± 0.001	0.0135 ± 0.0001	0.25 ± 0.03
15	85	0.0021 ± 0.0007	0.024 ± 0.002	0.0027 ± 0.0004	0.0253 ± 0.0004	0.006 ± 0.001	0.006 ± 0.001	0.101 ± 0.002
5	95	0.001 ± 0.001	0.006 ± 0.001	0.0008 ± 0.0006	0.0007 ± 0.0002	0.003 ± 0.001	0.002 ± 0.001	0.021 ± 0.001
0	100	0.0002 ± 0.0005	0.0174 ± 0.0008	0.0000 ± 0.0003	0.003 ± 0.001	0.0010 ± 0.0004	0.0018 ± 0.0004	0.0100 ± 0.0002

tion of (A) compounds is expected to be the easiest because of the sharper decrease in solubility, followed by (B) and finally (C) compounds.

Maximum Supersaturation. Sugar solutions can be highly supersaturated by cooling. Here, the maximally obtainable supersaturation caused by the addition of ethanol as antisolvent was studied with sucrose, lactose, maltose, and mannitol The ethanol was added at a constant flow rate. Saturation was achieved after (10 to 50) min depending on the composition of the initial solution (i.e., sugar species and ethanol fraction). In the case of the sugars, the width of the metastable zone is larger than the one observed with the polyol mannitol (Figure 3). The supersaturation curves drawn for the sugars all had a characteristic shape; the solubility remained relatively high until a certain ethanol concentration was reached, and then the solubility dropped suddenly. This was particularly apparent with the sugars with the higher solubilities: the solubilities of sucrose and maltose dropped at 75 % ethanol, whereas the solubility of lactose steeply decreased from 50 % ethanol.

Because of the respective shapes of their solubility curves, the widths of the metastable zone of maltose and lactose are wider than the one of sucrose. This should translate into a faster precipitation once the corresponding supersaturation conditions are reached, giving rise to the production of an amorphous product. In a similar fashion, the width of the metastable zone measured for mannitol is very narrow. Therefore, crystallization is expected to occur soon after the ethanol concentration in solution begins to increase.

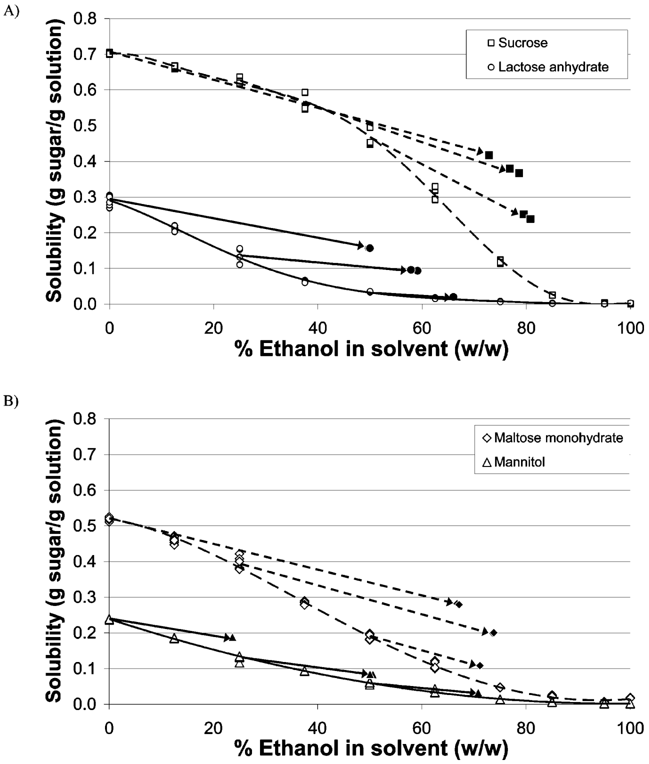

Figure 3. Metastable zone widths, solubility, and supersaturation of (A) sucrose, and lactose and (B) maltose and mannitol at 310 K caused by the constant addition of ethanol at 10 mL·min^{-1}. Filled markers correspond to the solubility and open markers to the supersaturated conditions.

Table 2. Viscosity of Aqueous 10 % Sugar, Polyol, and Polysaccharide Solutions at 310 K

solution 10 %	viscosity (mPa s)	solution 10 %	viscosity (mPa s)
glucose	1.00	mannitol	1.03
lactose	1.16	sorbitol	1.02
leucrose	1.05	xylitol	1.03
maltose	1.09	cyclodextrin	1.09
raffinose	1.22	dextran, short	1.62
sucrose	1.06	dextran, long	5.03
trehalose	1.10	inulin	1.12
maltitol	1.08		

Viscosity. The viscosity of aqueous 10 % sugar, polyol, and polysaccharide solutions (Table 2) and some saturated water−ethanol sugar and polyol solutions (Figure 4) was investigated. When the viscosities of 10 % aqueous solutions are compared, a significant difference is observed only with the dextrans.

As the evaporation of the solvent itself is hindered by the high viscosity of a solution, the viscosity of saturated solutions was investigated (Figure 4; Table 3).

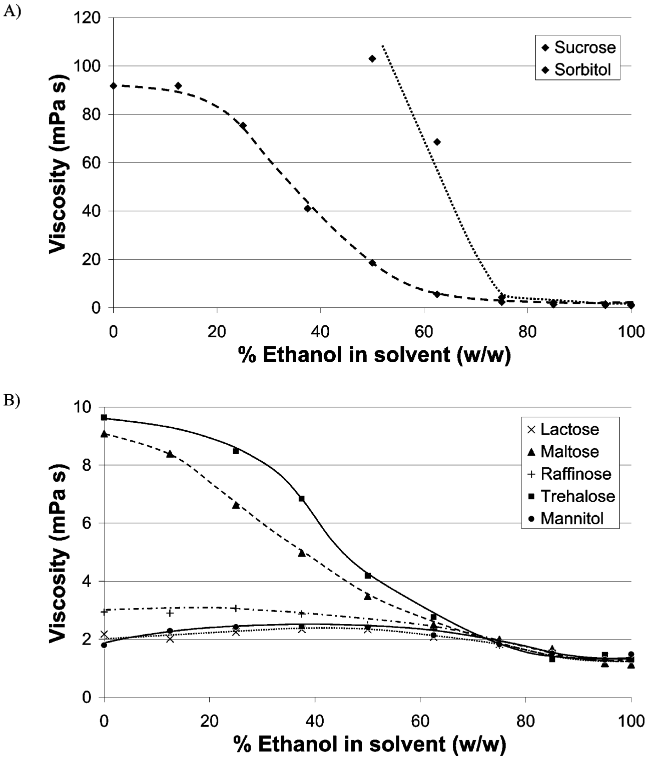

Figure 4. Viscosity of saturated sugar solutions at 310 K in various water−ethanol mixtures.

Table 3. Viscosity of Saturated Sugar Solutions at 310 K in Various Water−Ethanol Mixtures

		viscosity/mPa s						
% water (w/w)	% ethanol (w/w)	lactose	maltose	raffinose	sucrose	trehalose	mannitol	sorbitol
100	0	2.17	9.08	2.93	91.74	9.64	1.79	n/a
87.5	12.5	2.01	8.39	2.90	91.79	n/a	2.28	n/a
75	25	2.25	6.62	3.06	75.32	8.48	2.42	n/a
62.5	37.5	2.34	4.97	2.87	41.03	6.84	2.44	n/a
50	50	2.33	3.48	2.49	18.56	4.18	2.41	103.01
37.5	62.5	2.06	2.50	2.33	5.60	2.76	2.14	68.54
25	75	1.83	1.99	1.79	2.32	1.86	1.82	4.27
15	85	1.50	1.67	1.54	1.37	1.30	1.51	2.13
5	95	1.39	1.17	1.30	1.12	1.46	1.29	1.52
0	100	n/a	1.12	1.23	0.89	1.30	1.48	1.27

The viscosity of the sorbitol and sucrose solutions (Figure 4A) is much higher than the viscosity of the other sugars and polyols studied (Figure 4B). Furthermore, the viscosity of sorbitol increases much more rapidly than that of sucrose, even though their solubilities are similarly affected by ethanol above a content of 25 % in solvent. The viscosities of maltose and trehalose increase in a similar way, and that increase is a mimic of the solubility. However, for almost highly similar solubilities, the viscosity of trehalose is slightly higher. The viscosities of lactose, mannitol, and raffinose are quite low [(1.1 to 3.0) mPa s]. Lactose and mannitol are very similar, and both show a slight trend of maximum viscosity around 60 % water in solvent, even though this corresponds to less than 50 % of the maximum solubility of these sugars. This trend was less significant with raffinose.

In aqueous solution, the viscosity of sugar solutions is known to strongly increase with the concentration. The increase depends on the molecular weight of the carbohydrate, the structure of the molecule, and the orientation of the OH groups as they have a positive interaction with water.[40] In water−ethanol saturated solutions, a similar trend was observed, except for the sugars and polyols with low solubilities. Polyol solutions are generally less viscous than sucrose solutions at the same concentration[41] as we measured at 10 % concentration (Table 2). However, the viscosity of a polyol such as sorbitol can be extremely high when the solution is saturated (Figure 4).

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