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Viscosities and Densities of Concentrated Aqueous NaOH/NaAl(OH)$_4$ Mixtures at 25 °C

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The dynamic viscosities (η) and densities (ρ) of aqueous NaOH/NaAl(OH)$_4$ mixtures have been determined for solutions at constant ionic strength and total concentrations of $[\mathrm{NaOH}]_T \leq 13.6$ mol·kg^{-1} and $[\mathrm{Al(III)}]_T/[\mathrm{Na}^+]_T \leq 0.6$ at 25 °C using a Ubbelohde viscometer and a vibrating tube densimeter, respectively. The viscosities are precise to ±0.1% and the densities to $\pm 5 \times 10^{-6}$ g·cm^{-3}. The densities of the mixtures at constant ionic strength vary linearly with substitution of OH$^-$ by Al(OH)$_4$$^-$, consistent with Young's rule. This enables calculation of the density of the hypothetical aqueous solution of pure "NaAl(OH)$_4$" at $[\mathrm{Al(III)}]_T \leq 13$ mol·kg^{-1}. Viscosities of these mixtures, as with other electrolytes, are overestimated by Young's rule. Viscosities of the hypothetical pure "NaAl(OH)$_4$" solutions (≤6 mol·kg^{-1}) were accordingly determined by nonlinear extrapolation. The η values so obtained are unusually high but show a dependence on $[\mathrm{NaAl(OH)_4}]_T$ similar to that of common electrolyte solutions. An empirical correlation for the viscosities of NaOH/NaAl(OH)$_4$ mixtures is also given.

Introduction

During the Bayer process for the recovery of gibbsite, Al(OH)$_3$(s), from bauxitic ores, hot (≤250 °C), concentrated solutions containing NaOH ($[\mathrm{Na}^+]_T \leq 6$ mol·L^{-1}, M) and NaAl(OH)$_4$ ($[\mathrm{Al(III)}]_T \leq 4$ M) are utilized. Accurate values for the densities and viscosities of such solutions (known industrially as Bayer liquors) are needed over a wide range of concentrations and temperatures for optimization of plant design.

Density data for such solutions are abundant in the open literature[1−10] but have been determined largely in an industrial context and are thus often not of sufficient quality for the present purposes. Moreover, these densities were measured mainly at superambient temperatures (≥50 °C), and reliable data for synthetic Bayer liquors (SBLs) at the standard temperature (25 °C) are relatively scarce. Of the available data, the most accurate are probably those of Hovey et al.[7] and Sanjuan and Milchard.[8] However, these authors covered only very limited concentration ranges ($[\mathrm{Na}^+]_T \leq 2.31$ and 1.00 mol·kg^{-1} and $[\mathrm{Al(III)}]_T \leq 1.17$ and 0.06 mol·kg^{-1}, respectively) which are well below the industrially relevant region.

Viscosities of Bayer liquors, both synthetic and "real" (industrial), are much less well characterized.[9−11] Many of the data are of dubious quality, and only one paper presents (a limited number of) measurements at 25 °C.[11]

As part of a wider investigation of the physicochemical properties of SBLs and related solutions,[12] a systematic investigation of the densities and viscosities of NaOH/NaAl(OH)$_4$ mixtures has been undertaken over the industrially important concentration range of $1 \leq [\mathrm{NaOH}]/\mathrm{mol\cdot kg^{-1}} \leq 13.6$ and $0 \leq [\mathrm{Al(III)}]_T/[\mathrm{Na}^+]_T \leq 0.6$. The data have been used to construct empirical mixing rules to correlate the physicochemical properties of the mixtures in terms of those of the pure binary (i.e., one solute and one solvent) electrolyte solutions. In particular, the application of Young's rule has been investigated in terms of both molarity (M) and molality (mol·kg^{-1}) concentration units for densities and in molalities (only) for viscosities.

Experimental Section

Solution Preparation. Solution series, consisting of individual samples of 100 mL of total volume, were prepared by mixing appropriate amounts of the following components: (a) concentrated (~50 mass %, 20 M) NaOH (Ajax, >99.0% grade) solutions of accurately known concentration, density[12] and with a carbonate concentration < 0.1% of the total alkalinity;[13] (b) concentrated alkaline aluminate solutions of known density[12] and concentrations of aluminum (~5 M) and NaOH (~8 M), prepared from high-purity Al-wire (99.9%, Ajax) as described elsewhere;[14] and (c) high-purity water (Millipore MilliQ system) boiled and purged with nitrogen to remove traces of CO$_2$.

Solution series of constant ionic strengths, both in terms of molarity and of molality, were investigated. For the isomolality series, calculated amounts of the three components were weighed accurately into dry Pyrex screw-top bottles. For the isomolarity series, stock solutions were dispensed gravimetrically into A-grade 100 mL volumetric flasks and were brought to the mark with high-purity water. Accurate concentrations were calculated using buoyancy corrections. The overall uncertainty of the concentrations is expected to be ≤0.1% for the isomolality and ≤0.2% for the isomolarity series. The solutions were filtered through a supported membrane filter (Versapor 450, pore size 0.45 μm) immediately after preparation, and their densities and viscosities were determined within 8 h. Although some of the solutions were supersaturated with respect to Al(OH)$_3$(s), no precipitation was observed during this period.

Density Measurements. Densities were measured using Anton Paar DMA O2D or DMA 602 vibrating tube densimeters. The experimental protocol was identical to that

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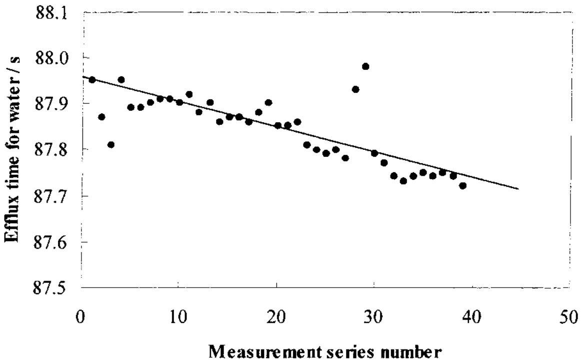

Figure 1. Long term (~6 months) effect of concentrated alkaline solutions on the efflux time of water at 25 °C in an Ubbelohde glass capillary viscometer.

used previously.[12] The densimeter tube was thermostated to (25.00 ± 0.01) °C with a Julabo F33 thermostat. The temperature of the bath was checked regularly using a quartz crystal thermometer (Hewlett-Packard, Model HP 2804A) that had been calibrated against the triple point of water with a probable accuracy of ±0.02 °C.

Viscosity Measurements. Ubbelohde-type suspended-level glass capillary viscometers (Schott-Geräte, type 53010 with capillary number I and a capacity of 15−20 mL) were used for determining solution viscosities as described previously.[12] The temperature of the water bath was maintained at (25.00 ± 0.005) °C using the room environment (20 ± 2) °C as the heat sink. The temperature in the immediate vicinity of the viscometer bulb in the bath was continuously monitored with a quartz crystal thermometer.

The dynamic viscosity of the solutions was determined from eq 1

$$\eta = \eta_0 \frac{t - \Theta}{t_0 - \Theta_0} \frac{\rho}{\rho_0} \tag{1}$$

where η is the dynamic viscosity, ρ is the density, t is the flow time, Θ is the flow-time-dependent empirical correction factor (the so-called Hagenbach correction), and the subscript 0 denotes a reference liquid (water). The values of Θ were those provided by the viscometer manufacturer (*Operating Instructions Ubbelohde Viscometers*, EDV 27131, Ver. 930609M, Schott Gerate GbmH).

High-purity water (for which $\eta_0 = 0.8904$ mPa·s and $\rho_0 = 0.997\ 043$ g·cm^{-3}), degassed and filtered (0.45 μm), was used to calibrate the viscometer. From several hundred independent runs, the efflux times for water were initially found to be (87.95 ± 0.02) s, but this value gradually dropped to about (87.75 ± 0.02) s over a period of ~6 months (Figure 1). This variation is consistent with a very slow dissolution of the glass in the caustic solutions causing a small but noticeable increase in the capillary bore. This dissolution was sufficiently slow to have no practical effect on the measured viscosities. The efflux times for the sample solutions varied between 100 and 600 s and were determined to the nearest 0.01 s with an automatic Schott-Geräte AVS 310 measuring unit and a Schott-Geräte AVS/S measuring stand.[12] To enhance accuracy, t_0 was determined daily using three independent viscometer charges (each yielding six readings) before and after the test solution measurements. The efflux times of the test solutions were also measured from triplicate charges, each of six readings. Measurements were accepted when the results were reproducible to ±0.02 s (calibrations) and ±0.05 s (samples). This level of reproducibility indicated no significant variations in the solution composition (e.g., from absorption of atmospheric CO_2 or moisture).

Table 1. Densities (ρ) and Dynamic Viscosities (η) of Aqueous $NaOH/NaAl(OH)_4$ Mixtures at Constant Molality (mol·kg^{-1}) at 25 °C

$[NaOH]_T$	$[Al(III)]_T$	C^a	A/C^b	ρ	η
mol·kg^{-1}	mol·kg^{-1}	g·L^{-1}		g·cm^{-3}	mPa·s
		$I = 1$ mol·kg^{-1}			
0.9993	0	52.91	0	1.038 999	1.0941
1.0027	0.1005	52.84	0.096	1.042 166	1.1157
0.9975	0.2014	52.33	0.194	1.044 957	1.1362
1.0023	0.3006	52.35	0.289	1.048 250	1.1607
0.9981	0.4069	51.87	0.392	1.050 820	1.1801
0.9994	0.5003	51.74	0.482	1.054 085	1.2070
1.0027	0.6002	51.67	0.576	1.056 912	1.2300
0	1.00^c	0	∞	1.067 92	1.336
		$I = 2$ mol·kg^{-1}			
2.0054	0	106.04	0	1.077 799	1.3636
2.0039	0.2004	104.99	0.096	1.083 354	1.4164
1.9980	0.3991	103.81	0.192	1.089 253	1.4674
2.0012	0.5998	102.97	0.288	1.094 098	1.5292
2.0009	0.7995	102.04	0.384	1.099 340	1.5870
1.9999^d	0.7994	102.00	0.385	1.099 420	1.5898
2.0007	1.0000	101.14	0.481	1.104 697	1.6540
1.9984	1.1994	100.15	0.577	1.109 731	1.7177
0	2.00^c	0	∞	1.128 56	1.998
		$I = 3$ mol·kg^{-1}			
2.9990	0	158.03	0	1.113 597	1.7236
3.0023	0.2995	156.02	0.096	1.121 277	1.8185
3.0049	0.5996	154.07	0.192	1.129 022	1.9271
3.0014	0.8988	151.85	0.288	1.136 204	2.0373
3.0018	1.1997	149.89	0.384	1.143 549	2.1600
2.9954	1.4971	147.66	0.481	1.150 312	2.2835
2.9992	1.7997	145.93	0.577	1.157 210	2.4231
0	3.00^c	0	∞	1.181 50	3.122
		$I = 4$ mol·kg^{-1}			
4.0024	0	209.72	0	1.147 027	2.1816
4.0122	0.3992	206.68	0.096	1.158 304	2.3828
4.0040	0.7992	202.36	0.192	1.165 850	2.5351
4.0033^d	0.7995	202.36	0.192	1.166 056	2.5359
4.0084	1.1993	199.18	0.288	1.175 726	2.7498
4.0016	1.5997	195.28	0.385	1.183 177	2.9361
4.0016	1.9992	192.04	0.481	1.191 772	3.1689
4.0004	2.3992	188.78	0.577	1.199 607	3.4092
0	4.00^c	0	∞	1.228 12	4.734
		$I = 5$ mol·kg^{-1}			
5.0125	0	261.25	0	1.180 655	2.8348
5.0150	0.5017	255.54	0.096	1.192 030	3.1164
5.0065	0.9992	249.39	0.192	1.201 462	3.3815
5.0054	1.4993	244.18	0.288	1.212 515	3.7321
5.0043	1.9989	238.71	0.384	1.220 654	4.0347
4.9999	2.4993	233.94	0.481	1.231 606	4.4765
4.9996	2.9993	228.80	0.577	1.238 269	4.7954
0	5.00^c	0	∞	1.269 47	6.601
		$I = 6$ mol·kg^{-1}			
6.0089	0	311.14	0	1.211 898	3.6862
6.0153	0.5993	302.68	0.096	1.222 334	4.0410
6.0068	1.1996	294.37	0.192	1.233 457	4.4741
6.0030	1.7989	286.95	0.288	1.245 777	5.0217
6.0022	2.3990	280.04	0.384	1.256 508	5.5967
6.0024^d	2.3992	280.05	0.385	1.256 542	5.5942
6.0025	3.0000	273.39	0.481	1.266 915	6.2411
6.0007^d	2.9994	273.31	0.481	1.266 831	6.2261
0	6.00^c	0	∞	1.306 35	10.43

[a] Concentration of Na^+ expressed in terms of grams of Na_2CO_3 per liter of solution. [b] Mass ratio of total Al(III) (expressed as grams of Al_2O_3) to total Na^+ (expressed as grams of Na_2CO_3). [c] Hypothetical values obtained by extrapolating to 100% substitution of OH^- with $Al(OH)_4^-$. [d] Independent duplication.

Results and Discussion

The observed densities and dynamic viscosities of aqueous $NaOH/NaAl(OH)_4$ mixtures at 25 °C are presented in

Table 2. Densities, ρ, of Aqueous NaOH/NaAl(OH)$_4$ Mixtures at Constant Molarity (M) at 25 °C

$[NaOH]_T$/M	$[Al(III)]_T$/M	C^a/g·L^{-1}	A/C^b	ρ/g·cm^{-3}
		$I = 1$ M		
1.021	0	54.11	0	1.039 686
1.003	0.200	53.13	0.192	1.045 158
0.999	0.400	52.96	0.385	1.051 780
		$I = 2$ M		
1.999	0	105.93	0	1.077 364
2.000	0.399	105.99	0.192	1.090 050
1.999	0.600	105.93	0.289	1.096 395
1.999	0.799	105.94	0.384	1.102 639
1.996	0.999	105.79	0.481	1.108 861
		$I = 3$ M		
3.022	0	160.13	0	1.114 558
3.008	0.299	159.39	0.096	1.122 875
2.983	0.600	158.10	0.193	1.131 578
2.996	0.899	158.75	0.289	1.141 557
3.104	1.199	164.51	0.372	1.154 387
2.989	1.498	158.42	0.482	1.159 745
		$I = 4$ M		
3.997	0	211.80	0	1.147 476
4.005	0.400	212.26	0.096	1.159 923
4.028	0.799	213.47	0.191	1.173 281
3.996	1.199	211.77	0.289	1.184 087
3.997	1.599	211.81	0.385	1.196 815
3.996	1.997	211.76	0.481	1.208 913
		$I = 6$ M		
5.991	0	317.49	0	1.210 702
6.016	0.599	318.82	0.096	1.229 850
6.015	1.200	318.78	0.192	1.247 757
6.002	1.798	318.06	0.288	1.265 374
5.987	2.397	317.28	0.385	1.283 070
5.991	2.997	317.48	0.481	1.302 077
		$I = 8$ M		
8.024	0	425.23	0	1.269 834
8.015	1.000	424.76	0.120	1.299 683
8.004	1.998	424.19	0.240	1.329 606
7.991	2.997	423.49	0.361	1.358 642
7.991	3.996	423.48	0.481	1.388 467
		$I = 10$ M		
10.037	0	531.91	0	1.323 813
10.264	1.00	543.92	0.094	1.359 322
9.988	1.998	529.30	0.192	1.381 355
9.991	2.997	529.49	0.289	1.411 125
10.014	4.004	530.68	0.385	1.441 134

[a] Concentration of Na$^+$ expressed in terms of grams of Na$_2$CO$_3$ per liter of solution. [b] Mass ratio of total Al(III) (expressed as grams of Al$_2$O$_3$) to total Na$^+$ (expressed as grams of Na$_2$CO$_3$).

Tables 1 and 2 and Figures 2−4. Concentrations are expressed both in terms of molality and molarity. For convenience, the data have also been converted into two industrial units: C (concentration of NaOH expressed as grams of Na$_2$CO$_3$/liter of solution) and the alumina-to-caustic ratio, A/C (the mass ratio between the mass of aluminum expressed as grams of Al$_2$O$_3$/liter and the mass of NaOH expressed as grams of Na$_2$CO$_3$/liter). Replicate measurements on independently prepared solutions (Table 1) were found to agree to ±50 ppm for densities and ±0.2% for viscosities. The present data are also in excellent agreement with those obtained previously by us for NaOH solutions under identical conditions.[12]

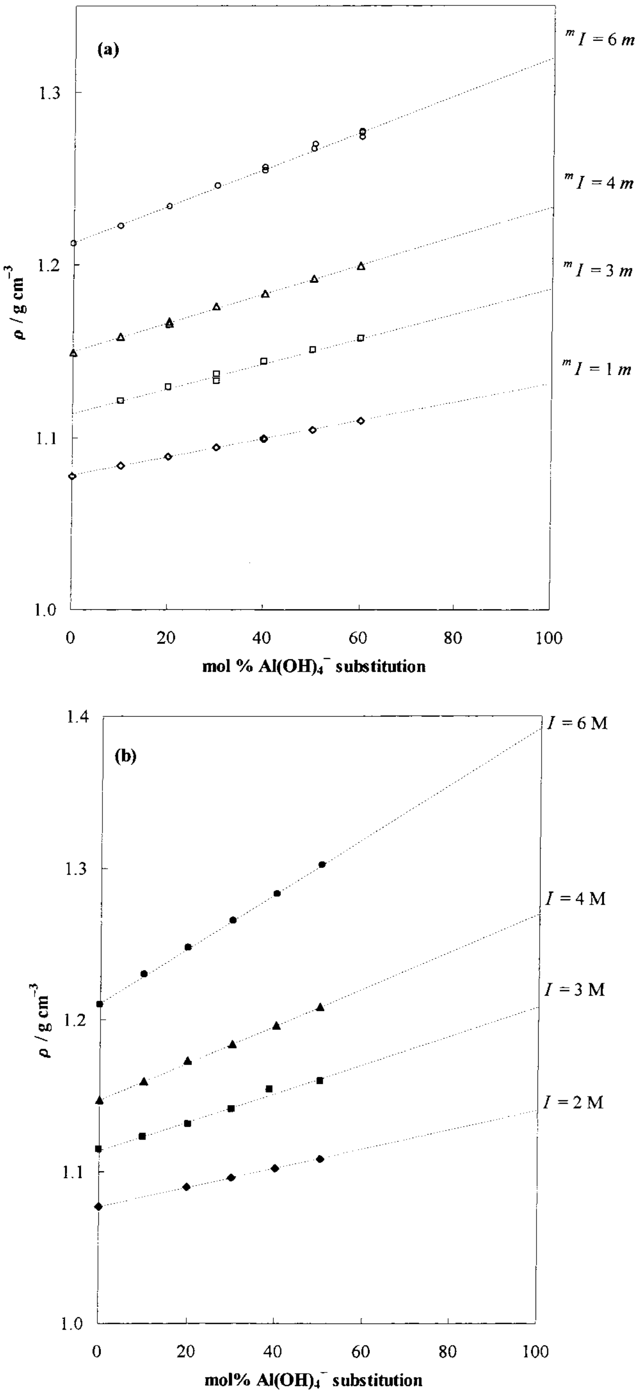

Figure 2. Densities (ρ) of (a) isomolal and (b) isomolar series of NaOH/NaAl(OH)$_4$ mixtures at various ionic strengths.

Densities. Assuming that NaOH/NaAl(OH)$_4$ mixtures consist only of Na$^+$, OH$^-$, Al(OH)$_4^-$, and water,[15] then they can be regarded as mixtures of NaOH(aq) and the hypothetical pure salt solution, "NaAl(OH)$_4$(aq)". The densities of such ternary (i.e., two electrolytes and one solvent) mixtures can be expressed in terms of the densities of the respective binary solutions. Young's rule[16] expresses the density of a ternary solution as the ionic strength-weighted sum of the relevant binary solution densities. Thus, Young's rule for the density of an aqueous NaOH/NaAl(OH)$_4$ mixture, $\rho_T(I_T)$, can be written as

$$\rho_T(I_T) = \frac{I_{NaOH}}{I_T}\rho_{NaOH}(I_T) + \frac{I_{NaAl(OH)_4}}{I_T}\rho_{NaAl(OH)_4}(I_T) \quad (2)$$

where I_T is the total (stoichiometric) ionic strength of the mixture in molarity units, I_{NaOH} and $I_{NaAl(OH)_4}$ are the ionic strength (or, for 1:1 electrolytes, concentration) contributions of NaOH and "NaAl(OH)$_4$", respectively, and $\rho_{NaOH}(I_T)$ and $\rho_{NaAl(OH)_4}(I_T)$ are the densities of the respective binary

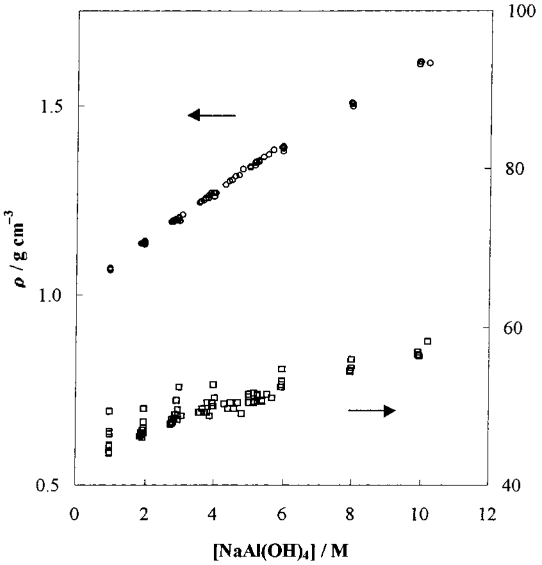

Figure 3. Densities (ρ) and apparent molar volumes (V_ϕ) of the hypothetical pure "NaAl(OH)4" aqueous solutions (calculated using Young's rule) as a function of ionic strength.

solutions at I_T. Young's rule can also be expressed

$$\rho_T({}^mI_T) = \frac{{}^mI_{NaOH}}{{}^mI_T}\rho_{NaOH}({}^mI_T) + \frac{{}^mI_{NaAl(OH)_4}}{{}^mI_T}\rho_{NaAl(OH)_4}({}^mI_T) \tag{3}$$

where the pre-superscript m indicates stoichiometric ionic strengths expressed in terms of molality. It can be shown that if the density of the mixture, $\rho_T(I_T)$, varies linearly with $I_{NaAl(OH)_4}$, then the system follows Young's rule and the mixing in terms of densities can be considered ideal. As seen in Figure 2, the mixture densities display satisfactorily linear dependence (with $R^2 > 0.999$) on substitution of OH^- by $Al(OH)_4^-$. It is noteworthy that the linear behavior of the density versus ionic strength functions is valid for both units (molar and molal) applied, even though Young's rule is strictly followed only on the molarity scale.[17] For this reason and also because volume-based units are preferred in engineering contexts, the molarity-based Young's rule was selected to predict the densities and apparent molar volumes of the hypothetical pure binary "NaAl(OH)4" solution (Figure 3).

Any significant nonideal mixing would manifest itself as a nonuniform change in the slope of the curves with respect to ionic strength. Such effects are not apparent in the density curve but are clear (Figure 3) in the much more sensitive values of apparent molar volume (V_ϕ) that were calculated according to

$$V_\phi = \frac{1000(\rho_{H_2O} - \rho)}{c\rho_{H_2O}} + \frac{M_2}{\rho_{H_2O}} \tag{4}$$

where M_2 is the molar mass of the solute. The nonuniform variations in V_ϕ might indicate a second-order nonideal mixing effect, but they could also be due to small systematic experimental errors (most likely associated with solution concentrations), to which V_ϕ is particularly sensitive.

The density of hypothetical "NaAl(OH)4" solutions at 25 °C was correlated with the empirical Masson equation[18]

$$\rho = \rho_{H_2O} + aI + bI^{3/2} \tag{5}$$

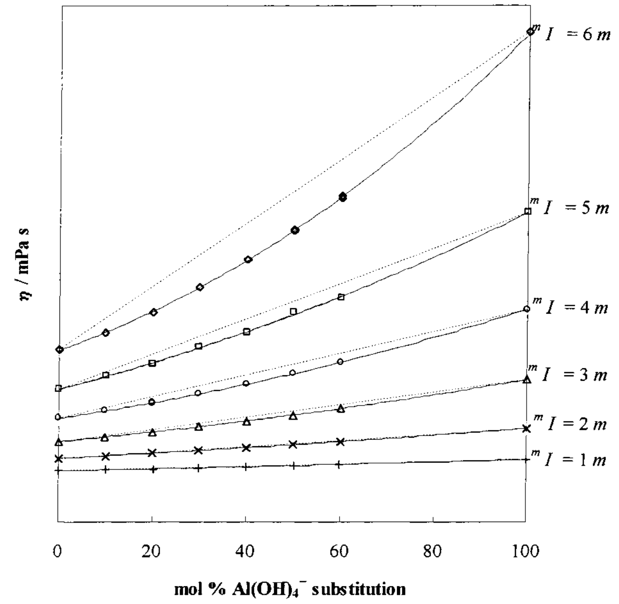

Figure 4. Viscosities (η) of isomolal series of NaOH/NaAl(OH)4 mixtures at various ionic strengths.

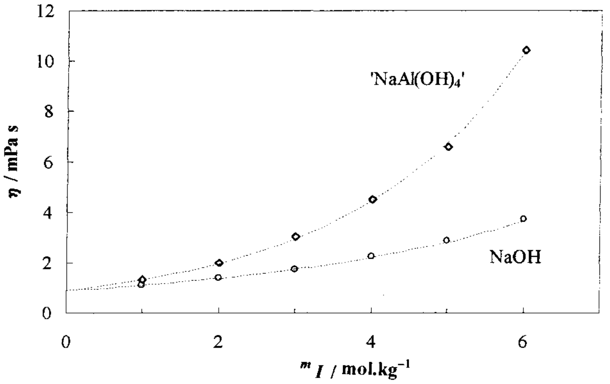

Figure 5. Viscosities (η) of NaOH and the hypothetical pure "NaAl(OH)4" aqueous solutions as a function of the molal ionic strength. The dotted lines are calculated using the data in Table 4.

Table 3. Parameters for Calculation of the Density of NaOH and NaAl(OH)4 Solutions via the Masson Equation at 25 °C

electrolyte	a	b	avg % residual
NaOH	46.922 10	−4.468 92	0.07
NaAl(OH)4	79.901 28	−5.835 43	0.22

where a and b are temperature-dependent empirical parameters. Despite its well-established inadequacies for correlating V_ϕ values at low ionic strengths,[19] the Masson equation provides an excellent fit of density data for many electrolyte solutions over very wide concentration ranges.[12,18,20] The values of a and b for "NaAl(OH)4" and for NaOH (latter based on NaOH density data from several sources at a range of ionic strengths[12,18,21−24]) and the average percentage residuals are given in Table 3. The higher residuals for "NaAl(OH)4" are probably associated with the uncertainties of the extrapolation but might also reflect speciation changes in the concentrated aluminate solutions.[15]

Viscosities. The viscosities of the NaOH/NaAl(OH)4 mixtures as a function of the substitution of OH^- with $Al(OH)_4^-$ are, as for the mixtures of simple electrolytes, characteristically concave at all ionic strengths (Figure 4). This is consistent with the observations of Nowlan et al.[25]

Table 4. Parameters for the Calculation of the Viscosity of NaOH/NaAl(OH)$_4$ Mixtures at 25 °C via Eqs 6 and 7

B_{NaOH}	0.091 517 9
D_{NaOH}	0.001 705 19
$B_{\mathrm{NaAl(OH)_4}}$	0.169 237
$D_{\mathrm{NaAl(OH)_4}}$	0.001 415 47
x	0.020 319 3
y	0.846 288
avg % residual	0.57

that the viscosity of ternary electrolyte mixtures is always lower than that predicted on a simple Young's rule basis. The viscosities of hypothetical pure "NaAl(OH)$_4$" solutions at 25 °C were determined by fitting each data set to a quadratic equation and extrapolation to 100% substitution of NaOH with NaAl(OH)$_4$ (Figure 4). Although the extrapolated values of η ("NaAl(OH)$_4$") are unusually high (Figure 5), the shape of the η versus [NaAl(OH)$_4$]$_{\mathrm{T}}$ curve is typical of other aqueous electrolyte solutions.[21,26]

Deviations in viscosity from Young's rule showed a quadratic dependence on the ionic strength fraction of "NaAl(OH)$_4$" and an exponential variation with ${}^mI_{\mathrm{T}}$. Introduction of a correction term in the isomolality mixing rule to account for this permits the complete matrix of NaOH/NaAl(OH)$_4$ solutions to be accurately correlated:

$$\eta_{\mathrm{T}}({}^mI_{\mathrm{T}}) = \frac{{}^mI_{\mathrm{NaOH}}}{{}^mI_{\mathrm{T}}}\eta_{\mathrm{NaOH}}({}^mI_{\mathrm{T}}) + \frac{{}^mI_{\mathrm{NaAl(OH)_4}}}{{}^mI_{\mathrm{T}}}\eta_{\mathrm{NaAl(OH)_4}}({}^mI_{\mathrm{T}}) - x\exp({}^mIy)\left\{\frac{{}^mI_{\mathrm{NaAl(OH)_4}}}{{}^mI_{\mathrm{T}}}\left(1-\frac{{}^mI_{\mathrm{NaAl(OH)_4}}}{{}^mI_{\mathrm{T}}}\right)\right\} \qquad (6)$$

where x and y are empirical parameters that quantify the changes in the deviation from ideal (Young's rule) mixing.

The viscosities of the pure binary solutions were expressed as

$$\log_{10}\frac{\eta_{\mathrm{NaX}}}{\eta_{\mathrm{H_2O}}} = B_{\mathrm{NaX}}{}^mI + D_{\mathrm{NaX}}{}^mI^2 \qquad (7)$$

where B_{NaX} and D_{NaX} ($\mathrm{X^-} = \mathrm{OH^-}$ and $\mathrm{Al(OH)_4}^-$) are empirical parameters. Values for x, y, B_{NaX}, and D_{NaX} for eqs 6 and 7 are given in Table 4. These parameters enable prediction of the viscosities of NaOH/NaAl(OH)$_4$ mixtures over the ranges $1 \le [\mathrm{NaOH}]_{\mathrm{T}}/\mathrm{mol{\cdot}kg^{-1}} \le 6$ and $0 \le [\mathrm{Al(III)}]_{\mathrm{T}}/[\mathrm{Na^+}]_{\mathrm{T}} \le 0.6$, with a 0.54% average error and a maximum error of 1.52% at $I = 6$ mol·kg^{-1} and $[\mathrm{Al(III)}]_{\mathrm{T}} = 0$.

Acknowledgment

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