Viscosity of the Tributyl Phosphate + Methyl Isobutyl Ketone + Phosphoric Acid System

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In this paper, kinetic viscosities of the phosphoric acid + tributyl phosphate + methyl isobutyl ketone, and + aviation kerosene systems with the content of P_2O_5 in the range of (0 to 17) % and temperature from (10 to 50) °C were presented. After data processing, kinetic viscosities were found well-correlated with temperature and concentration of P_2O_5, which can be demonstrated by linear regression equations. According to those equations, kinetic viscosities of the phosphoric acid + tributyl phosphate + methyl isobutyl ketone were compared with those of phosphoric acid + tributyl phosphate + aviation kerosene systems. The results showed that the dilution effect of methyl isobutyl ketone was better than that of aviation kerosene under the same temperature and concentration of P_2O_5. In addition, the concentration of P_2O_5 in the tributyl phosphate + methyl isobutyl ketone system can be higher than that in the tributyl phosphate + aviation kerosene system without any three-phase situation. So, there are many advantages for purifying wet-process phosphoric acid by solvent extraction with tributyl phosphate + methyl isobutyl ketone.

Introduction

One method used to obtain pure phosphoric acid is extracting wet-process phosphoric acid (WPA) with organic solvents. Many organic solvents can be used to purify WPA. As an example, methyl isobutyl ketone (MIBK) has already been widely used.[1−4] The use of tributyl phosphate (TBP) has also been reported.[5−11] Both solvents are attractive because of their immiscibility with aqueous solutions, good selectivity to phosphoric acid. and easy recovery. The extraction of phosphoric acid with MIBK can be carried out at room temperature, but the process will be effective only when the mass fraction of P_2O_5 is larger than 43.47 %.[12] TBP is often diluted by using kerosene at higher temperature because of its high viscosity. TBP has a better selectivity in phosphoric acid extraction than MIBK. However, the mass fraction of P_2O_5 in the organic phase can not be too high or three phases will appear.[13] Three phases do not occur in the TBP + MIBK mixture. Ahmed Hannachi et al.[14] have studied the mixture of TBP + MIBK and shown that the mixture extractant has good selectivity for phosphoric acid. However, the physicochemical properties of TBP + MIBK have not been reported. Therefore, the kinetic viscosities of phosphoric acid (PA) + TBP + MIBK in this work should be helpful for developing the corresponding extraction technology.

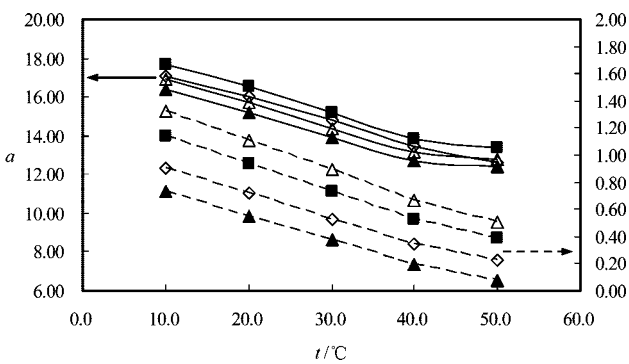

Figure 1. Coefficients *a* and *b* in eq 3 versus temperature *t* for mixtures of TBP + MIBK, and + AK. ■, a_A, b_A for mixtures of A (V_{TBP}/V_{MIBK} = 8/2); ◇, a_B, b_B for mixtures of B (V_{TBP}/V_{MIBK} = 7/3); ▲, a_C, b_C for mixtures of C (V_{TBP}/V_{MIBK} = 6/4); Δ, a_D, b_D for mixtures of D (V_{TBP}/V_{MIBK} = 7/3) by eq 3.

Table 1. Viscosity *η* of Pure Tributyl Phosphate and Methyl Isobutyl Ketone at 25 °C[a]

pure matter	η_{exptl}/(mPa·s)	$\eta_{lit.}$/(mPa·s)	100 Δ
tributyl phosphate	3.45	3.41[17]	1.2
methyl isobutyl ketone	0.56	0.542[18]	3.7

[a] Note: 100 Δ = $(\eta_{exptl} - \eta_{lit.})/\eta_{lit.}\cdot 100$.

Experimental Section

Materials. TBP, MIBK, and PA obtained from Chengdu Kelong Chemical Reagent Co. were all analytical grade. Their purity is above 98.5 %, 99.0 %, and 61.58 % in P_2O_5 mass fraction. Aviation kerosene (AK) was supplied by Henan Cangzhou Canglian Special Oil Co. Ltd. Its total efflux is 98 % at the distillation range where the initial boiling point is 233 °C and the dry point is 256 °C. The viscosities of pure TBP and MIBK are listed in Table 1.

Apparatus and Procedure. P_2O_5 content was determined by volumetric titration with NaOH using bromocresel green and phenolphthalein indicators with an uncertainty of ± 0.2 %.

The density was measured with an Ostwald−Sprenge-type pycnometers having a bulb volume of 25 cm^3 and an internal capillary diameter of about 1 mm. The internal volumes of the pycnometers were calibrated with pure water at each of the measured temperatures, and the densities of water were taken from the literature.[15] The thoroughly cleaned and dried pyc-

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Table 2. Values of Kinetic Viscosity v, and Density ρ of Mixtures A, B, C and D as a Function of Mass Fraction of w_1 of P_2O_5 at Different Temperatures[a]

	t = 10 °C				t = 20 °C				t = 30 °C				t = 40 °C				t = 50 °C			
$100\,w_1$	$\rho_{exptl.}$ $g \cdot cm^{-3}$	v_{exptl} $mm^2 \cdot s^{-1}$	$\ln v_{exptl}$	$100\,\Delta$	$\rho_{exptl.}$ $g \cdot cm^{-3}$	v_{exptl} $mm^2 \cdot s^{-1}$	$\ln v_{exptl}$	$100\,\Delta$	$\rho_{exptl.}$ $g \cdot cm^{-3}$	v_{exptl} $mm^2 \cdot s^{-1}$	$\ln v_{exptl}$	$100\,\Delta$	$\rho_{exptl.}$ $g \cdot cm^{-3}$	v_{exptl} $mm^2 \cdot s^{-1}$	$\ln v_{exptl}$	$100\,\Delta$	$\rho_{exptl.}$ $g \cdot cm^{-3}$	v_{exptl} $mm^2 \cdot s^{-1}$	$\ln v_{exptl}$	$100\,\Delta$
										A										
0.00	0.9485	3.12	1.14	−0.9	0.9418	2.55	0.94	0.2	0.9353	2.07	0.73	2.5	0.9290	1.68	0.52	7.1	0.9229	1.45	0.37	−2.8
1.20	0.9560	3.77	1.33	0.9	0.9502	3.02	1.10	2.7	0.9432	2.41	0.88	5.5	0.9350	1.95	0.67	8.7	0.9256	1.72	0.54	−3.5
3.26	0.9680	5.19	1.65	3.4	0.9642	4.09	1.41	4.4	0.9592	3.20	1.16	6.7	0.9530	2.52	0.92	9.9	0.9456	2.16	0.77	2.9
5.62	0.9943	8.99	2.20	−3.5	0.9868	6.87	1.93	−3.6	0.9803	5.18	1.65	−2.7	0.9748	3.93	1.37	−1.4	0.9703	3.28	1.19	−7.3
8.13	1.0074	13.69	2.62	−2.1	0.9996	10.12	2.31	−1.9	0.9926	7.36	2.00	−0.6	0.9864	5.39	1.68	1.2	0.9810	4.47	1.50	−4.5
12.41	1.0496	30.19	3.41	−2.6	1.0366	21.37	3.06	−3.0	1.0256	14.70	2.69	−2.0	1.0166	10.16	2.32	−0.4	1.0096	8.22	2.11	−5.4
17.45	1.0939	63.49	4.15	1.4	1.0854	42.47	3.75	1.3	1.0779	27.12	3.30	3.1	1.0714	17.49	2.86	5.6	1.0659	14.35	2.66	−0.4
										B										
0.00	0.9260	2.56	0.94	−4.9	0.9218	2.10	0.74	−3.1	0.9168	1.72	0.54	0.3	0.9110	1.41	0.34	7.3	0.9044	1.24	0.22	−9.2
1.15	0.9354	2.96	1.09	0.4	0.9302	2.43	0.89	1.7	0.9242	1.98	0.68	4.3	0.9174	1.62	0.48	9.1	0.9098	1.42	0.35	−2.5
3.34	0.9512	4.17	1.43	2.6	0.9463	3.35	1.21	3.4	0.9408	2.67	0.98	5.3	0.9347	2.13	0.76	8.7	0.9280	1.84	0.61	0.9
6.22	0.9709	7.04	1.95	0.5	0.9670	5.45	1.70	0.7	0.9625	4.19	1.43	1.8	0.9574	3.24	1.18	3.3	0.9517	2.77	1.02	−4.0
8.97	0.9920	12.03	2.49	−2.2	0.9882	9.02	2.20	−2.6	0.9832	6.67	1.90	−1.8	0.9770	4.94	1.60	−0.5	0.9696	4.09	1.41	−6.0
12.67	1.0219	22.97	3.13	−2.1	1.0187	16.48	2.80	−2.5	1.0139	11.57	2.45	−1.7	1.0075	8.22	2.11	−0.7	0.9995	6.81	1.92	−6.7
16.99	1.0663	42.57	3.75	1.6	1.0570	29.75	3.39	0.7	1.0515	19.89	2.99	1.7	1.0476	13.01	2.57	4.4	1.0446	9.73	2.28	2.5
										C										
0.00	0.9150	2.15	0.76	−6.0	0.9089	1.78	0.58	−4.2	0.9034	1.47	0.39	−1.2	0.8985	1.23	0.21	5.1	0.8942	1.10	0.09	−14.5
0.90	0.9203	2.39	0.87	−1.0	0.9169	1.99	0.69	0.1	0.9119	1.65	0.50	2.5	0.9053	1.36	0.31	8.6	0.8971	1.20	0.18	−12.0
3.18	0.9369	3.28	1.19	3.9	0.9320	2.68	0.98	4.8	0.9273	2.17	0.78	7.0	0.9228	1.76	0.57	11.5	0.9183	1.53	0.42	2.6
6.22	0.9626	5.59	1.72	0.4	0.9575	4.39	1.48	0.8	0.9522	3.42	1.23	2.2	0.9467	2.68	0.99	4.3	0.9412	2.29	0.83	−3.0
8.14	0.9785	7.87	2.06	−1.1	0.9717	6.04	1.80	−1.0	0.9657	4.59	1.52	0.2	0.9605	3.51	1.26	1.9	0.9561	2.97	1.09	−4.6
12.49	1.0110	18.07	2.89	−5.1	1.0061	12.51	2.53	−3.5	1.0014	8.50	2.14	−0.1	0.9967	6.05	1.80	2.7	0.9922	5.40	1.69	−7.2
17.39	1.0554	33.28	3.51	1.1	1.0483	23.30	3.15	0.9	1.0418	15.83	2.76	2.3	1.0359	10.88	2.39	4.2	1.0306	8.93	2.19	−1.3
										D										
0.00	0.9275	3.82	1.34	−1.9	0.9222	3.09	1.13	−1.8	0.9173	2.48	0.91	−1.0	0.9127	1.98	0.68	1.1	0.9083	1.69	0.53	−7.9
1.65	0.9379	4.98	1.61	−0.8	0.9334	3.94	1.37	−0.4	0.9286	3.09	1.13	1.0	0.9236	2.42	0.89	3.3	0.9187	2.06	0.72	−4.3
3.04	0.9476	6.13	1.81	0.8	0.9430	4.75	1.56	1.6	0.9381	3.65	1.29	3.6	0.9329	2.82	1.04	6.2	0.9278	2.40	0.87	−1.1
4.58	0.9592	8.18	2.10	−0.7	0.9536	6.25	1.83	−0.5	0.9485	4.71	1.55	1.0	0.9431	3.56	1.27	3.0	0.9382	2.99	1.10	−3.5
6.04	0.9710	10.58	2.36	−1.1	0.9636	7.95	2.07	−1.0	0.9582	5.88	1.77	0.3	0.9529	4.37	1.47	2.2	0.9483	3.62	1.29	−3.5
7.90	0.9874	14.36	2.66	−0.7	0.9765	10.54	2.35	−0.5	0.9706	7.59	2.03	1.0	0.9655	5.52	1.71	2.9	0.9616	4.58	1.52	−3.1
9.30	1.0006	18.25	2.90	−0.8	0.9863	13.17	2.58	−0.6	0.9797	9.31	2.23	0.9	0.9749	6.65	1.89	2.7	0.9720	5.51	1.71	−3.4

[a] Note: (1) $100\,\Delta = (\ln v_{exptl} - \ln v_{calcd})/\ln v_{exptl} \cdot 100$; (2) A, B, C, and D are the mixtures of PA + TBP + MIBK (V_{TBP}/V_{MIBK} = 8/2, 7/3, 6/4) and PA + TBP + AK (V_{TBP}/V_{AK} = 7/3).

Table 3. Coefficients *a* and *b* and Standard Deviations *σ* in Equation 3 at Different Temperatures

coefficients in eq 3	temperature, °C 10	20	30	40	50
			A		
a_A	17.675	16.545	15.158	13.808	13.379
b_A	1.1412	0.9354	0.7309	0.5293	0.3870
σ	0.066	0.065	0.065	0.064	0.051
			B		
a_B	17.059	16.038	14.781	13.454	12.621
b_B	0.9085	0.7160	0.5263	0.3421	0.2241
σ	0.052	0.046	0.042	0.047	0.067
			C		
a_C	16.356	15.202	13.878	12.703	12.399
b_C	0.7291	0.5503	0.3735	0.1960	0.0718
σ	0.071	0.048	0.028	0.022	0.041
			D		
a_D	16.940	15.740	14.380	13.164	12.793
b_D	1.3261	1.1112	0.8909	0.6678	0.5101
σ	0.015	0.017	0.018	0.016	0.013

Table 4. Coefficients *k*, *l*, *m*, and *n* and Standard Deviations *σ* in Equation 4 and Equation 5[a]

	eq 4			eq 5		
mixture	k	l	σ	m	n	σ
A	19.001	−0.0074	0.258	−0.0191	1.3191	0.023
B	18.568	−0.0078	0.148	−0.0174	1.0662	0.027
C	17.483	−0.0073	0.283	−0.0167	0.8848	0.020
D	18.132	−0.0074	0.269	−0.0208	1.5238	0.022

[a] Note: A, B, C, and D are the mixtures of PA + TBP + MIBK (V_{TBP}/V_{MIBK} = 8/2, 7/3, 6/4) and PA + TBP + AK (V_{TBP}/V_{AK} = 7/3).

nometers were first weighed on an electronic balance (type AR1140, USA. Ohaus Corps.) with precision within ± 0.0001 g and then filled with experimental liquid and immersed in a thermostat (type 501, Shanghai Laboratory Instrument Works Co., Ltd.) with constant temperature (± 0.1 °C). After thermal equilibrium had been achieved at the required temperature, the pycnometers were removed from the thermostat and properly cleaned, dried, and weighed. The density was then determined from the mass of the sample and the volume of the pycnometers. The readings from pycnometers were averaged. Uncertainties in the density measurements were estimated to be within ± 0.0001 $g \cdot cm^{-3}$.

The viscosity η was measured using a commercial Ostwald capillary viscometer (type1831-1, Shanghai Glass Instruments Factory, China) of 0.7 mm in diameter, calibrated at (10, 20, 30, 40, and 50) °C with doubly distilled water. A thoroughly cleaned and dried viscometer, filled with experimental liquid, was placed vertically in an insulated aquarium where constant temperature (± 0.1 °C) was maintained by circulating water from a thermostatically controlled water bath at the required temperature. After thermal stability was attained, the flow times of the liquids were recorded with an electronic digital stop watch with a precision of ± 0.01 s. At least three repetitions of each datum point obtained were reproducible to ± 0.05 s, and the results were averaged. The viscosity η of the liquids was calculated[16] using

$$\frac{\eta}{\eta_w} = \frac{\rho t}{\rho_w t_w} \tag{1}$$

and the kinetic viscosity v was calculated from the following relationship

$$v = \frac{\eta}{\rho} \tag{2}$$

where η, ρ, and t and η_w, ρ_w, and t_w are the viscositiy, density, and flow time of the mixtures and water, respectively. The values of the viscosity and density of pure water are collected from the literature.[15] The uncertainty of the kinetic viscosity is ± 0.01 $mm^2 \cdot s^{-1}$.

Results and Discussion

This paper presents data on the kinetic viscosity of mixtures of PA + TBP + MIBK, and + AK as follows

$$\text{Mixture A: PA} + \text{TBP} + \text{MIBK}(V_{TBP}/V_{MIBK} = 8/2)$$

$$\text{Mixture B: PA} + \text{TBP} + \text{MIBK}(V_{TBP}/V_{MIBK} = 7/3)$$

$$\text{Mixture C: PA} + \text{TBP} + \text{MIBK}(V_{TBP}/V_{MIBK} = 6/4)$$

$$\text{Mixture D: PA} + \text{TBP} + \text{AK}(V_{TBP}/V_{AK} = 7/3)$$

where V_{TBP}, V_{MIBK}, and V_{AK} are the volume of TBP, MIBK, and AK.

The experimental kinetic viscosities and densities for all experimental mixtures at all temperature are listed in Table 2.

A graphical analysis and mathematical processing of the data are shown in Table 2. In this table, v is logarithmic. It can be represented as

$$\ln(v/\text{mm}^2 \cdot \text{s}^{-1}) = aw + b \tag{3}$$

where w is the mass fraction of P_2O_5 and a, and b are coefficients of eq 3.

Table 3 lists values of coefficients a and b for each mixture at different temperatures. The data in Table 3 were used to plot the temperature dependence on coefficients a and b (see Figure 1) for mixtures TBP + MIBK, and + AK. It is worthy to note that the temperature dependence on coefficients is similar among all four mixtures. Coefficient a can be described as

$$a = k \exp(lt/°\text{C}) \tag{4}$$

and b as

Table 5. Kinetic Viscosities *v* Calculated by Equation 7, Equation 8, and Equation 10 for Mixtures A, B, and D at Different Temperature and Mass Fraction of P_2O_5

	t = 10 °C			t = 20 °C			t = 30 °C			t = 40 °C			t = 50 °C		
100 w_1	A	B	D	A	B	D	A	B	D	A	B	D	A	B	D
0	3.09	2.44	3.73	2.55	2.05	3.03	2.11	1.72	2.46	1.74	1.45	2.00	1.44	1.22	1.62
1	3.69	2.90	4.41	3.01	2.40	3.54	2.46	2.00	2.84	2.01	1.66	2.29	1.64	1.38	1.84
3	5.25	4.09	6.18	4.17	3.30	4.84	3.33	2.68	3.80	2.66	2.18	2.99	2.13	1.77	2.36
5	7.47	5.76	8.65	5.79	4.54	6.62	4.51	3.59	5.08	3.53	2.86	3.92	2.77	2.28	3.03
7	10.63	8.12	12.11	8.04	6.24	9.05	6.12	4.82	6.80	4.69	3.75	5.13	3.61	2.93	3.90
9	15.12	11.45	16.97	11.16	8.57	12.37	8.30	6.47	9.09	6.22	4.92	6.72	4.69	3.77	5.01
11	21.52	16.14	23.76	15.48	11.77	16.91	11.25	8.68	12.15	8.25	6.46	8.80	6.10	4.85	6.43
13	30.63	22.76		21.49	16.17		15.25	11.64		10.94	8.47		7.93	6.24	
15	43.60	32.09		29.82	22.22		20.67	15.62		14.51	11.12		10.31	8.02	
17	62.05	45.24		41.38	30.53		28.03	20.95		19.25	14.60		13.40	10.31	

$$b = mt/\,^{\circ}\mathrm{C} + n \qquad (5)$$

where t is temperature; k and l are coefficients of eq 4, and m and n are coefficients of eq 5.

Table 3 lists the standard deviations σ for each mixture at different temperature. σ was calculated using the following formula

$$\sigma = \left(\sum_{i=1}^{p} (\ln v_{\mathrm{exptl}} - \ln v_{\mathrm{calcd}})^2/(p-q)\right)^{\frac{1}{2}} \qquad (6)$$

where p refers to the number of data points and q is the number of coefficients.

Standard deviation σ in Table 3 is very small, which demonstrates a high goodness of fit in eq 3.

Table 4 lists the values of the coefficients in eq 4 and eq 5, together with the standard deviations σ for all four types of mixtures. Also, it shows a pretty reasonable goodness of fit in eq 4 and eq 5.

Thus, the kinetic viscosity of mixture A, B, C, and D can be calculated by the equations below

$$\ln(v_{\mathrm{A}}/\mathrm{mm^2{\cdot}s^{-1}}) = 19.001 \exp(-0.0074t/^{\circ}\mathrm{C})w - 0.0191/^{\circ}\mathrm{C} + 1.3191 \qquad (7)$$

$$\ln(v_{\mathrm{B}}/\mathrm{mm^2{\cdot}s^{-1}}) = 18.568 \exp(-0.0078t/^{\circ}\mathrm{C})w - 0.0174t/^{\circ}\mathrm{C} + 1.0662 \qquad (8)$$

$$\ln(v_{\mathrm{C}}/\mathrm{mm^2{\cdot}s^{-1}}) = 18.483 \exp(-0.0073t/^{\circ}\mathrm{C})w - 0.0167t/^{\circ}\mathrm{C} + 0.8848 \qquad (9)$$

$$\ln(v_{\mathrm{D}}/\mathrm{mm^2{\cdot}s^{-1}}) = 18.132 \exp(-0.0074t/^{\circ}\mathrm{C})w - 0.0208t/^{\circ}C + 1.5238 \qquad (10)$$

where v_{A}, v_{B}, v_{C}, and v_{D} refer to the kinetic viscosity of mixture A, B, C, and D while $V_{\mathrm{TBP}}/V_{\mathrm{MIBK}} = 8/2$, 7/3, 6/4 and $V_{\mathrm{TBP}}/V_{\mathrm{AK}} = 7/3$.

Table 2 lists the errors between experimental data and calculation data from eq 7 to eq 10.

The formulation for error Δ is shown below

$$100\ \Delta = \frac{\ln v_{\mathrm{exptl}} - \ln v_{\mathrm{calcd}}}{\ln v_{\mathrm{exptl}}} \cdot 100 \qquad (11)$$

where $\ln v_{\mathrm{calcd}}$ is determined by eq 7, eq 8, eq 9, and eq 10.

Table 2 shows that the most error Δ is smaller than 10.00 %, which demonstrates a high goodness of fit in eq 7, eq 8, eq 9, and eq 10.

To compare v of mixtures A, B, and D, Table 5 list their kinetic viscosities at different temperature and concentration of P_2O_5. Kinetic viscosities are calculated according to eq 7, eq 8 and eq 10. It is easy to see that, under the same temperature and content of P_2O_5, the values of kinetic viscosity in those three types of mixtures decrease in the following order: $v_{\mathrm{D}} > v_{\mathrm{A}} > v_{\mathrm{B}}$. Thus, the diluent effect of MIBK is better than that of aviation kerosene. Moreover, the mass fraction of P_2O_5 in mixtures of TBP + MIBK can be higher than that in TBP + AK according to experiments. So there are many advantages for purifying WPA by solvent extraction with TBP + MIBK.

Conclusions

The kinetic viscosities of PA + TBP + MIBK, and + AK are obtained under the temperature range from (10 to 50) °C and $w_{\mathrm{P_2O_5}} = (0$ to $17)$ %, and they are found well-correlated with temperature and concentration of P_2O_5. According to the results, the diluent effect of MIBK should be better than that of AK, and the mass fraction of P_2O_5 in the mixture of TBP + MIBK can be higher than that in TBP + AK.

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