



DENSITY AND VISCOSITY OF TRIBUTYL PHOSPHATE + TRIOCTYLAMINE + PHOSPHORIC ACID FROM (20~60)°C

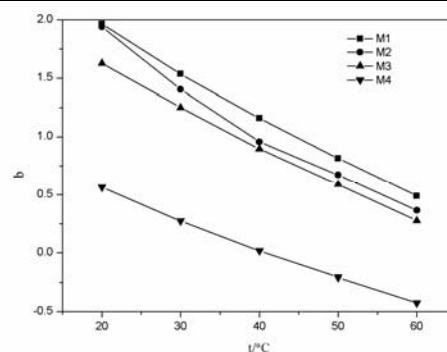
Kun ZHOU,^{a,b*} Ke WANG^a and Feng LIU^a

^a College of Materials and Chemistry & Chemical Engineering, Chengdu University of Technology, Chengdu 610059, China

^b Mineral Resources Chemistry Key Laboratory of Sichuan Higher Education Institutions, Chengdu University of Technology, Chengdu 610059, China

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The viscosities and densities of tributyl phosphate (TBP) + trioctylamine (TOA) + phosphoric acid (PA) with the mass fraction of H_3PO_4 in the range of (0 to 9) % and under temperature from (10 to 60)°C were measured. After a series of data processing, it is found that the measured viscosities are well-correlated with the temperature and mass fraction of H_3PO_4 and fitted to regression equations. The result shows that the effect of the mass fraction of TBP in the mixture (TBP+TOA) on the viscosity of TBP+TOA+PA system is in the order of $M1 > M2 > M3 > M4$ (M for the ratio of TBP and TOA, $M1 = 8:2$, $M2 = 7:3$, $M3 = 6:4$, $M4 = 5:5$) under the same temperature and mass fraction of H_3PO_4 . Meanwhile, with a series of studying on experimentation, the technical routes and parameters are optimized.



INTRODUCTION

Phosphoric acid (PA) is one of the most widely produced and utilized acids in the world. It has many different kinds of applications, such as a chemical intermediate in the production of numerous phosphate fertilizers, food additives, detergents and agricultural feeds.^{1,2} There are two common industrial techniques to produce phosphoric acid in the world. They are called thermal and wet process technologies.³ There are some papers which have provided a reference for wet process phosphoric acid in recent years.⁴⁻⁸ TBP as an important extractant has been reported.⁹⁻¹⁵ But the densities and viscosities measurements under TBP+TOA at different conditions has been rarely covered. TBP can't be used alone in extraction of phosphoric acid because of its high viscosity.¹⁶ The system of TBP+TOA has a good

extraction effect and entrained little water phase. The experiment of PA+TBP+TOA can provide basic data about purification for the phosphate chemical industry, and can adapt to the rapid development of wet-process phosphoric acid industry.

The viscosity of extractant have a lot of draw backs to the extraction process. The P_2O_5 can be transferred to the organic phase. Meanwhile, the organic phase mixed with P_2O_5 and water phase can be separated well to facilitate subsequent operating smoothly, or it will increase the consumption of washing, even can not meet the industrial requirements. The high viscosity of the extractant is unfavorable to extraction process because phosphoric acid is a high sticky liquid. The experiment has measured density and viscosity of tributyl phosphate (TBP) + trioctylamine (TOA) + phosphoric acid (PA) system from (20 ~ 60)°C

* Corresponding author: kunzhou925@163.com

(The ratio of trioctylamine and TBP is from 8/2 to 6/4, the mass fraction of PA is 0%~9%) and determined the best proportion of tributyl phosphate (TBP) and trioctylamine (Suitable extraction ratio). The experimental data about this work should be helpful for developing the corresponding extraction technology.

EXPERIMENTAL

TBP, trioctylamine and hydrochloric acid were supplied by Chengdu Ke-long Chemical Reagent Co. were all analytical grade. The purity of them is above 98.5%, 99% and 36.0%. The PA (The mass concentration is 85%, industrial grade) was offered by Gui Zhou Hong Fu Co., Ltd. For the viscosity measurements, the Oswald capillary viscometer (type 1831-1, Shanghai Glass Instruments Factory, China) 0.7 mm in diameter has been used. The analytical balance (with uncertainty of ± 0.0001 g) was supplied by Shanghai Heng ping Scientific Instrument Co., Ltd. The electronic balance (with uncertainty of ± 0.01 g) was obtained from Shanghai Ping Xuan Scientific Instrument Co., Ltd. The frequency conversion mixer was from Chengdu Yu Feng Industrial Enamel Co., Ltd.

The Ostwald capillary viscometer washed by distilled water and alcohol in turn was fixed on the bracket. Then, the viscometer was immersed in the aquarium which was full of water. In order to make sure the temperature is constant, the water in the aquarium should exceed the fixed glass rod of viscometer. The viscometer should be upright with the vertical. The thermometer was hanged inside the aquarium to check the temperature of the water.

The 25 mL of distilled water was added into viscometer. The water calibration was performed at 20°C, 30°C, 40°C, 50°C, 60°C (± 0.2) and controlled by a thermostat (type 501, Shanghai Laboratory Instrument Works Co., Ltd.). In order to keep a constant temperature, the viscometer was controlled by water bath for 10 minutes. The distilled water was sucked into the goal of the viscometer by the washing ear ball and it fixed glass rod which was submerged. But the distilled water was not into the washing ear ball. Then, the wash ears ball was taken and the water flowed down along the viscometer tube. The rolling time of the ball through the distilled water (τ_w) from the alignment A to B was measured with an electronic digital stop watch with precision of ± 0.01 s. The values of the process were at six replicates and the average was calculated. The density of distilled water of measured with Ostwald-Sprengel type pycnometers with a volume of 10 mL and the internal capillary diameter was about 1mm. The pycnometers empty and filled with distilled water were weighed on an electronic balance (type AR1140, U.S. Ohaus Corps.) at each of the experimental temperatures for three times. The uncertainty of the density measurements were estimated to be within $0.0001 \text{ g}\cdot\text{cm}^{-3}$.

The different ratio of extracting agents were stirred by a blender with 300rpm about ten minutes, then 25 ml extraction agent was taken five minutes later to join the viscometer. The processes for viscosity and density measurements of the liquid

mixtures were the same as for distilled water. The viscosity η of liquids was calculated by

$$\frac{\eta}{\eta_w} = \frac{\rho\tau}{\rho_w\tau_w} \quad (1)$$

where η , ρ , and τ and η_w , ρ_w , and τ_w are the viscosity, density, and flow time of the mixtures and water, respectively. The values of the density and viscosity of pure water are acquired from the literature.¹⁷ The uncertainty of viscosity was ± 0.03 mPa·s.

RESULTS AND DISCUSSION

The data on viscosity and density of the PA+TBP+TOA mixtures are presented as it follows: mixture (M₁), PA + TBP + TOA ($V_{\text{TBP}}: V_{\text{TOA}} = 8:2$); mixture (M₂), PA + TBP + TOA ($V_{\text{TBP}}: V_{\text{TOA}} = 7:3$); mixture (M₃), PA + TBP + TOA ($V_{\text{TBP}}: V_{\text{TOA}} = 6:4$); mixture (M₄) PA + TBP + TOA ($V_{\text{TBP}}: V_{\text{TOA}} = 5:5$).

Where V_{TBP} and V_{TOA} are volume of TBP and TOA.

The experimental viscosities and densities for all prepared mixtures at different temperatures are listed in Table 1 along with relative deviation of viscosity data from Eq. (1).

The viscosity dependence of PA mass fraction from Table 1 can be correlated with Eq. (2) by using least square method.

$$\ln(\eta/\text{mPa}\cdot\text{s}) = aw + b \quad (2)$$

where w is the mass fraction of H_3PO_4 , the a and b are coefficients of eq 2.

Table 2 lists values of coefficients a and b for eq 2. The coefficients a and b of the mixtures affected by the change of temperature can be easily seen in Fig. 1 and Fig. 2.

The temperature dependence of the coefficients a and b from eq 2 can be described by the following equations

$$a = k \exp(lt/^\circ\text{C}) \quad (3)$$

$$a = dt/^\circ\text{C} + q \quad (4)$$

and b can be described by

$$b = r \exp(st/^\circ\text{C}) \quad (5)$$

$$b = mt/^\circ\text{C} + n \quad (6)$$

where k , l , d , q , r , s , m , and n are the coefficients of eq 3, eq 4, eq 5 and eq 6, t is the temperature.

Table 1

Viscosity η and Density ρ for Mixture M1, M2, M3, and M4 in (20-60) °C as a Function of Mass Fraction of w of PA^a

w	t=20 °C				t=30 °C				t=40 °C				t=50 °C				t=60 °C			
	ρ g/cm ³	η mPa.s	100 Δ	η mPa.s	ρ g/cm ³	η mPa.s	100 Δ	η mPa.s	ρ g/cm ³	η mPa.s	100 Δ	η mPa.s	ρ g/cm ³	η mPa.s	100 Δ	η mPa.s	ρ g/cm ³	η mPa.s	100 Δ	η mPa.s
	M1																			
0.00%	0.8510	7.4214	3.941	4.9263	0.8455	4.9263	2.253	3.3590	0.8395	3.3590	1.624	2.4159	0.8342	2.4159	6.44	1.8001	0.8259	1.8001	0.245	0.245
0.94%	0.8561	10.0617	6.107	6.5536	0.8526	6.5536	5.093	4.2759	0.8468	4.2759	3.512	2.7751	0.8383	2.7751	-0.006	1.9212	0.8343	1.9212	1.905	1.905
3.44%	0.8763	18.6694	3.913	12.1324	0.8701	12.1324	4.471	6.9707	0.8637	6.9707	-0.965	4.7597	0.8578	4.7597	1.253	3.2871	0.8515	3.2871	5.513	5.513
4.85%	0.8855	30.6678	7.224	18.1892	0.8814	18.1892	6.141	11.0882	0.8764	11.0882	5.421	7.5874	0.8702	7.5874	9.506	4.4537	0.8632	4.4537	6.455	6.455
5.48%	0.8903	39.0210	8.890	21.4276	0.8831	21.4276	6.225	11.6730	0.8789	11.6730	-8.893	7.8588	0.8707	7.8588	-5.785	5.3182	0.8687	5.3182	9.089	9.089
6.55%	0.9009	53.8545	9.333	24.7647	0.8966	24.7647	2.453	16.8425	0.8839	16.8425	5.973	11.1132	0.8788	11.1132	9.167	6.7945	0.8775	6.7945	9.904	9.904
7.13%	0.9021	60.4863	8.254	26.4629	0.8979	26.4629	5.470	18.2376	0.8916	18.2376	4.088	12.4586	0.8856	12.4586	9.249	7.6639	0.8791	7.6639	9.718	9.718
8.63%	0.9148	80.3296	5.371	32.1561	0.9096	32.1561	4.590	21.0361	0.9012	21.0361	-2.418	13.4167	0.8963	13.4167	-0.902	8.6529	0.8907	8.6529	1.342	1.342
	M2																			
0.00%	0.8763	6.6033	2.329	4.4012	0.8713	4.4012	1.772	3.0409	0.8634	3.0409	4.005	2.2044	0.8576	2.2044	9.355	1.6394	0.8485	1.6394	2.676	2.676
1.06%	0.8834	9.1709	5.076	5.5623	0.8767	5.5623	0.768	3.5423	0.8701	3.5423	-3.000	2.5700	0.8638	2.5700	4.311	1.8212	0.8592	1.8212	5.636	5.636
2.93%	0.8964	12.6574	-0.942	7.4726	0.8893	7.4726	-6.350	4.8813	0.8818	4.8813	-8.332	3.4832	0.8763	3.4832	-3.985	2.4333	0.8688	2.4333	1.107	1.107
4.77%	0.9052	17.7582	-4.746	10.7500	0.9001	10.7500	-8.135	6.3726	0.8931	6.3726	-2.057	4.4344	0.8887	4.4344	-6.594	3.0919	0.8823	3.0919	-7.398	-7.398
5.69%	0.9076	21.6754	-5.292	14.9852	0.9046	14.9852	-2.793	9.2173	0.8987	9.2173	-4.890	6.3029	0.8940	6.3029	-2.112	4.3284	0.8872	4.3284	2.189	2.189
6.33%	0.9105	23.7645	-7.188	17.5438	0.9085	17.5438	-2.347	12.3539	0.9007	12.3539	1.683	7.7313	0.8963	7.7313	1.485	5.3257	0.8890	5.3257	6.637	6.637
7.31%	0.9161	25.9213	-4.830	21.6748	0.9121	21.6748	-2.742	15.3522	0.9063	15.3522	1.547	9.6598	0.8987	9.6598	2.043	6.2139	0.8934	6.2139	3.758	3.758
8.18%	0.9235	27.5545	-5.890	24.4125	0.9150	24.4125	-5.267	20.3673	0.9096	20.3673	4.378	12.3814	0.9028	12.3814	4.413	7.9615	0.8987	7.9615	6.842	6.842
	M3																			
0.00%	0.8844	5.5020	6.187	3.8115	0.8767	3.8115	5.576	2.6326	0.8670	2.6326	4.210	1.9580	0.8579	1.9580	8.4	1.4204	0.8514	1.4204	7.54	7.54
1.22%	0.8883	6.9658	2.316	4.5371	0.8805	4.5371	-1.789	3.1653	0.8736	3.1653	-2.760	2.3041	0.8677	2.3041	0.553	1.6637	0.8601	1.6637	6.229	6.229
3.04%	0.9022	10.3067	-0.235	6.5865	0.8958	6.5865	-3.498	4.3067	0.8917	4.3067	-7.329	2.9808	0.8828	2.9808	-8.654	2.1160	0.8758	2.1160	-7.978	-7.978

Table 1 (continued)

3.83%	0.9062	10.7733	-6.443	0.8978	6.9835	-9.575	0.8948	4.7727	-8.778	0.8877	3.4740	-7.726	0.8811	2.3044	-1.963
5.11%	0.9135	15.9085	-2.689	0.9098	10.4103	-3.259	0.9035	6.4338	-7.597	0.8965	4.4623	-6.465	0.8894	3.1113	-4.565
6.58%	0.9242	26.2108	2.074	0.9173	14.5318	-2.813	0.9102	9.3114	-3.639	0.9057	6.3314	-1.89	0.9022	4.3186	0.543
7.37%	0.9293	31.4537	1.685	0.9247	18.6652	-0.124	0.9176	10.9784	-3.457	0.9123	7.2231	-2.929	0.9121	5.0795	1.607
8.67%	0.9396	37.1429	-2.531	0.9351	23.3944	-2.278	0.9275	12.5428	-8.829	0.9237	8.4573	-7.256	0.9235	6.0234	-2.258
0.00%	0.9449	1.7660	5.765	0.9386	1.3182	-4.837	0.9319	1.0296	-6.615	0.9218	0.8347	-9.629	0.9111	0.6589	-7.691
0.96%	0.9514	1.9600	2.684	0.9468	1.4776	-1.955	0.9375	1.1260	-3.310	0.9266	0.8857	-9.906	0.9249	0.7022	-3.857
1.90%	0.9608	2.2712	5.968	0.9550	1.6313	-3.031	0.9478	1.2314	-2.521	0.9397	0.9683	-8.340	0.9325	0.7635	-6.279
2.75%	0.9691	2.5360	5.793	0.9631	1.8192	-0.303	0.9551	1.3538	-6.003	0.9487	1.0563	-0.506	0.9394	0.7942	7.085
3.55%	0.9744	2.7569	3.777	0.9708	1.9996	0.345	0.9642	1.4740	-6.217	0.9550	1.1527	-7.032	0.9460	0.8770	-6.744
4.35%	0.9840	3.1069	5.178	0.9780	2.2271	2.472	0.9701	1.6571	-9.054	0.9616	1.2510	-3.821	0.9546	0.9651	-5.282
5.08%	0.9874	3.4411	5.698	0.9854	2.4245	2.515	0.9769	1.7688	-9.398	0.9703	1.3530	-9.452	0.9637	1.0244	-8.267
5.77%	0.9947	3.8550	7.299	0.9910	2.6963	5.102	0.9845	1.9519	-7.505	0.9767	1.4700	-9.510	0.9718	1.1027	-2.903

M4

^aNote: $100\Delta = 100(\ln_{\text{exp}} - \ln_{\text{calcd}}) / \ln_{\text{exp}}$, M1, M2, M3 and M4 are the system of V_{TBP} : $V_{\text{TOA}} = (8:2, 7:3, 6:4, 5:5)$

Table 2
Coefficients a and b and R^2 for Equation 2 at temperature from (20–60) °C^a

coefficients in eq2	temperature, °C				
	20	30	40	50	60
M1=8:2					
a1	25.911	23.959	22.171	20.648	19.575
b1	1.9619	1.5364	1.15792	0.81524	0.48917
R^2	0.06130	0.02543	0.02021	0.02934	0.01171
M2=7:3					
a2	23.874	22.547	22.071	21.104	19.607
b2	1.93925	1.40407	0.95716	0.67184	0.36525
R^2	0.1164	0.00502	0.02822	0.1219	0.01099
M3=6:4					
a3	24.055	21.759	20.127	18.935	17.665
b3	1.62629	1.25004	0.89513	0.58869	0.27598
R^2	0.0099	0.0057	0.0058	0.0050	0.0048
M4=5:5					
a4	12.381	11.328	10.254	9.398	8.302
b4	0.56362	0.27282	0.0159	-0.20863	-0.42718
R^2	0.0016	0.0013	0.0012	0.0009	0.0008

^a Note: 1. The M1, M2, M3 and M4 are mixture (M₁), PA+TBP+TOA (V_{TBP}:V_{TOA}=8:2); mixture (M₂), PA+TBP+TOA (V_{TBP}:V_{TOA}=7:3); mixture (M₃), PA+TBP+TOA (V_{TBP}:V_{TOA}=6:4) and mixture (M₄)PA+TBP+TOA (V_{TBP}:V_{TOA}=5:5). 2. R^2 is the value of curve correlation factor for Equation 2

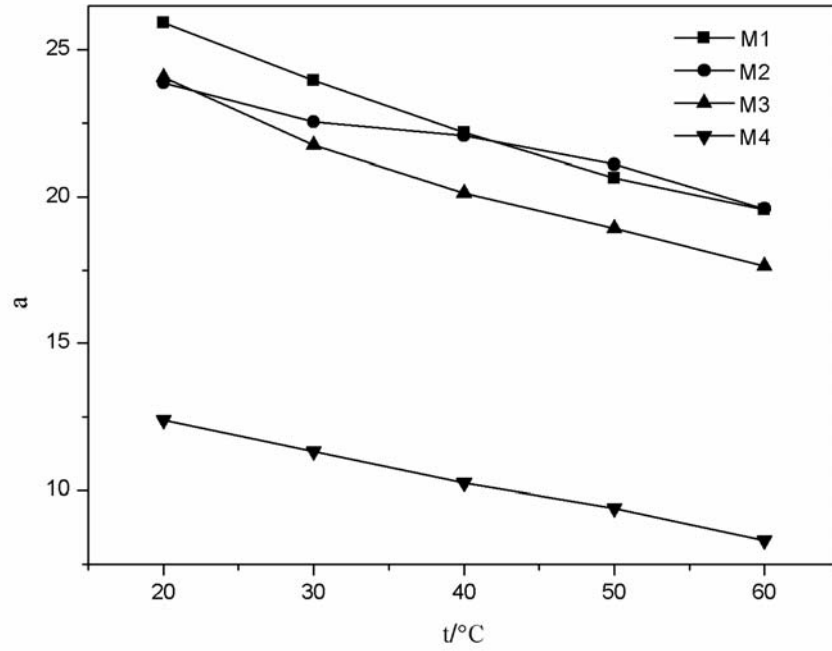


Fig. 1 – Coefficients a in eq 2 versus temperature (t) for mixtures of PA+TBP+TOA. a1, for mixtures of M₁(V_{TBP}:V_{TOA}=8:2); a2, for mixtures of M₂(V_{TBP}:V_{TOA}=7:3); a3, for mixtures of M₃(V_{TBP}:V_{TOA}=6:4); a4, for mixtures of M₄(V_{TBP}:V_{TOA}=5:5).

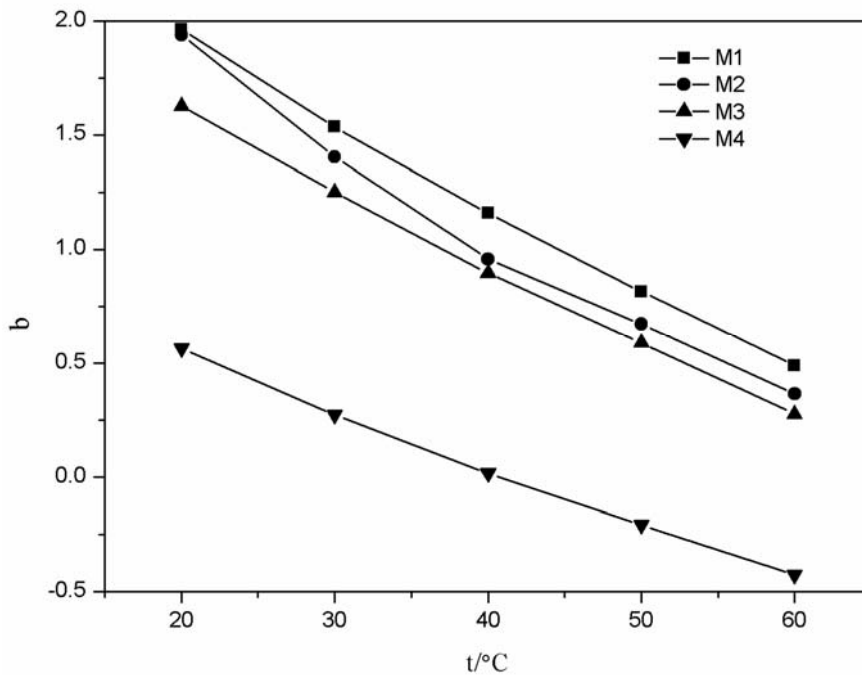


Fig. 2 – Coefficients b in eq2 versus temperature (t) for mixtures of PA+TBP+TOA. b1, for mixtures of M₁(V_{TBP}:V_{TOA}=8:2); b2, for mixtures of M₂(V_{TBP}:V_{TOA}=7:3); b3, for mixtures of M₃(V_{TBP}:V_{TOA}=6:4); b4, for mixtures of M₄(V_{TBP}:V_{TOA}=5:5).

The standard deviations R^2 from Table 3 and Table 4 is about each mixture at different temperature, the R^2 can be described by

$$R^2 = \left(\sum_{i=1}^y (\ln \eta_{\text{exptl}} - \ln \eta_{\text{calcd}})^2 / (y - x) \right) \quad (7)$$

where y is the number of data points and x is the number of coefficients.

Table 3
The Coefficients k, l, d, q and R² about eq3 and eq4.^a

mix	k	l	R ²	d	q	R ²
M1	29.76021	-0.00717	0.01084	-0.15983	28.8460	0.00888
M2	27.11323	-0.00502	0.00418	-0.11117	26.6674	0.00417
M3	28.04485	-0.00809	0.0005	-0.15604	26.7498	0.00048
M4	14.91473	-0.00927	0.00009	-0.10088	14.3678	0.00007

^aNote: The M1, M2, M3 and M4 are mixture (M₁), PA+TBP+TOA (V_{TBP}:V_{TOA}=8:2); mixture (M₂), PA+TBP+TOA (V_{TBP}:V_{TOA}=7:3); mixture (M₃), PA+TBP+TOA (V_{TBP}:V_{TOA}=6:4) and mixture (M₄), PA+TBP+TOA (V_{TBP}:V_{TOA}=5:5).

Table 4
The Coefficients r, s, m, n and R² about eq5 and eq6.^a

mix	r	s	R ²	m	n	R ²
M1	3.70089	-0.0305	0.02394	-0.03667	2.65877	0.01055
M2	4.13491	-0.03709	0.01032	-0.0388	2.61961	0.00924
M3	3.41888	-0.03549	0.00849	-0.03362	2.27201	0.00155
M4	0.14188	-0.00009	0.21961	-0.02463	1.02853	0.00042

^aNote: The M1, M2, M3 and M4 are mixture (M₁), PA+TBP+TOA (V_{TBP}:V_{TOA}=8:2); mixture (M₂), PA+TBP+TOA (V_{TBP}:V_{TOA}=7:3); mixture (M₃), PA+TBP+TOA (V_{TBP}:V_{TOA}=6:4) and mixture (M₄), PA+TBP+TOA (V_{TBP}:V_{TOA}=5:5).

Table 5

Viscosity η Calculated by eq 8 to eq 11 for Mixture M1,M2,M3,and M4 in (20~60)^oC as a Function of Mass Fraction of w of PA^a

w	t=20°C				t=30°C				t=40°C			
	M1	M2	M3	M4	M1	M2	M3	M4	M1	M2	M3	M4
1.00	8.8332	8.0756	6.3129	1.9345	6.0023	5.4132	4.4352	1.4957	4.0849	3.6307	3.1197	1.1575
3.00	14.6553	13.188	10.2631	2.4785	9.5743	8.6304	6.9721	1.8747	6.2836	5.6578	4.753	1.4221
5.00	24.3149	21.537	16.6852	3.4785	15.2721	13.7596	10.9601	2.3498	9.6659	8.8167	7.2414	1.7472
6.00	31.3192	27.5225	21.2744	3.1755	19.2882	17.3738	13.7417	2.6307	11.9883	11.0061	8.9383	1.9367
7.00	40.3413	35.1716	27.1259	3.5944	24.3606	21.9373	17.2292	2.9452	14.8687	13.7393	11.0327	2.1466
8.00	51.9624	44.9464	34.5868	4.6052	30.7668	27.6995	21.6018	3.2974	18.4412	17.1511	13.6179	2.3794
9.00	66.9311	57.4378	44.0997	5.2127	38.8578	34.9753	27.0841	3.6916	22.8720	21.4103	16.8089	2.6374
w	t=50°C				t=60°C							
	M1	M2	M3	M4	M1	M2	M3	M4				
1.00	2.7839	2.4365	2.1967	0.8966	1.8998	1.636	1.5484	0.6951				
3.00	4.1415	3.7153	3.2507	1.0817	2.7402	2.4436	2.23	0.8247				
5.00	6.1610	5.6652	4.8104	1.3049	3.9526	3.6499	3.2117	0.9786				
6.00	7.5144	6.9957	5.8518	1.4333	4.7471	4.4608	3.8543	1.0659				
7.00	9.1653	8.6386	7.1185	1.5743	5.7013	5.4518	4.6255	1.1611				
8.00	11.1787	10.6673	8.6595	1.7292	6.8472	6.6629	5.5511	1.2648				
9.00	13.6345	13.1725	10.5341	1.8992	8.2236	8.1432	6.6618	1.3777				

^aNote: The M1, M2, M3 and M4 are mixture (M₁), PA+TBP+TOA (V_{TBP}:V_{TOA}=8:2); mixture (M₂), PA+TBP+TOA (V_{TBP}:V_{TOA}=7:3); mixture (M₃), PA+TBP+TOA (V_{TBP}:V_{TOA}=6:4) and mixture (M₄), PA+TBP+TOA (V_{TBP}:V_{TOA}=5:5)

According to R^2 values from Tables 3 and 4, it can be concluded that the eqs (3) and (6) are more appropriate than eqs. (4) and (5) for the a and b coefficients representation. Therefore, in eq. (2),

$$M1:\ln(\eta/\text{mPa.s})=29.76021\exp\times(-0.00809(t^{\circ}\text{C}))w-0.03667\times(t^{\circ}\text{C})+2.65877 \quad (8)$$

$$M2:\ln(\eta/\text{mPa.s})=27.11323\exp\times(-0.00502(t^{\circ}\text{C}))w-0.0388\times(t^{\circ}\text{C})+2.61961 \quad (9)$$

$$M3:\ln(\eta/\text{mPa.s})=28.04485\exp\times(-0.00717(t^{\circ}\text{C}))w-0.03362\times(t^{\circ}\text{C})+2.27201 \quad (10)$$

$$M4:\ln(\eta/\text{mPa.s})=14.91473\exp\times(-0.00927(t^{\circ}\text{C}))w-0.02463\times(t^{\circ}\text{C})+1.02853 \quad (11)$$

The Table 1 lists the parameter $w \leq 9\%$, and the eq 8, eq 9, eq 10 and eq 11 can be used to calculate viscosity at different mixtures. By data analysis in Table 1, the viscosities of the four different mixtures are decreasing in the following order: $M1 > M2 > M3 > M4$. The viscosities of M1 are much larger than of M2, M3 and M4 under the same conditions. Densities varies in the order: $M1 < M2 < M3 < M4$ and are much larger for M4 when comparing with those of M1, M2 and M3 under the same conditions.

CONCLUSIONS

In this paper, the experimental viscosities of PA+TBP+TOA mixtures are consistent with those calculated at temperature range from $(20\sim 60)^{\circ}\text{C}$ and the mass fraction of H_3PO_4 in the range of (0 to 9) %. And the viscosity of the mixture is minimum under TBP:TOA=5:5 by the viscosity data analysis.

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