Temperature-Dependent Density and Viscosity of the Ionic Liquids 1-Alkyl-3-methylimidazolium Iodides: Experiment and Molecular Dynamics Simulation

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The density and viscosity of synthesized 1-alkyl-3-methylimidazolium iodide ([$C_n$mim]I, $n = 4, 6, 8$) were measured in the wide range of temperature of (298 to 393) K. Using a vacuum line, measurements of the viscosity were made under a water-vapor free atmosphere. The viscosity decreases sharply with temperature and increases as the alkyl chain length increases. The molecular dynamics simulation was performed for the densities of these ionic liquids to remedy the lack of literature experimental data. The results are quite in agreement with the experiments, with a maximum deviation of 3.00 % due to [C_8mim]I at 358 K. The viscosities fit best in the modified Arrhenius, Vogel−Fulcher−Tammann (VFT), and Litovitz equations. The viscosities also fit in the simple linear equation we proposed recently with accuracies comparable with Litovitz and VFT.

Introduction

In the past few decades, inquiry for the properties of selected ionic liquids (ILs) has been accelerated. This is because the properties of not orderly packed lattice of bulky cation could be combined with the diverse available anions to provide many interesting physicochemical properties for ILs. These ILs exhibit highly desirable physical properties, wide liquid range, high thermal stability, good ionic conductivity, high electrochemical stability, low vapor pressure, and favorable solubility behavior.[1−4]

There is much interest in the transport properties of ILs due to their growing industrial applications. Transport properties basically represent the fluid fundamental feature with good accuracy. These properties not only determine the engineering aspects of the fluids but also provide a good insight into different fluid types of various classes of fluids on the molecular basis. They are essential for most industrial applications, and intensive research has been done for their measurement.[5−7] As a transport property, liquid viscosity has a great influence on the rate of mass transport and can be measured with high accuracy.

Despite numerous investigations, information on the thermophysical properties of ILs and their temperature dependence is incomplete. In some cases, available viscosities are single measurements or in a limited range of temperature. Besides, the water content and impurities of the ILs are known to influence the density (1 to 2) % and viscosity (up to orders of magnitude).[8]

The temperature dependence of viscosity is a phenomenon by which the viscosity (η) of a liquid decreases or equivalently its fluidity (η^{-1}) increases as the temperature increases. The most commonly used equations that fit the temperature-dependent viscosity of ILs accurately are the Vogel−Fulcher−Tammann (VFT) and Litovitz equations. These empirical models which are extensions of the Arrhenius equation have been used widely to model various ILs.[8−15]

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Halides, among other ILs, have been synthesized, and their thermophysical properties have been investigated. Most of the studies on the 1-alkyl-3-methylimidazolium halides are focused on chlorides. Seddon et al. have measured the viscosity of [$C_n$mim]Cl ($n = 2, 4, 6, 8$) over the temperature range of $T =$ (283.15 to 363.15) K by a Cone and Plate viscometer and have fitted the results in the VFT equation.[15] Values of viscosity of [C_6mim]Cl and [C_8mim]Cl from $T =$ (298.15 to 343.15) K have been also measured using an automatic viscometer by Gomez et al.[16] The room temperature (298.2 K) viscosity of [C_4mim]Br has been measured using an Ubbelohde viscometer by Kim et al.[17] They have also measured the viscosity of [C_4mim]I from $T =$ (298.15 to 323.15) K.[18]

In this work, three ILs, 1-alkyl-3-methyl-imidazolium iodide, [$C_n$mim]I with $n = 4, 6,$ and 8 representing butyl, hexyl, and octyl alkyl groups, respectively, are synthesized, and their densities and viscosities are measured. Attention is paid to conduct an accurate viscosity measurement by using a vacuum line to remove dissolved water and atmospheric gases from ILs as well as performing measurements under a water-vapor free atmosphere. The temperature dependence of the viscosity of these ILs in the range $T =$ (298.15 to 393.15) K is studied by a new simple relation we developed recently and is compared with the Litovitz equation and VFT, which are known to apply to the ILs quite accurately. The density of these ILs was also simulated by classical molecular dynamics over the temperature range (298 to 358) K. This is to remedy the lack of experimental density data in literature. To our knowledge this is the first systematic measurement of the temperature-dependent viscosity as well as measurement and simulation of density of imidazolium-based iodide ILs.

Experimental Section

ILs were synthesized and purified according to literature,[19] the details of which were given elsewhere.[20] The ILs produced were initially dried in vacuum oven at 343.15 K for 24 h. The ^{1}H NMR spectra indicated that there was no presence of starting materials or solvent used in the purification processes.

10.1021/je901092b
Published on Web 06/28/2010

A 300 Cannon-Fenske capillary viscometer was used. This viscometer provides a practical vacuum line set up to furnish a water-vapor free atmosphere for viscosity measurement. The dried IL sample was put in the viscometer under vacuum (0.133 Pa) for about 2 h, until effervescence is stopped. At low temperatures, extensive effervescence was seized over a relatively long time, while at the high temperatures the effervescence process seized quickly. Beside the fact that the sample was already dried in vacuum oven, at this point it was expected that most dissolved water and gases were removed. During viscosity measurement, however, the vacuum was removed, and dried air was let in. The water content after first drying trial by vacuum oven and after evacuation by vacuum line was measured by Karl Fischer (870 KF Titrino Plus, Metrohm).

An oil bath with a glass thermometer to control the temperature within ± 0.5 K was used. The system was allowed to equilibrate for at least 30 min. The viscosity of samples was measured in the temperature range of T = (298.15 to 393.15) K, using a digital stopwatch to record time within ± 0.01 s. The viscometer was calibrated by using diisodecyl phthalate (Merck, for synthesis grad, lot no. S5148332 batch 914 with a nominal purity of 99 %). The calibration was based on the published data,[21] at 298.15 K and 87.62 mPa·s. We estimated the combined expanded uncertainty (with $k = 2$) for the viscosity values to be less than 4 %.

To measure the density, a standard 5.00 mL pycnometer (Marienfeld) was calibrated by using deionized water at 298 K (0.9970 g·cm^{-3}).[22] The pycnometer was filled with dried and degassed IL sample and thermostatted as in the viscosity measurement. The combined expanded uncertainty (with k = 2) of the measured densities is estimated to be 0.12 %.

The absolute value of viscosity was then calculated by using the measured time (t) and the adjusted density (next section) of the IL sample (ρ_{IL}) using eq 1.

$$\eta_{IL} = \eta_{ref}(\rho \cdot t)_{IL}/(\rho \cdot t)_{ref} \quad (1)$$

Results and Discussion

Sample Preparation and Measurements. No evidence of trace of starting material was observed by the ^{1}H NMR spectra of the synthesized ILs. Despite all precautions, not all of the dissolved water can be removed. Trace amounts of water reduces slightly the density of imidazolium-based IL but reduces its viscosity dramatically.[8,23]

Experiments involving ILs are normally performed under inert gas and a moisture-free atmosphere using a glovebox. However, dissolved gas among other factors has been known to affect the measured properties.[19,20,23] The viscosity of an IL is affected highly by moisture.[23−25] The effects of these factors on the accuracy of the temperature-dependent surface tension have been reported.[20] In this work, the samples were initially dried in a vacuum oven and evacuated to remove dissolved atmospheric gases and remaining traces of moisture as much as possible, and then the measurement was made under dried atmosphere using the vacuum line. The values of water content measured by Karl Fischer before (and after) evacuation by vacuum line are as following: [C$_4$mim]I, 3471 (3370) ppm; [C$_6$mim]I, 3200 (3147) ppm; [C$_8$mim]I, 2758 (2165) ppm. It can be seen that the [C$_8$mim]I with the longer alkyl chain length contains less water content than those with short alkyl chain. Also [C$_8$mim]I lost more water content upon evacuation.

Temperature Dependence of Density. The density for iodide ILs was measured over the wide temperature range and explored exclusively here. Values of the measured density are shown in Table 1. The data were correlated by fitting to a quadratic polynomial:

$$[C_4mim]I \qquad \rho/g\cdot cm^{-3} = -1.061\cdot10^{-6}\,T^2/K^2 - 6.045\cdot10^{-5}\,T/K + 1.548 \quad (2a)$$

Table 1. Density Values of Synthesized ILs Measured Experimentally, Determined by Molecular Dynamics Simulation, and Those Given in Literature

T / K	ρ / g·cm^{-3}	$(\rho - \rho_{cal})100/\rho_{cal}$	ρ_{lit} / g·cm^{-3}	ρ_{simu} (% dev.)[a] / g·cm^{-3}
		[C$_4$mim]I		
298.15			1.46[17] (1.44)[19]	1.438
299.15	1.434	−0.041		
303.15			1.45[17]	
304.15	1.432	0.064		
308.15	1.430	0.123	1.45[17]	
313.15	1.426	0.095	1.44[17]	
318.15	1.422	0.071	1.44[17]	
323.15	1.418	0.050	1.44[17]	1.422 (0.282)
328.15	1.414	0.033		
333.15	1.406	−0.264		
338.15	1.400	−0.416		1.405 (0.357)
343.15	1.398	−0.281		
348.15	1.396	−0.140		1.392 (−0.287)
353.15	1.394	0.004		
358.15	1.392	0.154		1.370 (−1.58)
363.15	1.388	0.164		
368.15	1.386	0.322		
373.15	1.382	0.340		
378.15	1.376	0.217		
383.15	1.370	0.096		
388.15	1.360	−0.314		
		[C$_6$mim]I		
298.15				1.349
305.15	1.326	0.023		
309.15	1.324	0.000		
314.15	1.322	0.005		
318.15	1.320	−0.020		
323.15	1.318	−0.015		1.329 (0.835)
328.15	1.316	−0.010		
333.15	1.314	−0.006		
338.15	1.312	−0.003		1.323 (0.838)
343.15	1.310	0.000		
348.15	1.308	0.002		1.312 (0.306)
353.15	1.306	0.003		
358.15				1.298 (−0.460)
359.15	1.304	0.033		
363.15	1.302	0.003		
373.15	1.298	0.000		
378.15	1.296	−0.002		
383.15	1.294	−0.005		
388.15	1.292	−0.009		
		[C$_8$mim]I		
298.15				1.296
300.15	1.258	0.052		
303.15	1.254	−0.067		
308.15	1.250	−0.055		
313.15	1.246	−0.045		
319.15	1.242	0.029		
323.15	1.240	0.130		1.266 (2.10)
328.15	1.234	−0.029		
333.15	1.230	−0.029		
338.15	1.228	0.133		1.257 (2.36)
343.15	1.224	0.129		
348.15	1.218	−0.041		1.253 (2.87)
353.15	1.212	−0.216		
358.15	1.210	−0.063		1.246 (3.00)
363.15	1.206	−0.078		
368.15	1.204	0.071		
373.15	1.200	0.052		
378.15	1.196	0.030		

[a] % dev. = $100(\rho_{simu} - \rho_{exp})/\rho_{exp}$.

$$[C_6mim]I \qquad \rho/g\cdot cm^{-3} = 1.814\cdot10^{-7}\,T^2/K^2 - 5.303\cdot10^{-4}\,T/K + 1.471 \quad (2b)$$

$$[C_8mim]I \qquad \rho/g\cdot cm^{-3} = 5.984\cdot10^{-7}\,T^2/K^2 - 1.197\cdot10^{-3}\,T/K + 1.563 \quad (2c)$$

The experimental densities and the deviations of eqs 2a to 2c from the experiments are shown in Table 1. Densities smoothly decrease with temperature and in general are inversely proportional to the alkyl chain length. It is worth mentioning that the densities of the iodides are larger than those of chlorides.

To remedy the lack of experimental data, classical molecular dynamics simulations were performed on the densities of [C_4mim]I, [C_6mim]I, and [C_8mim]I in the temperature range of (298 to 358) K. In each case, an ensemble comprising 512 ion pairs was simulated. The complete results on other properties of the ILs including the bulk and the interface will be published elsewhere. Supporting Information gives some details concerning the simulation. The results are shown in Table 1 and compared with our experimental results and the literature data. Deviations from our experiment measurements are also shown. The agreement is quite good, and the maximum deviation is 3.00 % due to [C_8mim]I at 358 K. The deviation of the simulated density of [C_4mim]I with that reported in literature are −1.51 %[17] and −0.280 %.[19]

Temperature Dependence of Viscosity. Viscosities of ILs were measured also in a wide temperature range. The viscosity values are shown in Table 2 and plotted in Figure 1. As with other ILs, the viscosity of the iodides increases rather rapidly at low temperatures and decreases to a small value at high temperatures asymptotically. The longer the alkyl chain length is, the higher the viscosity. Strong electrostatic interaction between the anion and the imidazolium ring cation plays a major role, as is determined by the anion type and the alkyl chain length. The viscosity of iodides is much lower than those reported for corresponding chlorides.[15,16]

To our knowledge, only two reported viscosity measurements for [C_4mim]I are available, which are included in the Figure 1. The first set,[18] involves viscosity values in the limited range T = (298.15 to 318.15) K. At low temperatures these are lower than our measurements but at high temperatures likely approaching each other. The single measurement by Huddlestone et al., at 298 K for [C_4mim]I,[19] is much higher than the result of this work (see Figure 1). The disagreements commonly observed between measured viscosities of ILs are also seen here. Sources of deviation from true values can be the difference in the level of water content (which basically lowers the viscosity), impurities due to the starting materials in the synthesis, and the level of accuracy of the method of measurement. In this work care has been taken particularly in drying IL samples as well as performing viscosity measurement under a water-vapor free atmosphere.

Table 2. Values of Viscosities of Synthesized ILs Measured Experimentally

[C_4mim]I		[C_6mim]I		[C_8mim]I	
T/K	η/mPa·s	T/K	η/mPa·s	T/K	η/mPa·s
299.15	565.9	305.15	771.9	300.15	1245
304.15	421.9	309.15	572.0	303.15	935.2
308.15	321.2	314.15	412.7	308.15	692.7
313.15	217.3	318.15	318.6	313.15	499.8
318.15	165.7	323.15	234.1	319.15	385.4
323.15	127.9	328.15	181.2	323.15	282.8
328.15	100.4	333.15	139.6	328.15	218.4
333.15	77.69	338.15	110.7	333.15	166.2
338.15	62.77	343.15	83.92	338.15	132.0
343.15	50.93	348.15	70.39	343.15	99.90
348.15	42.59	353.15	57.79	348.15	84.85
353.15	34.81	359.15	46.20	353.15	69.75
358.15	28.98	363.15	40.25	358.15	55.68
363.15	24.89	373.15	29.02	363.15	47.28
368.15	21.45	378.15	25.37	368.15	41.19
373.15	19.47	383.15	22.11	373.15	33.91
378.15	17.25	388.15	19.35	378.15	29.95
383.15	14.51				
388.15	12.50				

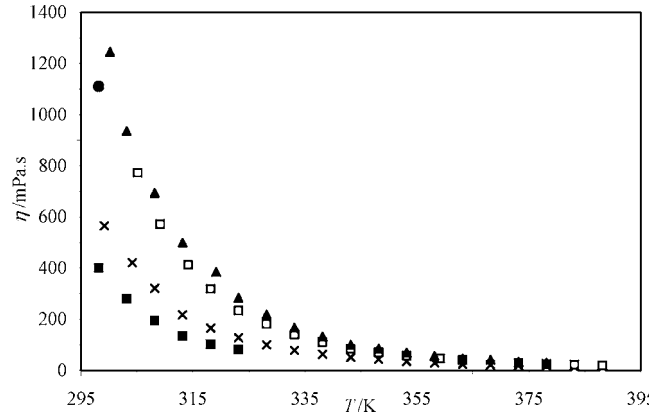

Figure 1. Measured viscosities versus temperature for the synthesized ILs. Experimental viscosities of [C_4mim]I in the range T = (298 to 318) K[18] and at 298 K[19] are also included for comparison; ×, [C_4mim]I; □, [C_6mim]I; ▲, [C_8mim]I; ■, [C_4mim]I;[18] ●, [C_4mim]I.[19]

Although the data reported by Huddlestone on [C_4mim]I have not been provided in detail, considering other reported data on viscosities,[19] including the [C_6mim]Cl (= 716 mPa·s) and [C_8mim]Cl (= 337 mPa·s) at 298 K, may shade on the level of goodness of their measurement. More recent data reported at the same temperature by experts in this field (for example Gomez et al.,[16]) on the viscosity of [C_6mim]Cl and [C_8mim]Cl is about 18089 mPa·s and 20883 mPa·s, respectively. On the other hand, it has been known that viscosity in general increases with increasing the alkyl chain length of the imidazolium-based ILs, while the reverse trend has been reported by Huddlestone et al.[19]

Correlations. Earlier, it has been known that the viscosity of the ILs exhibits non-Arrhenius temperature dependence even over a short-range of temperatures.[26,27] Although VFT equation has been known to fit the viscosity of most ILs quite well,[9,11−13,15,23] it does not fit dried [C_4mim]PF_6 satisfactorily.[8] A reliable comparison with the available viscosities can be obscured because of different levels of accuracies due to small errors in the measurement of temperature particularly at low temperatures as well as the level of water content.

The viscosity values of the iodide ILs obtained in this work can be fitted in the Litovitz equation,

$$\eta = A\exp(B/RT^3) \quad (3)$$

and the VFT equation,

$$\eta = A'\exp(k/(T - T_0)) \quad (4)$$

Fitting parameters, the correlation coefficient squared (R^2), and % AAD of the fit to eqs 3 and 4 are listed in Table 3.

Table 3. Fitted Parameters of the Litovitz and VFT Equations[a]

ln(A/mPa·s)	[C_4mim]I	[C_6mim]I	[C_8mim]I
Litovitz Equation: $\eta = A\exp(B/RT^3)$			
ln(A/mPa·s)	-0.686 ± 0.035	-0.544 ± 0.027	-0.344 ± 0.038
$(B/R)\cdot 10^{-8}$/K^3	1.876 ± 0.013	2.032 ± 0.010	2.017 ± 0.014
R^2	0.9992	0.9996	0.9993
% AAD	2.9	1.9	2.2
VFT Equation: $\eta = A'\exp(k/(T - T_0))$			
ln(A'/mPa·s)	-2.194 ± 0.214	-2.192 ± 0.126	-3.443 ± 0.522
k/K	946 ± 63	1023 ± 38	1500 ± 183
T_0/K	189 ± 5	190 ± 3	158 ± 11
R^2	0.9996	0.9999	0.9993
% AAD	2.0	0.85	2.2
Recent Work's Equation: $(1/\eta)^{0.3} = a + bT$			
a	-0.929 ± 0.008	-0.887 ± 0.004	-0.834 ± 0.010
b	0.00355 ± 0.00002	0.00330 ± 0.00001	0.00311 ± 0.00003
R^2	0.9993	0.9998	0.9986
% AAD	2.4	1.5	4.3

[a] Those of eq 5 are included.

$$\%\ \mathrm{AAD} = \frac{1}{N}\sum_{i}^{N}\left|\frac{(\eta_{\mathrm{cal}} - \eta_{\mathrm{expt}})\cdot 100}{\eta_{\mathrm{cal}}}\right|$$

The correlation coefficients and the deviations of experimental data (% AAD) from eqs 3 and 4 indicate that both Litovitz and VFT equations can describe the temperature-dependent viscosity of the iodide ILs measured in this study quite accurately. However, the VFT equation fits the three ILs with lower deviations than Litovitz. The higher accuracy of the VFT than the Litovitz is due to the higher number of parameters of the former.

Recently, according to the fact that fluidity (e.g., $1/\eta$) contrary to the viscosity is a smooth function of temperature, we have shown that the temperature dependence of the viscosity can be described by the following simple linear equation:[28]

$$\left(\frac{1}{\eta}\right)^{\phi} = a + bT \qquad (5)$$

where a and b are constants characteristics of the IL and ϕ is a characteristic exponent. It has been shown that the eq 5 fits numerous ILs where the exponent ϕ (= 0.300 with standard deviation 0.004) is universally applied.[28] These ILs are including imidazolium-, pyrrolidinium-, quaternary ammonium-, and nicotinium-based ILs with a variety of anions (such as halides, PF_6^-, BF_4^-, $(CN)_2N^-$, $(CF_3SO_2)_2N^-$, CH_3COO^-, $CF_3SO_3^-$, $CH_3SO_4^-$, $C_2H_5SO_4^-$). The accuracies in general are comparable with accuracies of the eqs 3 and 4. With the universal value of ϕ for the ILs, therefore, this two-parameter equation also is used for fitting the experimental viscosities of iodide ILs measured in the present work.

Deviation plots are shown in Figure 2 for eqs 3, 4, and 5. From the fitting parameters shown in Table 3 and from the deviation plots, it can be seen that the quality of the fits by eq 5 for [C_4mim]I and [C_6mim]I is better than Litovitz and close to VFT, which fits all of the ILs with high accuracy. Deviation of [C_8mim]I viscosity from the fitted one by eq 5 is rather small though is slightly higher than both Litovitz and VFT equations.

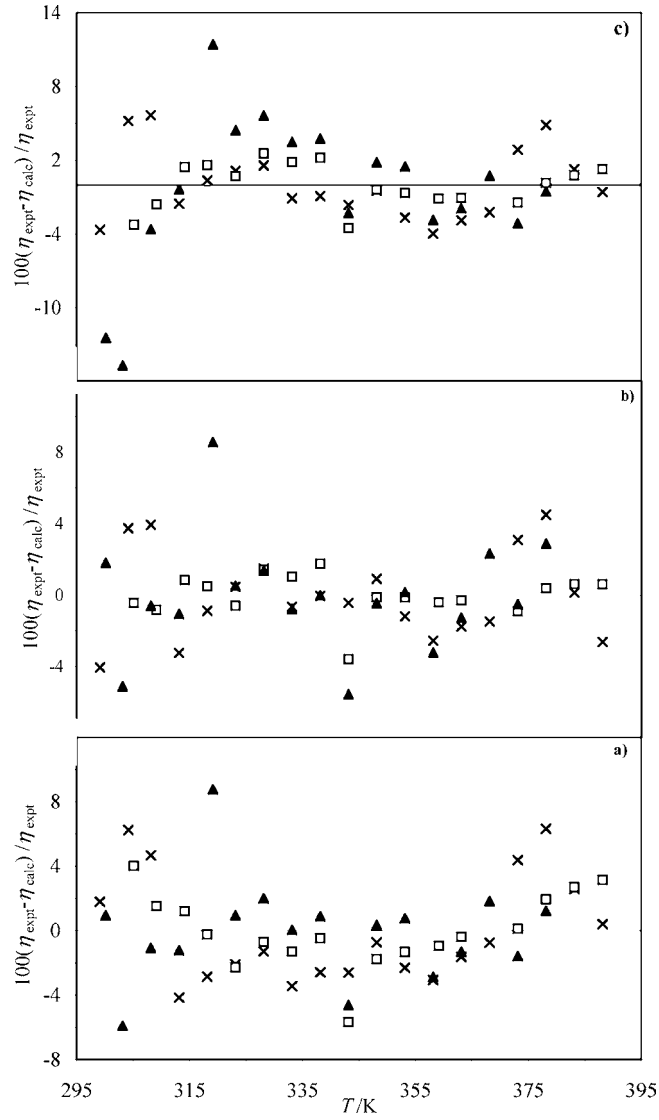

Figure 2. Residuals for the fit of the experimental viscosities for ×, [C_4mim]I; □, [C_6mim]I; ▲, [C_8mim]I to (a) Litovitz equation, (b) VFT equation, and (c) eq 5 as a function of temperature.

Conclusions

ILs 1-alkyl-3-methylimidazolium iodide ([$C_n$mim]I, n = 4, 6, 8) have been synthesized, and their viscosities and densities have been measured experimentally in the range of T = (298 to 393) K. The IL with a longer chain length has a higher viscosity and a lower density. The densities are consistent with the results of the classical molecular dynamics simulation with a maximum deviation of 3.00 % due to [C_8mim]I at 358 K. The viscosity of iodides is much lower than the corresponding chlorides. Both Litovitz and VFT equations fit the viscosities of these ILs with high accuracies. The simple linear equation we proposed fits the viscosities with accuracy better than Litovitz, though VFT is notable.

Acknowledgment

Help for data analysis by Dr. Neamatollahi, Dr. Kharati, and Miss Sajadnia of the Department of Statistics is greatly acknowledged.

Supporting Information Available:

The basic information on the simulation of densities of [C_4mim]I, [C_6mim]I, and [C_8mim]I by molecular dynamics is given. The complete results on the bulk and interfacial properties of these ILs will be published elsewhere. This material is available free of charge via the Internet at http://pubs.acs.org.

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Received for review December 29, 2009. Accepted June 14, 2010. The authors are indebted to the research council of the Shiraz University for financial support. Financial support by the EOR Research Center, School of Chemical & Petroleum Engineering is also acknowledged.

JE901092B