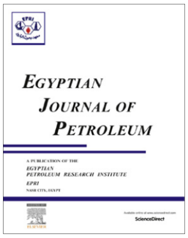
Egyptian Journal of Petroleum (2015) **24**, 103–111

HOSTED BY

Egyptian Petroleum Research Institute

Egyptian Journal of Petroleum

www.elsevier.com/locate/egyjp
www.sciencedirect.com



EGYPTIAN
JOURNAL OF
PETROLEUM

FULL LENGTH ARTICLE

Highly effective ionic liquids for biodiesel production from waste vegetable oils

Fathy A. Yassin [a], **Fathy Y. El Kady** [b], **Hoda S. Ahmed** [b], **Latifa K. Mohamed** [b], **Seham A. Shaban** [b,*], **Ahmed K. Elfadaly** [b]

[a] *Faculty of Science, Zagazig University, Zagazig, Egypt*
[b] *Refining Division, Egyptian Petroleum Research Institute, Nasr City, Cairo 11727, Egypt*

Received 16 May 2014; accepted 13 November 2014
Available online 16 April 2015

KEYWORDS

Biodiesel;
Catalyst;
Energy sources;
Ionic liquid;
Vegetable oil

Abstract As conventional energy sources deplete, the need for developing alternative energy resources which are environment friendly becomes more imperative. Vegetable oils are attracting increased interest in this purpose. The methanolysis of vegetable oil to produce a fatty acid methyl ester (FAME, i.e., biodiesel fuel) was catalyzed by commercial ionic liquid and its chloride modification. The imidazolium chloride ionic liquid was frequently chosen for the synthesis of biodiesel. The dual-functionalized' ionic liquid is prepared by a direct combination reaction between imidazolium cation and various metal chlorides such as CoCl2, CuCl2, NiCl2, FeCl3 and AlCl3. Imidazolium tetrachloroferrate was proved to be a selective catalyst for the methanolysis reaction at a yield of 97% when used at 1:10, catalyst: oil ratio for 8 h at 55 °C. Operational simplicity, reusability of the used catalyst for 8 times at least, high yields and no saponification are the key features of this methodology. The dynamic viscosity and density of the upgraded vegetable oil decreased from 32.1 cP and 0.9227 g/cm^3 to 10.2 cP and 0.9044 g/cm^3 respectively, compared to those of the base vegetable oil. The objective of this study was the synthesis and characterization of biodiesel using commercial ionic liquid and its chloride modification. The ionic liquid catalysts were characterized using FTIR, Raman spectroscopy, DSC, TG and UV.

1. Introduction

Energy consumption augment is inevitable for human existence. There are various reasons for the search of an alternative fuel, that is technically feasible, environmentally acceptable, economically competitive, and readily available and basically as an alternative to fossil fuels. The most foremost reason to pursue the search for alternative and health safe fuels is the increasing demand for fossil fuels in all of the human life activities such as transportation, power generation, industrial processes, and residential consumption [1]. World energy consumption doubled between 1971 and 2001 and the world energy demand will increase 53% by the year 2030. The second reason is that fossil-fuel resources are non-renewable, and they will be exhausted within 40–60 years if

---

[*] Corresponding author.
E-mail address: sehamshaban@yahoo.com (S.A. Shaban).

Peer review under responsibility of Egyptian Petroleum Research Institute.

http://dx.doi.org/10.1016/j.ejpe.2015.02.011

the consumption pace remains constant [2]. Finally, the price instability of fossil fuel crude oil is considered as a serious threat to countries with limited financial and economic resources [3]. Several alternatives of energy sources such as wind, solar, hydro, nuclear, biofuel [4–7], and biodiesel are already used and elevating progress is still in concern. Exhausted vegetable oils are now being considered as a more suitable renewable and potentially inexhaustible source of energy that is close to diesel fuel [8] due to an advantageous economical cost without being a burden on its nutritional aspects. Vegetable oil trans-esterification producing biodiesel fuel has been efficiently carried out in the presence of catalysts as ionic liquids due to their increased Lewis acidity and hence increases their yield of biodiesel [9–12].

2. Experimental

2.1. Materials

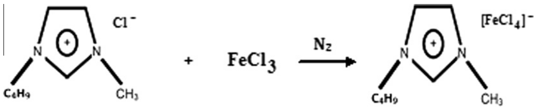
Waste vegetable oils collected from fried food companies. Methanol, 1-butyl-3-methyl imidazolium chloride [BMIM][Cl], Iron (III)chloride, Aluminum (III) Chloride, Cobalt (II) chloride, Cupric (II) chloride and Nickel (II) chloride were all of Sigma Aldrich make.

2.2. Methods and procedures

2.2.1. Preparation of modified ionic liquids

The ionic liquid metal chlorides designated; [bmim][FeCl$_4$], [bmim][AlCl$_4$], [bmim][CoCl$_4$], [bmim][CuCl$_4$], and [bmim][NiCl$_4$] were prepared by a direct combination reaction between imidazolium cation and the metal chlorides MCln [13]. 1-Butyl-3-Methyl Imidazolium chloride [BMIM][Cl] and xMCln (where x represents the molar ratio) were stirred under nitrogen atmosphere at 90 °C for 45 min in a three-necked round bottom flask equipped with a condenser.

The combination reaction is exemplified as follows:



(1)

2.2.2. Characterization of the ionic liquid and its metal chlorides modification

The chemical structure of original ionic liquid and its metal chlorides modification were characterized by FT-IR spectroscopy, Raman spectra and UV–Vis spectrometer. FT-IR spectra for the ionic liquid and its metal chlorides product were recorded using a ATI Mattson Genesis FT-IR spectrometer through the use of a KBr disc. UV–Vis absorption spectra were recorded using a UV/Vis/NIR spectrophotometer (Jasco-V-570), while Raman spectra were obtained using a Raman Spectroscopy (senterra)-Braker, Germany at room temperature in small glass tubes by using laser light of 785 nm. The thermal stability of the catalyst was determined by Thermogravimetric analysis (TGA) and Differential scanning calorimetry (DSC) analysis by using a TGA-50 shimadzu instrument, in the range of 40–600 °C.

2.2.3. Trans-esterification reaction

In a three neck flask equipped with a reflux condenser, exhausted vegetable oil and methanol in a molar ratio of 1:9 was mixed before 5 g of metal chlorides liquid is added. The mixture is then stirred for 8 h at 55 °C. After completion of the reaction, the product obtained was centrifuged to separate the formed phases, the upper layer contains the biodiesel product (methyl esters) while the middle layer contains the by-product glycerol and the lower aqueous layers contain the ionic liquid and the excess methanol. The upper layer (biodiesel) was separated and washed by warm water to remove any residual glycerol or methanol, but the ionic liquid was recycled through washing with n-hexane and dried at 70 °C for 4 h. The yield of biodiesel was calculated according to Eq. (2) [14,15] and GC analysis. Density and viscosity were also measured for the collected biodiesel.

$$\text{Yield of FAME} = \frac{\text{Actual weight of biodiesel(g)}}{\text{Theoretical weight of biodiesel(g)}} \times 100 \tag{2}$$

3. Results and discussion

3.1. Ionic liquid characterization

3.1.1. Fourier transform infrared (FT-IR) spectroscopy

Fig. 1 illustrates the IR spectra of imidazolium chloride ionic liquid [bmim][Cl] and its metals chloride modification [bmim][MCl$_4$] in the range from 4000 to 400 cm^{-1}. The peaks in the range between 3000 and 2800 cm^{-1} are characterizing the spectra of the –C–H stretching band of the –CH2 and –CH3 stretching vibrations of the alkyl groups located at the nitrogen atoms of the imidazolium ring. The bands between 3200 and 3100 cm^{-1} are assigned to all –C–H stretching vibrations of the imidazolium cation [16,17] while, the skeleton vibrations of the imidazole ring are observed at 1650–1500 cm^{-1} and 1450 cm^{-1} [18], while the peak around 3600–3200 cm^{-1} is indicative for the –OH vibration [19]. The transmission bands around 3200–3100, 3000–2800, 1650–1500, 1450 and 1200–1100 cm^{-1} are designated for the original ionic liquid and its metal chloride modification [20]. The characteristic bands of metals chloride modification ionic liquid show the same behavior as those of the original ionic liquid with a slight shift in the position of the band and/or its intensity. This may be due to the formation of a complex compound of the metal chloride and the ionic liquid. This complex may be considered as the main cause of the catalytic activity of the metals chloride imidazolium as will be observed from the activity results of the sole imidazolium and its metal form compounds.

3.1.2. Raman spectra

Fig. 2 shows Raman spectra of the original ionic liquid [bmim][Cl] and its metals chloride modification [bmim][MCl$_4$]

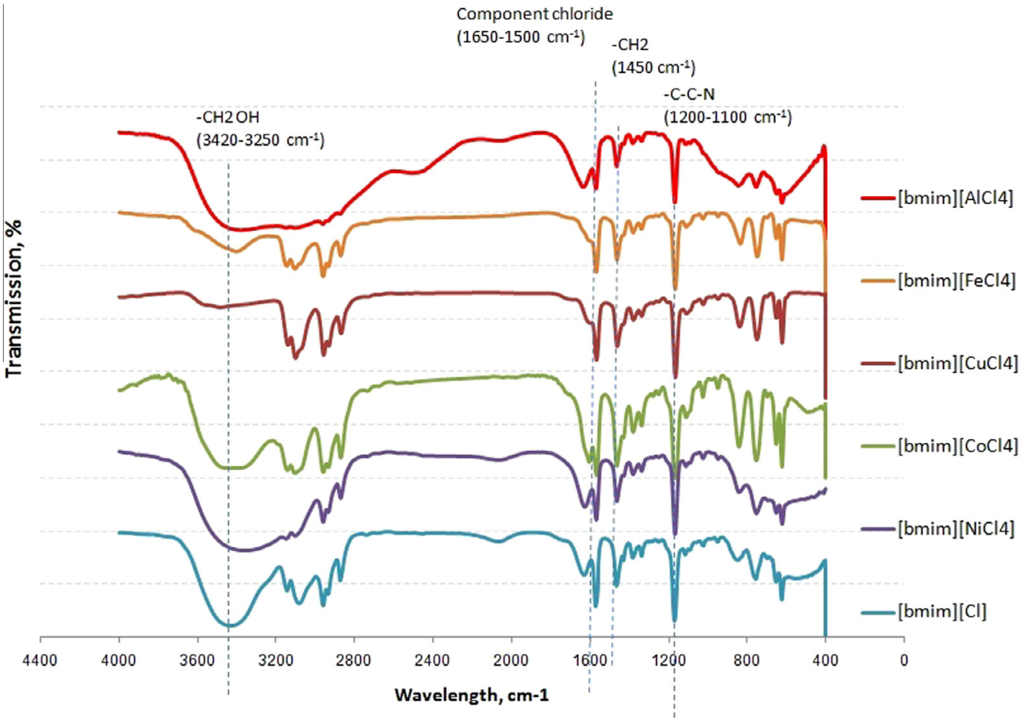


Figure 1 FTIR of imidazolium ionic liquid [bmim][Cl] and its Ni, Co, Cu, Fe and Al chlorides modification.

are also presented in Fig. 2. The two main absorption bands of the original ionic liquid [bmim]Cl shown at 85.86 and 205.77 cm^{-1} are assigned to the vibrations of the [bmim] + cation [21] which are observed with various metals with a small shift in position and a difference in band area. The Raman spectrum of [bmim][FeCl$_4$]$^-$ and [bmim][CuCl$_4$]$^{--}$ exhibits strong absorption bands at 315.43 and 291.42 cm^{-1} which are assigned to the symmetric Fe–Cl and Cu–Cl stretching vibration [22]. The Raman spectrum of [bmim][AlCl$_4$]$^-$ shows, however, a broad absorption band at 333.65 cm^{-1}, as assigned to the symmetric Al–Cl stretching vibration of [AlCl$_4$]$^-$ anion [23]. [bmim][CoCl$_4$]$^{--}$ is distinguished by two broad absorption bands at 256.61 and 325.23 cm^{-1} assigned to the symmetric Co–Cl stretching vibration of [CoCl$_4$]$^{--}$ anion [23]. The Raman spectrum of [bmim][NiCl$_4$]$^{--}$ exhibiting weak bands at 250 cm^{-1} is assigned to the symmetric Ni–Cl stretching vibration of [NiCl$_4$]$^{--}$ anion. Thus, the Raman spectroscopy results indicate the formation of the complexes [bmim][NiCl$_4$]$^{--}$, [bmim][CoCl$_4$]$^{--}$, [bmim][CuCl$_4$]$^{--}$, [bmim][AlCl$_4$]$^-$ and [bmim][FeCl$_4$]$^-$ which will be supported by the UV–Visible spectroscopy results.

3.1.3. *UV–Vis absorption spectra*

Fig. 3 exhibits the UV–Vis spectra of the original ionic liquid [bmim][Cl] and its metals chloride modification [bmim][MCl$_4$]. The original ionic liquid [bmim]Cl is distinguished by one main absorption band at 290 nm while the spectra of the metal chloride complexes differ from each other's and show absorption bands at 210 and 228 nm for [bmim][AlCl$_4$]$^-$, 342 nm for [bmim][FeCl$_4$]$^-$, 432 nm for [bmim][NiCl$_4$]$^{--}$, 528 nm for [bmim][CuCl$_4$]$^{--}$ and 682 nm for [bmim][CoCl$_4$]$^{--}$ as reported earlier [24]. Thus, spectroscopic results prove the respective Complexes formation (see Table 1).

3.1.4. *Thermal analysis: Differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA)*

The differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) behavior of the original ionic liquid [bmim][Cl] and its metals chloride modification [bmim][MCl$_4$] are represented in Figs. 4 and 5. The original ionic liquid [bmim][Cl] exhibits several endothermic peaks as the temperature is increased. The first peak located at 42.5 °C is related to the water loss at 7.5% while, the increase of temperature was accompanied by the successive appearance of other endothermic peaks, their maximum are located at 282.2 and 424.2 °C with decomposition beginning at 230° and 350° at weight loss of 77.4% and 15.1% of sample original weight respectively. On the other hand, the DSC and TGA behaviors of metals chloride forms [bmim][MCl$_4$] exhibited the same peaks of the original ionic liquid but with a slight shift, the 1st peak is a relatively located at about 34.5, 38.5, 34.5, 40.0 and 41.5 °C indicating the loss of water of crystallization H_2O for [bmim]Cl$^-$, [bmim][NiCl$_4$]$^{--}$, [bmim][CoCl$_4$]$^{--}$, [bmim][CuCl$_4$]$^{--}$, [bmim][FeCl$_4$]$^-$ and [bmim][AlCl$_4$]$^-$, respectively [25].

The addition endothermic peaks, their maximum are located at 370.4 and 423.5 °C for [bmim][NiCl$_4$]$^{--}$, 450.65 °C for [bmim][CoCl$_4$]$^{--}$, 219.7, 249.8, 373.7 and 429.4 °C for [bmim][CuCl$_4$]$^{--}$, 278.4 and 395.8 °C for [bmim][FeCl$_4$]$^-$ and 280.3 and 419.1 °C for [bmim][AlCl$_4$]$^-$, respectively. These peaks are related to the successive decomposition of the produced type of ionic liquid metals chloride modification, which

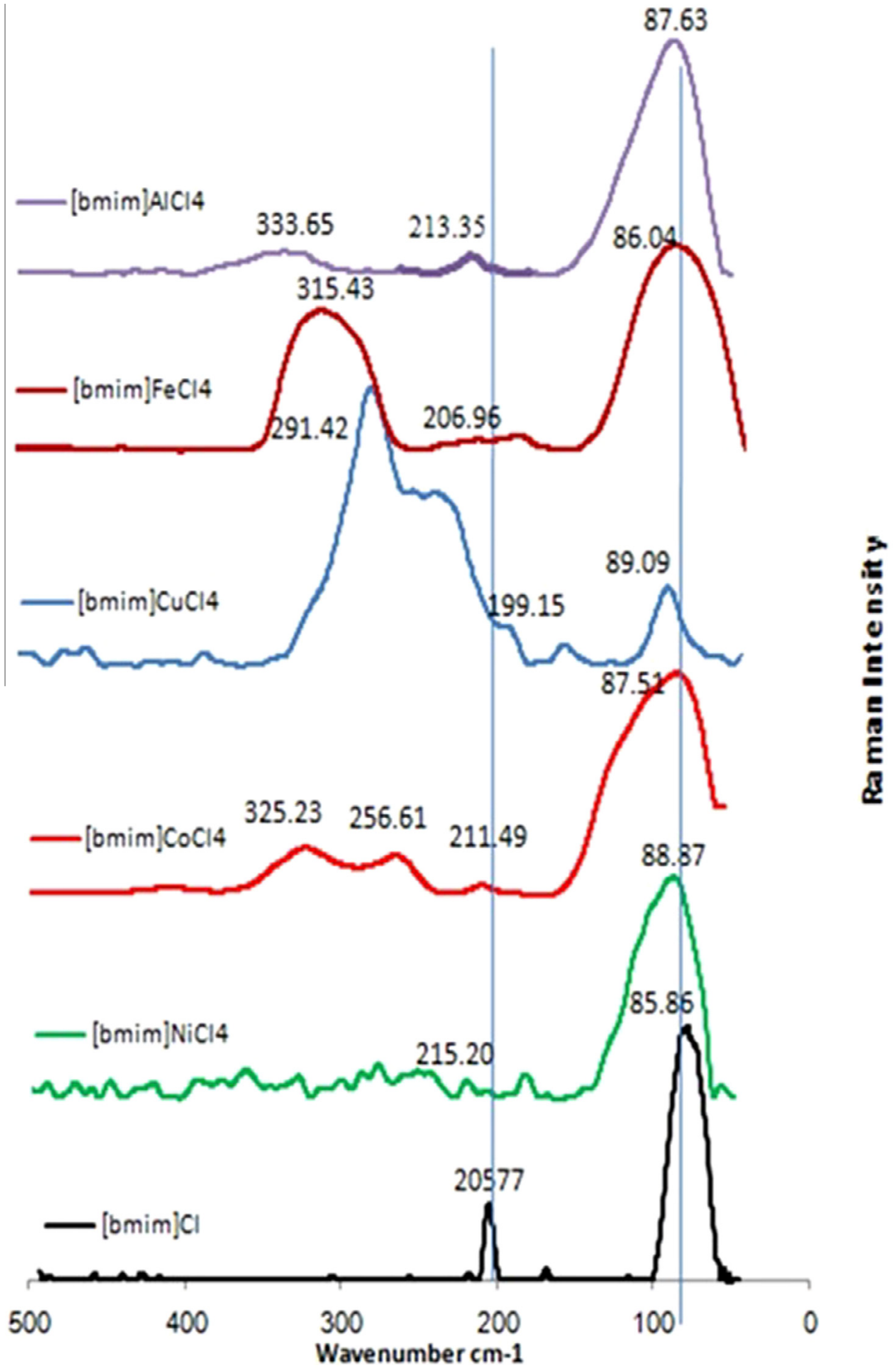


Figure 2 Normalized Raman spectra between 0 and 500 cm^{-1} for the original ionic liquid [bmim][Cl] and its Ni, Co, Cu, Fe and Al chlorides modification.

is slightly affected by the variation of different anion complexes. Also, these endothermic peaks of ionic liquid metal chlorides modification started to decomposed at 34.5, 325 and 410 °C with weight loss of 1.6%, 63.3% and 27.1% for $[\mathrm{bmim}][\mathrm{NiCl_4}]^{--}$, 38.5, 340 and 400 °C with weight loss of 2.7%, 57.2% and 12.7% for $[\mathrm{bmim}][\mathrm{CoCl_4}]^{--}$, 34.5, 240 and 330 °C with weight loss of 2.4%, 22.1% and 53.8% for $[\mathrm{bmim}][\mathrm{CuCl_4}]^{--}$, 40.0, 250 and 390 °C with weight loss of 2.5%, 31.6% and 38.5% for $[\mathrm{bmim}][\mathrm{FeCl_4}]^{-}$ and finally 41.5, 260 and 375 °C with weight loss of 5.6%, 31.1% and 56.6% for $[\mathrm{bmim}][\mathrm{AlCl_4}]^{-}$. Thermogravimetric analyses indicated that, the thermal stability of ionic liquid metals chloride modification is higher than the original one [bmim]Cl, which was stable up to 230 °C, while $[\mathrm{bmim}][\mathrm{NiCl_4}]^{--}$, [bmim]

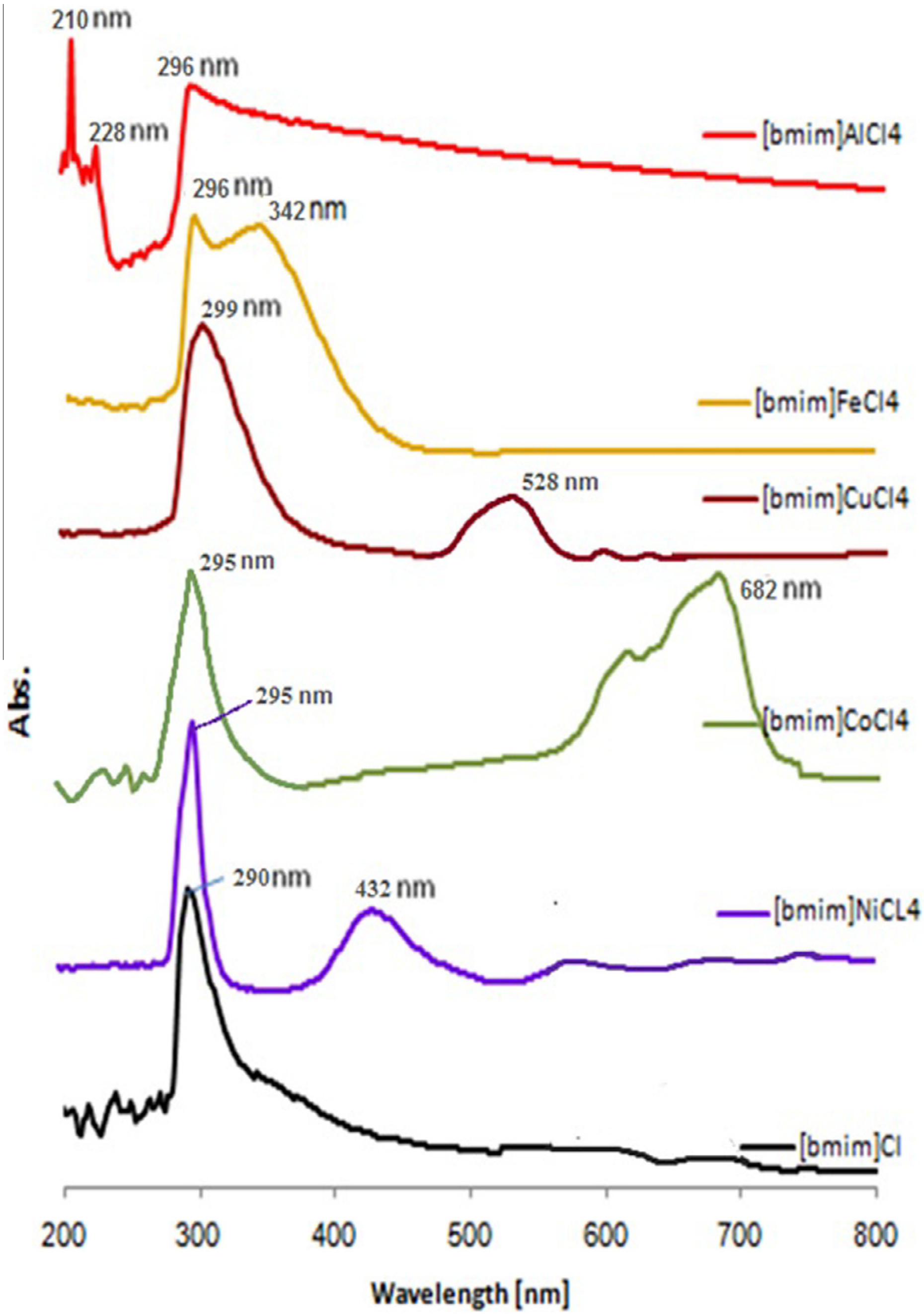


Figure 3 UV–Vis absorption spectra of the original ionic liquid [bmim][Cl] and its Ni, Co, Cu, Fe and Al chlorides modification.

$[CoCl_4]^{--}$, $[bmim][CuCl_4]^{--}$, $[bmim][FeCl_4]^{-}$ and $[bmim][AlCl_4]^{-}$ were slightly stable at 325°, 340°, 240°, 250° and 260°, respectively [26,27].

3.2. Effect of ionic liquid metals chloride combination on transesterification reaction

3.2.1. Mechanism of the reaction

The mechanism of the transesterification reaction catalyzed by ionic liquid modified by metals chloride was discussed in our previous publication [12]. The reaction mechanism for the transesterification of fatty acids which is comparable with vegetable oil and methanol over original ionic liquid [bmim][Cl] and its metals chloride modification $[bmim][MCl_4]$ is shown in steps 1–5. The first is the combination of a carbonyl group with a proton to form a carbocation. The final is the reaction of the carbocation with methanol and the formation of methyl esters (biodiesel product) and methanol.

Step 1: Acid takes a proton (a hydrogen ion) from the ionic liquid. The proton becomes attached to one of the lone pairs on the oxygen which is double bonded to the carbon.

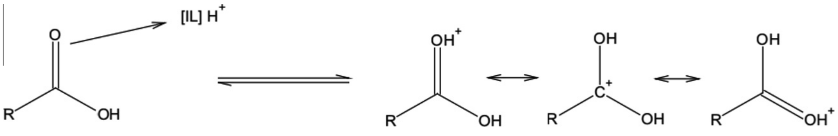


Table 1 The weight loss of the original ionic liquid [bmim][Cl] and its Ni, Co, Cu, Fe and Al chlorides modification.

Samples	Weight loss, %		
	1st peak	2nd peak	3rd peak
[bmim][Cl]	7.5	77.4	15.1%
$[bmim][NiCl_4]^{--}$	1.6	63.3	27.1
$[bmim][CoCl_4]^{--}$	2.7	57.2	12.7
$[bmim][CuCl_4]^{--}$	2.4	22.1	53.8
$[bmim][FeCl_4]^{-}$	2.5	31.6	38.5
$[bmim][AlCl_4]^{-}$	5.6	31.1	56.6

Step 2: The positive charge on the carbon atom is attacked by one of the lone pairs on the oxygen of the alcohol molecule.

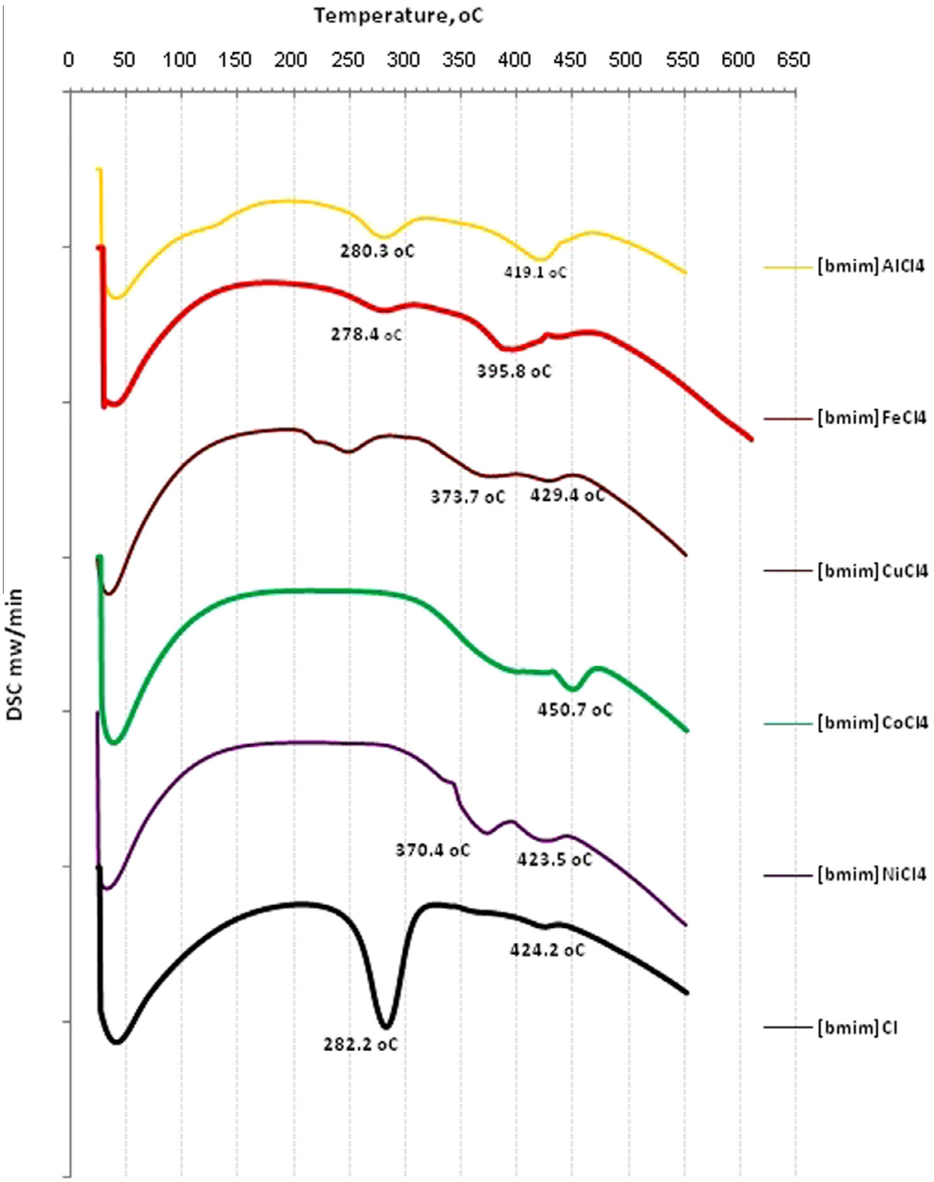


Figure 4 DSC curves of the original ionic liquid [bmim][Cl] and its Ni, Co, Cu, Fe and Al chlorides modification.

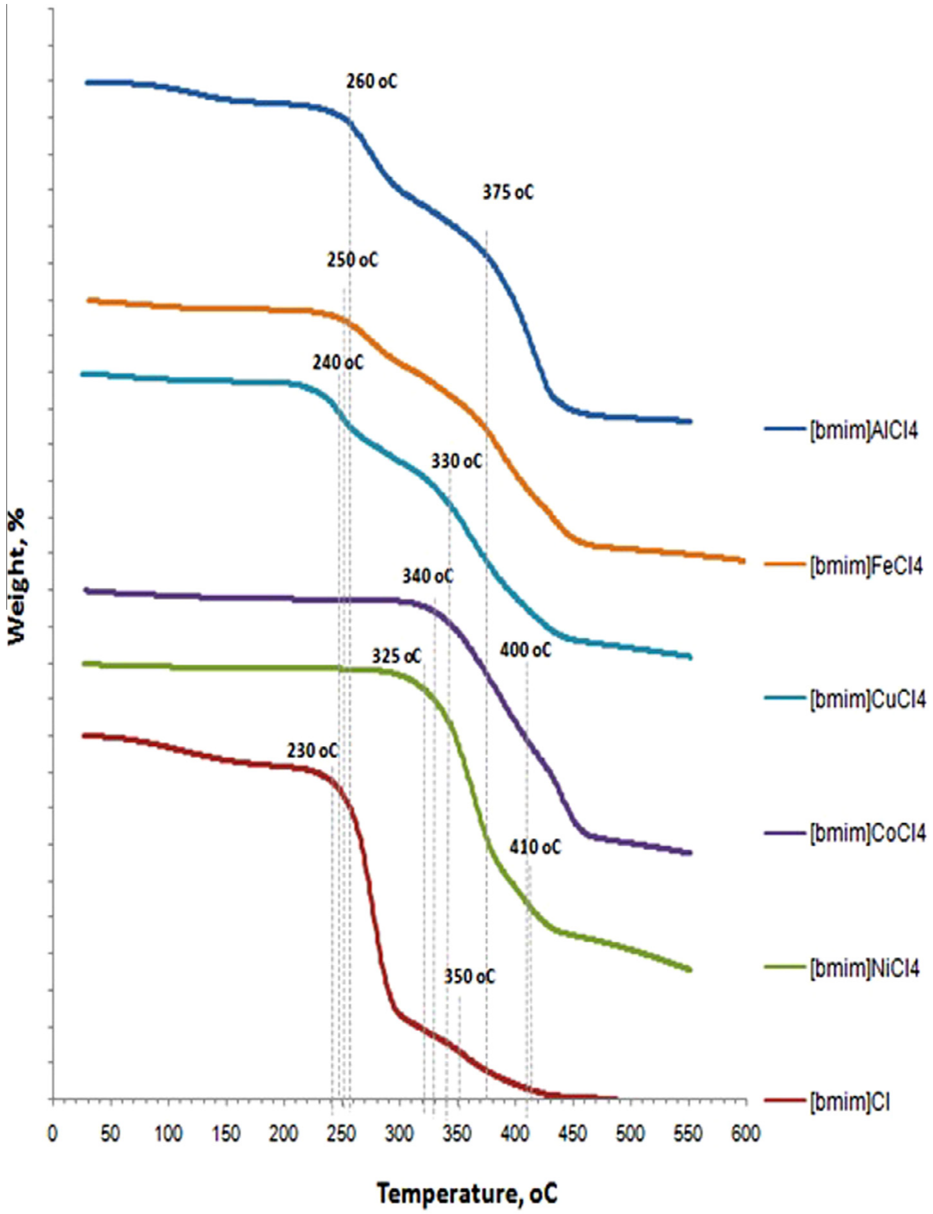
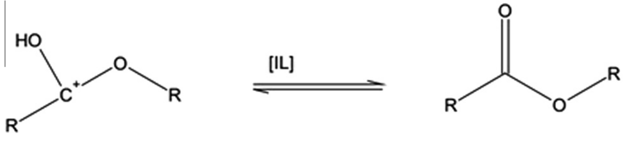


Figure 5 TGA curves of the original ionic liquid [bmim][Cl] and its Ni, Co, Cu, Fe and Al chlorides modification.

Step 3: Proton gets transferred from one of the oxygen atoms to the other.

Step 4: A molecule of water is lost from the ion.

Step 5: The hydrogen is removed from the oxygen by reaction with the IL ion which was formed in the first step.



3.2.2. Yield of biodiesel

Fig. 6 illustrates the relation between the original ionic liquid [bmim]Cl and its metals chloride forms and its efficiency as a catalyst for the production of biodiesel represented as yield %. The results indicate that the yield of biodiesel yield is increased according to the following order:[bmim]Cl^-

$<[\text{bmim}][\text{CoCl}_4]^{--} < [\text{bmim}][\text{NiCl}_4]^{--} < [\text{bmim}][\text{CuCl}_4]^{--} < [\text{bmim}][\text{AlCl}_4]^{-} < [\text{bmim}][\text{FeCl}_4]^{-}$.

As noted, the trivalent metallic ions forms of Fe3+ and Al3+ exhibit higher yields of 97% and 86%, respectively, due mainly to their stronger Lewis acidity compared to the bivalent metallic ions of Cu2+, Ni2+ and Co2+. The divalent metal chloride forms by CuCl2, NiCl2 and CoCl2 seem to have promoted the transesterification process of used vegetable oil as indicated by their lower efficiency to produce the biodiesel fuel at only 66%, 60% and 18%, respectively. Cu, Ni and Co are all belongs to transition metals fourth cycle elements in the periodic table with an atomic radius order: −Cu < Ni < Co which is directly proportional to its Lewis acidity and as described by Feng et al. [28].

3.2.3. Viscosity and density

The viscosity of biodiesel is an important factor in the performance of fuel in an engine as both low and high viscosities can have negative effects on engine performance. Low viscosity liquids do not provide sufficient lubrication for the precision fit of fuel injection pumps (which results in leakage or increase of frictional wear), while high viscosity fuel liquids lead to the formation of large droplets on injection, poor combustion and increased exhaust emissions [29]. Figs. 7 and 8 illustrate the effect metals chloride forms on the viscosity and density of the produced biodiesel compared to the untreated waste vegetable oil. The viscosity and density of biodiesel were found to be in the following order: $-[\text{bmim}][\text{CoCl}_4]^{--} > [\text{bmim}][\text{NiCl}_4]^{--} > [\text{bmim}][\text{CuCl}_4]^{--} > [\text{bmim}][\text{AlCl}_4]^{-} > [\text{bmim}][\text{FeCl}_4]^{-}$.

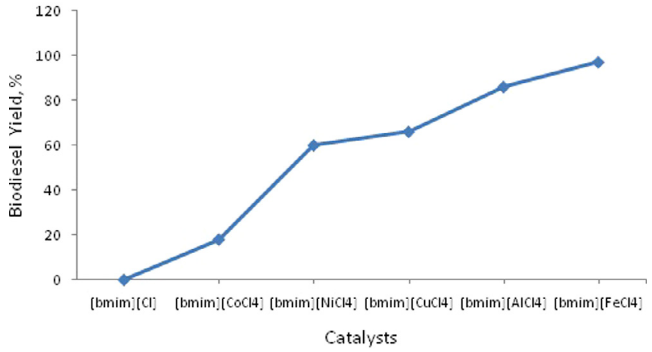


Figure 6 The yield of biodiesel as a function of modification of ionic liquid by metals chloride.

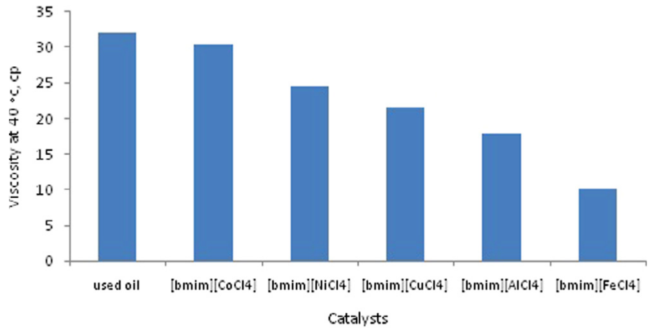


Figure 7 The viscosity of biodiesel as a function of modification of ionic liquid by metals chloride.

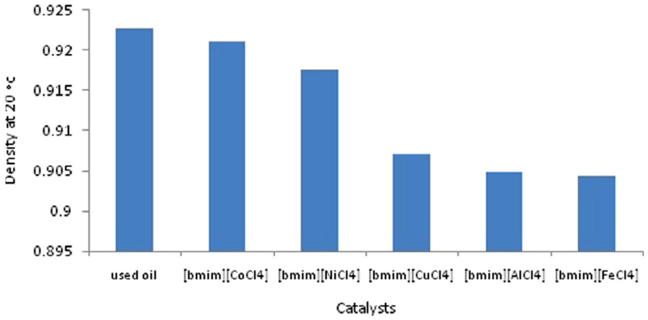


Figure 8 The density of biodiesel as a function of modification of ionic liquid by metals chloride.

The viscosity of biodiesel produced using $[\text{bmim}][\text{FeCl}_4]$-was significantly the least at 10.2 cP. This decrease in viscosity; compared to the modified waste oil viscosity; could probably be due to the high ratio of conversion of the oil triglycerides to the methyl esters biodiesel fuel, that is distinguished by a higher fluidity than its respective oil. The decrease in the biodiesel viscosity was also accompanied by a decrease in its density from 0.9227 to 0.9044 g/cm^3, as the density is directly proportional to the viscosity and this depends on the methyl esters content [30].

4. Conclusions

The ionic liquid metals chloride modification [bmim][MCln] exhibit good catalytic performances in the transesterification process than the original ionic liquid $[\text{bmim}]\text{Cl}^{-}$. By using $[\text{bmim}][\text{FeCl}_4]^{-}$ and $[\text{bmim}][\text{AlCl}_4]^{-}$ as catalysts in the transesterification process, the yield of biodiesel reached 97 and 86 wt.%, respectively. The biodiesel liquid fuel viscosity and density were recorded at suitable liquid fuels of 10.2, 17.8 cP and 0.9048, 0.9044 g/cm^3 for density, respectively. The ionic liquid was also easily separated from the reaction mixture; implying that these bi cation ionic liquids with imidazolium structure could be considered and recommended as suitable catalysts for the synthesis of biodiesel. Among the metal chlorides used Fe and Al proved to acquire the highest yields and suitable biodiesel liquid fuel within the metal series studied.

Acknowledgments

The authors would like to thank Science and Technology Development Fund (STDF), Egyptian Petroleum Research Institute (EPRI) and the Faculty of Science – Zagazig University for financial support.

References

[1] J. Dupont, P.A.Z. Suarez, M.R. Meneghetti, S.M.P. Meneghetti, Energy Environ. Sci. 2 (2009) 1258–1265.
[2] L. Andreani, J.D. Rocha, Braz. Chem. Eng. 29 (1) (2012) 1–13.
[3] W.N.R.W. Isahak, M. Ismail, J. MohdJahim, J. Salimon, M.A. Yarmo, Trend Appl. Sci. Res. 6 (9) (2011) 1055–1062.
[4] F.A. Dawodu, O.O. Ayodele, T. Bolanle-Ojo, Egypt. J. Petrol. 23 (2) (2014) 191–199.
[5] T.M. Labib, S.I. Hawash, K.M. El-Khatib, A.M. Sharaky, G.I. El Diwani, E. Abdel Kader, Egypt. J. Petrol. 22 (1) (2013) 9–16.

[6] T.S. Gendy, S.A. El-Temtamy, Egypt. J. Petrol. 22 (1) (2013) 43–51.
[7] S.R. Tewfik, N.M. El Defrawy, M.H. Sorour, Egypt. J. Petrol. 22 (2) (2013) 269–276.
[8] G. Santori, G.D. Nicola, M. Moglie, F. Polonara, Appl. Energy 92 (2012) 109–132.
[9] Z. Yang, K.P. Zhang, Y. Huang, Z. Wang, J. Mol. Catal. B Enzym. 63 (2010) 23–30.
[10] E.A. Shalaby, N.S. El-Gendy, Two steps alkaline transesterification of waste cooking oil and quality assessment of produced biodiesel, Int. J. Chem. Biochem. Sci. 1, 30–35.
[11] N.S. El-Gendy, S.F. Deriase, A. Hamdy, Energy Sources Part A 36 (6) (2014) 623–637.
[12] X.B. Cui, J.L. Cai, Y. Zhang, R. Li, T.Y. Feng, Ind. Eng. Chem. Res. 50 (20) (2011) 11521–11527.
[13] T. Long, Y. Deng, S. Gan, J. Chen, Chin. J. Chem. Eng. 18 (2) (2010) 322–327.
[14] M.S.A. Ginting, M.T. Azizan, S. Yusup, J. Fuel 93 (2012) 82–85.
[15] Ru Yang, Mengxing Su, J. Zhang, J. Fuqiang, C. Zha, M. Li, X. Hao, Bioresour. Technol. 102 (3) (2011) 2665–2671.
[16] P. Vidya, A. Chadha, Mol. Catal. Enzym. 57 (2009) 145–148.
[17] Z.S. Qureshia, K.M. Deshmukha, M.D. Bhora, B.M. Bhanage, Catal. Commun. 10 (2009) 833–837.
[18] M. Hayyan, F.S. Mjalli, M.A. Hashim, I.M. Al-Nashef, X.M. Tan, K.L. Chooi, Appl. Sci. 10 (2010) 1176–1180.
[19] D. Zhao, Z. Fei, C. André, G. Laurenczy, P.J. Dyson, J. Chem. Commun. 34 (2004) 2500–2503.
[20] J. Liang, X. Ren, J. Wang, M. Jiang, Z. Li, Fuel Chem. Technol. 38 (3) (2010) 275–280.
[21] X. Sun, S. Zhao, M. Zhang, Petrol. Sci. 2 (1) (2005) 134–139.
[22] R.E.D. Sesto, T.M. McCleskey, A.K. Burrell, G.A. Baker, J.D. Thompson, B.L. Scott, Chem. Commun. 4 (2008) 447–449.
[23] M.S. Sitze, E.R. Schreiter, E.V. Patterson, R.G. Freeman, Inorg. Chem. 40 (2) (2001) 298–304.
[24] P. Xiaowei, Y. Hai, Y. Liangyu, P. Yang, J. Wang, R. Xua, et al, Carbon 48 (2010) 2501–2505.
[25] S.C. Paul, C.S. Catherine, B. Denis, F. Bernard, R. Leszek, Y. Chauvin, G.E. Marcelle, B. Catherine, A.L. Rollet, Dalton Trans. 39 (5) (2010) 1379–1388.
[26] L. Huang, W. Huang, H.Y. Fu, G.Z. Wu, Z.J. Guo, W.S. Wu, et al, Phys. Chem. 58 (10) (2013) 1150–1155.
[27] B.A. Melquizedeque, P.U. Alexandre, O.S. Vianney, C.D. Valerio, D. Jairton, C.R. Joel, et al, Thermochim. Acta 502 (2010) 20–23.
[28] G. Feng, F. Zhen, X. Tian, Y. Long, L. Jiang, Bioresour. Technol. 102 (2011) 6469–6472.
[29] Cennatek Bioanalytical Services, Kinematic Viscosity According to ASTM D445, 2013 (retrieved June 13, 2013).
[30] C. Laureano, R. Miguel, Q. Enrique, A. Ramón, L. Magın, O. Fermı́n, Ind. Eng. Chem. Res. 47 (2008) 7997–8004.