A Green Way for the Synthesis of Ester Oil by an Ionic Liquid as Both a Catalyst and Lubricant Additive

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Characterizations, determination of acid number and the yield of pentaerythrotol tetra-hexanoate, Figure S1, Figure S2, Figure S3, Table S1.

Characterizations

The kinematic viscosities of PETH and PETH+ [HMIM][DEHP] were measured using SVM 3000 (Anton Paar), and the viscosity index was determined according to ASTMD 2270-10.

The copper strip test was conducted in accordance with ASTMD 130-10 (temperature: 100 ℃, time: 3 h).

Using the Pyris Diamond TG/DTA analyzer, thermogravimetric measurements were conducted under a nitrogen atmosphere from 25 to 1000°C at a heating rate of 10°C/min.

XPS analysis was measured by using an Al Kα (1486.8 eV) radiation gun (Thermo K-Alpha+)

The NMR spectra of esters were measured using Bruker ARX 400 spectrometer operating at 400 MHz (1H). All spectra were recorded in CDCl3 and chemical shifts (δ) are given in ppm relative to the residual solvent peak with respect to tetramethylsilane.

The morphology of wear tracks lubricated with PETH and PETH + [HMIM][DEHP] was scanned by using FESEM.

The OM was used to measure the wear scar diameter of the upper running ball lubricated by PETH and PETH + [HMIM][DEHP].

The wear volume of the lower test block disc was measured using a KLA-TENCOR MicroXAM-800 white light interferometry three-dimensional profiler.

Determination of acid number:

The AN_a and AN_b were determined by titration method according to ISO 6618: 1997(E):

Into a 250-ml conical flask, introduce 2.0 g sample. Add 100 ml of the titration solvent A (containing toluene: isopropyl alcohol: water at a ratio of 500:495:5) and 0.5 ml of the indicator solution (solution:1.0 g p-naphtholbenzein in 100 mL solvent A), and without stoppering, swirl until the test portion is completely dissolved by the solvent. Add the potassium hydroxide solution (0.1 mol/L standard volumetric alcoholic solution)in increments and swirl to disperse the potassium hydroxide as necessary.

Calculate the acid number, AN, in milligrams of KOH per gram of the test sample,from the equation:

$$AN = \frac{(V_1 - V_0)c_{KOH} \times 56.1}{m}$$

V_1 is the volume, in millilitres, of potassium hydroxide solution required for titration of the test portion;

V_o is the volume, in millilitres, of potassium hydroxide solution required for titration of the blank solution;

c_{KOH} is the concentration, in moles per litre, of the standard volumetric potassium hydroxide solution;

m is the mass, in grams , of the test portion.

Determination of the yield of pentaerythrotol tetra-hexanoate

The yield of pentaerythrotol tetra-hexanoate was determined by quantitative ^{1}H NMR with triphenylmethane as internal standard of these products and was calculated by the following formula:

$$\text{Yield}/\% = \frac{m}{m_{\text{weighting}}} \times 100\%$$

The m was calculated by this formula:

$$\frac{\frac{m_{\text{triphenylmethane}}}{M_{\text{triphenylmethane}}}}{\frac{8m}{M_{\text{pentaerythrotoltetrahexanoate}}}} = \frac{n_{1H}}{n_{8H}}$$

Where m represents the mass of pentaerythrotol tetrahexanoate in the product, m_{weighing} represents the mass of the product, $m_{\text{triphenylmethane}}$ represents the mass of the standard substance triphenylmethane, $M_{\text{tripheylmethane}}$ represents the molecular weight of triphenylmethane, $M_{\text{pentaerythrotoltetrahexanoate}}$ represents the molecular weight of pentaerythrotol tetrahexanoate.

Figure S1

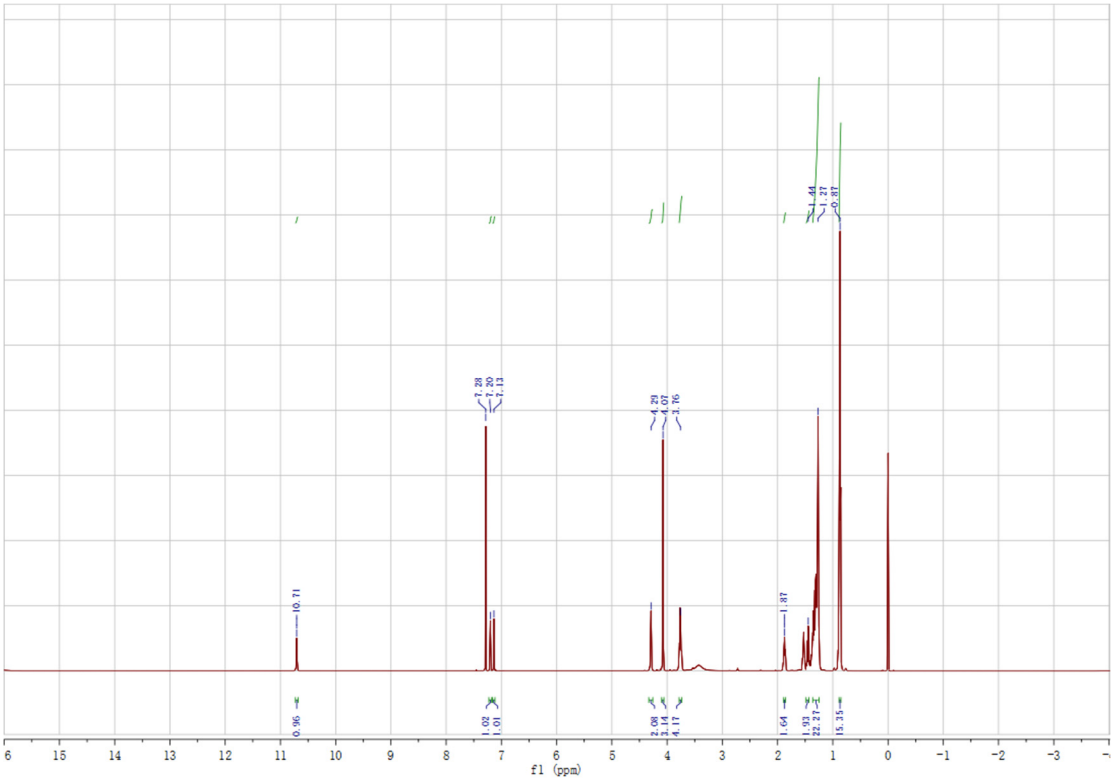

Figure S1. The ^{1}H NMR spectrum of [HMIM][DEHP].

^{1}H NMR: (400 MHz, CHLOROFORM-d) δ = 10.71 (s, 1H), 7.20 (s, 1H), 7.13 (s, 1H), 7.60 (s, 2H), 4.29(t, 2H), 4.07(s, 3H), 3.70-3.80(m, 4H), 7.07 (s, 1H), 3.72-3.80 (m, 4H), 1.84-1.90(m, 2H), 1.40 - 1.50 (m, 2H), 1.25-1.37 (m, 22H), 0.85-0.90 (m, 15H).

Figure S2

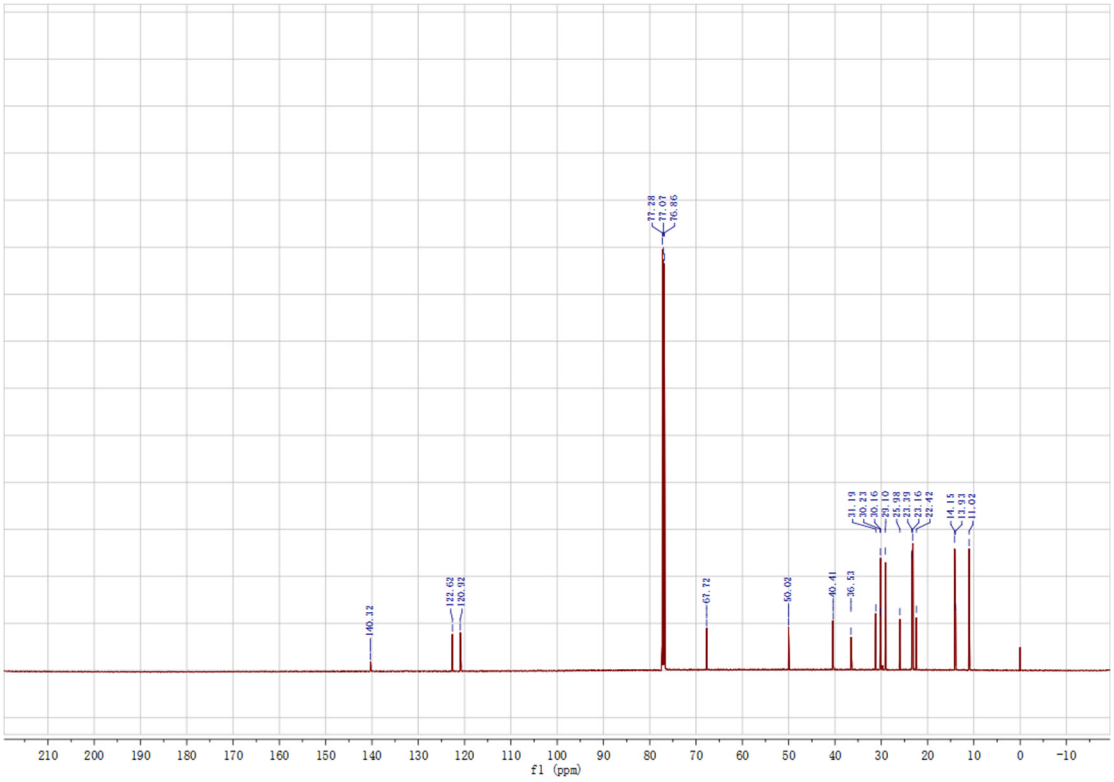

Figure S2. The ^{13}C NMR spectrum of [HMIM][DEHP].

^{13}C NMR: (400 MHz, CHLOROFORM-d) δ = 140.32, 122.62, 120.92, 77.28, 77.07, 76.86, 67.72, 50.02, 40.41, 36.53, 31.19, 30.23, 30.16, 29.1, 25.98, 23.39, 23.16, 22.42, 14.15, 13.93, 11.02.

Figure S3

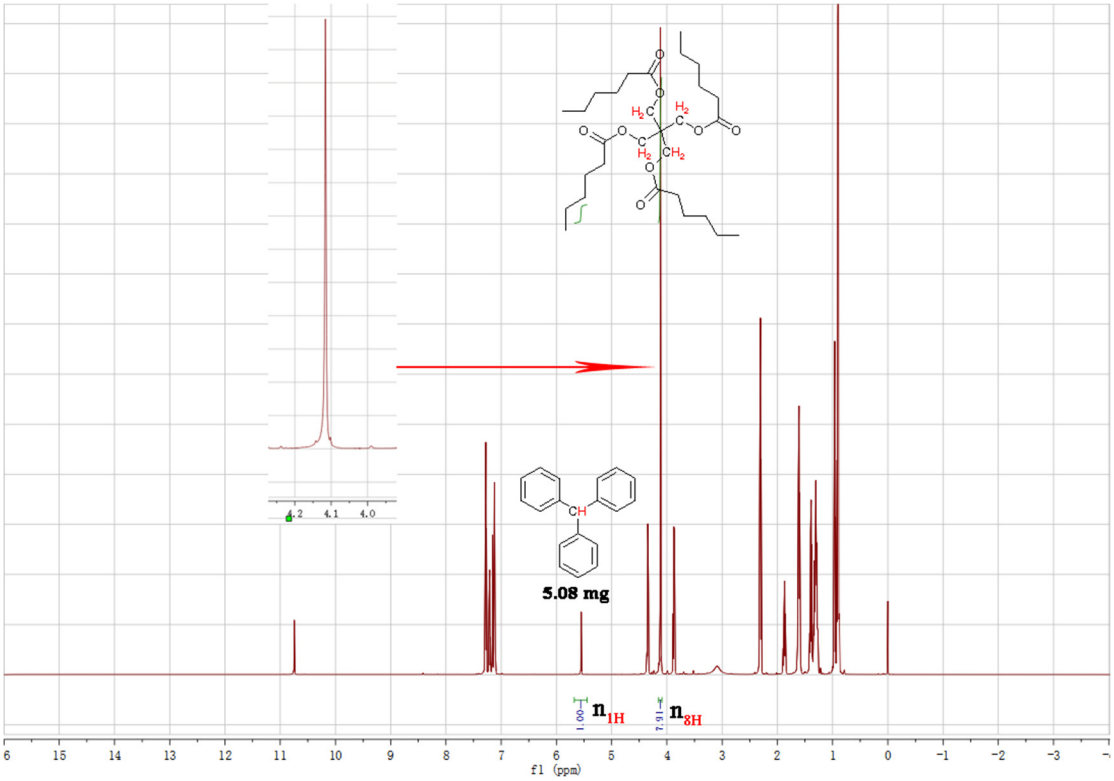

Figure S3. The ^{1}H NMR spectrum of PETH + [HMIM][DEHP].

Table S1 The vendors and quality or grade of chemicals

Chemicals	Purity	Vendor
1-Butyl-3-methylimidazole bromide	98%	J&K Scientific
1-Hexyl-3-methylimidazole bromide	98%	J&K Scientific
1-Octyl-3-methylimidazole bromide	98%	J&K Scientific
1-Decyl-3-methylimidazole bromide	98%	J&K Scientific
Bis(2-ethylhexyl) hydrogen phosphate	98%	MACKLIN
Dichloromethane	99%	MACKLIN
Sodium hydroxide	98%	MACKLIN
Pentaerythrotol	≥98%	Sinopharm Chemical Reagent
Hexanoic acid	99%	MACKLIN