Supporting Information

Solvent-free one step aminolysis and alcoholysis of low-quality triglycerides using sodium modified CaO nanoparticles as solid catalyst

Dinesh Kumar^{a*}, Soo Min Kim^b, and Amjad Ali^c

This pdf file includes:

Materials and methods, Data (XRD, Mass and ¹H-NMR)

Figures. S1–S7

1. Materials and methods

X-ray diffraction (XRD) data for powder samples were collected on Panalytical's X'Pert Pro with Cu K α radiation. The samples were scanned in the range of $2\theta = 5-70^{\circ}$ at the scanning speed of 2°/min. The surface areas of the catalyst were determined by using the adsorption/desorption method at 77 K by the standard Brunauer-Emmett-Teller (BET) method using Micromeritics Tristar 3000 equipment. All samples were degassed at 473 K for 90 min under nitrogen atmosphere to remove the physisorbed moisture from the catalysts. The basic strengths of the catalysts (H) were determined by Hammett indicators: neutral red (H = 6.8), bromthymol blue (H = 7.2), phenolphthalein (H = 9.3), Nile blue (H = 10.1), tropaeolin-O (H = 11.1), 2,4-dinitroaniline (H = 15.0), and 4- nitroaniline (H = 18.4). FTIR spectrum of triglycerides, FAMEs and corresponding amide derivative have been recorded on Thermo Nicolet iS10 spectrometer. Scanning electron microscopy (SEM) was performed on JEOL JSM 6510LV to collect the SEM images of the catalysts and transmission electron microscopy (TEM) were performed on HITACHI 7500 to record TEM images. Particle size analyzer (90-Plus, Brookhaven Instruments Corporation, USA) was used for particle size distribution of CaO and Na impregnated CaO. Fourier transform-nuclear magnetic resonance (FT-NMR) spectra of FAMEs and vegetable oil were recorded on a Bruker Avance-II (400 MHz) spectrophotometer. Mass spectra of amide derivative of methyl laurate were recorded on a Waters Micromass Q-ToF Micro mass spectrophotometer equipped with electrospray ionization (ESI) and atmospheric pressure chemical ionization (APcI) sources having mass range of 4000 amu in quadruple and 20000 amu in ToF.



Figure S1. Comparative XRD spectrum of 1-NaC/CaO-450, 2-NaC/CaO-450, 3-NaC/CaO-450, 4-NaC/CaO-450 and 5-NaC/CaO-450.



Figure S2. Comparative FTIR spectra of (a) cotton seed oil, (b) fatty acid amide of cotton seed oil, (c) cotton seed oil derived FAME, (d) fatty acid amide of cotton seed oil derived FAME, (e) mutton fat derived FAME, (f) fatty acid amide of mutton fat FAME, (g) methyl laurate, and (h) amide derivative of methyl laurate.



Figure S3. Comparison of ¹H-NMR spectra of (a) cotton seed oil, (b) fatty acid amide of cotton seed oil, (c) cotton seed oil FAME, (d) fatty acid amide of cotton seed oil FAME, (e) mutton fat FAME, (f) fatty acid amide of mutton fat FAME, (g) methyl laurate, and (h) amide derivative of methyl laurate.



Figure S4. Mass spectrum of fatty acid amide derived from methyl laurate.



Figure S5. Comparison of ¹H-NMR spectrum of triglycerides and corresponding methyl ester, (a) cotton seed oil, (b) cotton seed oil derived fatty acid methyl ester, (c) karanja oil, (d) karanja oil derived methyl ester, (e) jatropha oil, (f) jatropha oil derived methyl ester, (g) soybean oil, (h) soybean oil derived methyl ester, (i) castor oil, (j) castor oil derived methyl ester, (k) mutton fat and (l) mutton fat derived methyl ester.



Figure S6. (A) XRD patterns and (B) FTIR spectrum of fresh NaC/CaO and recycled NaC/CaO.



Figure S7. TEM image of recycled NaC/CaO.