Electronic Supporting Information (ESI)

Aminolysis of triglycerides using nanocrystalline nickel doped CaO as an efficient solid catalyst

Dinesh Kumar, KM Abida and Amjad Ali*

This pdf file includes:

Figures : S1, S2, S3, S4, S5, S6 and S7

Data : FTIR and ^{1}H-NMR

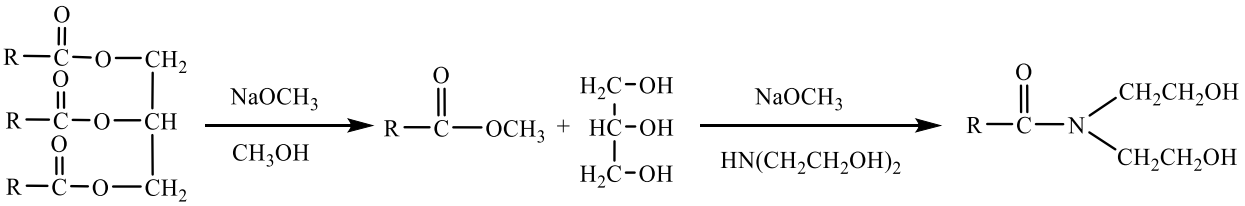

Fig S1. Industrial method for the preparation of fatty acid amides from triglycerides.

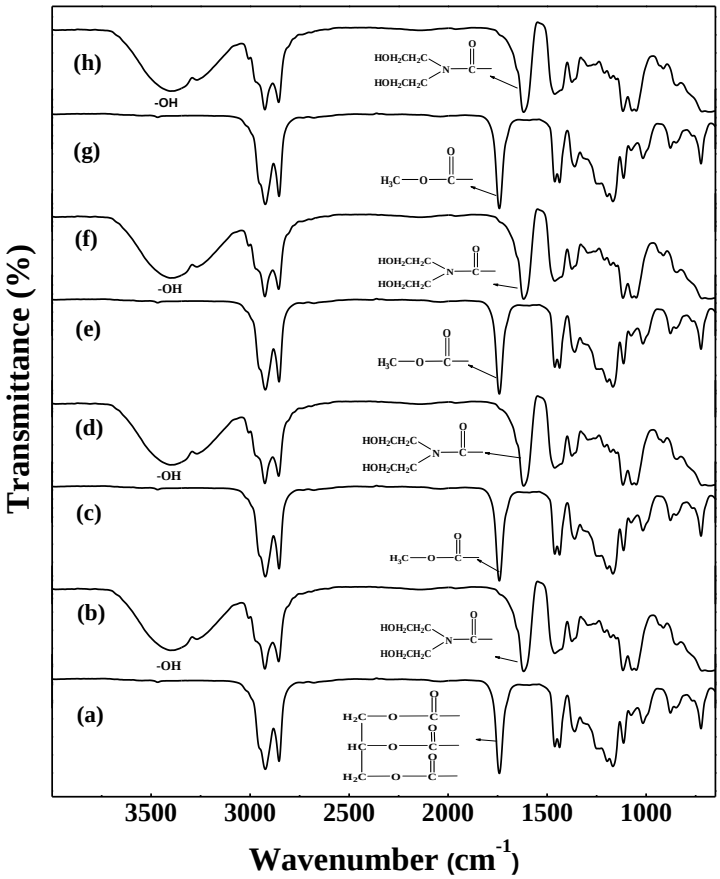

Fig S2. Comparative FTIR spectra of (a) cotton seed oil, (b) fatty acid amide of cotton seed oil, (c) cotton seed oil derived FAME, (d) fatty acid amide of cotton seed oil derived FAME, (e) mutton fat derived FAME, (f) fatty acid amide of mutton fat derived FAME, (g) methyl laurate, and (h) amide derivative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

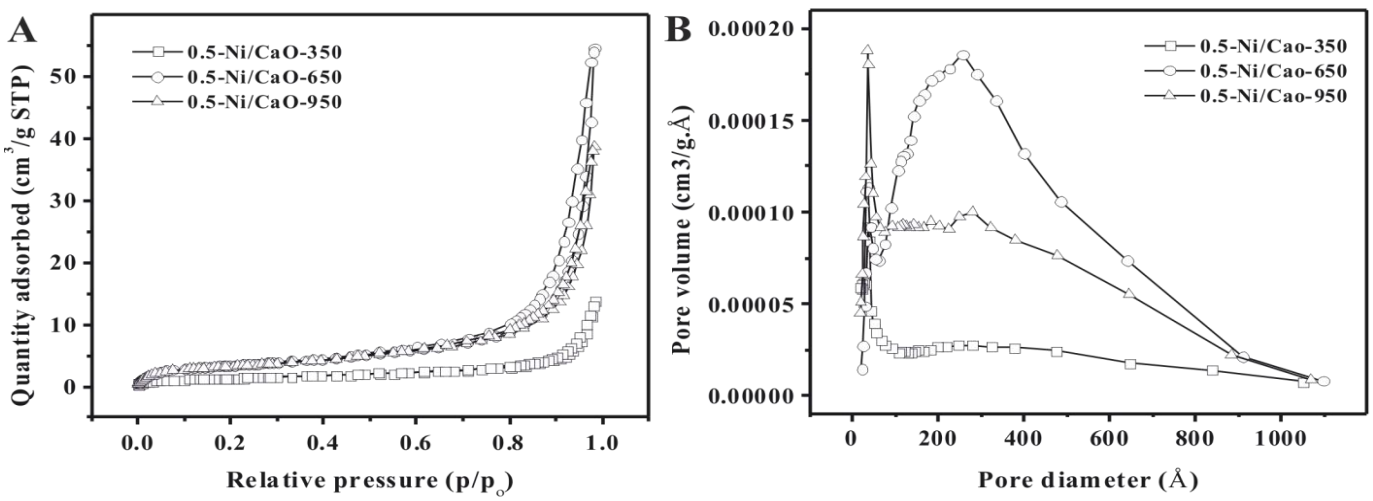

Fig S3. (A) Nitrogen adsorption-desorption isotherms of 0.5-Ni/CaO-350, 0.5-Ni/CaO-650 and 0.5-Ni/CaO-950; **(B)** BJH pore-size distribution profile of 0.5-Ni/CaO-350, 0.5-Ni/CaO-650 and 0.5-Ni/CaO-950.

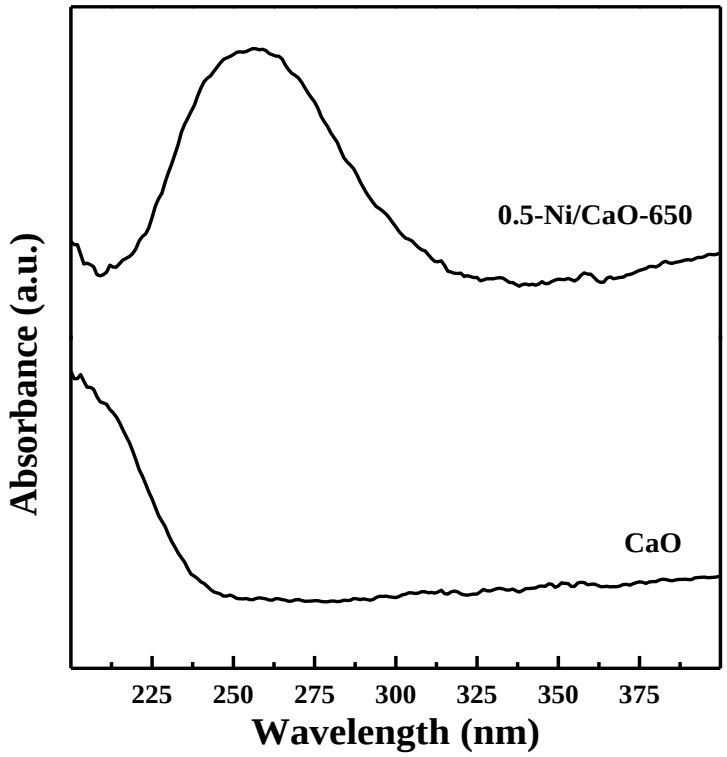

Fig S4. Comparative solid state UV-Visible spectrum of CaO and Ni/CaO and calcined in the temperature range of 150-950 °C.

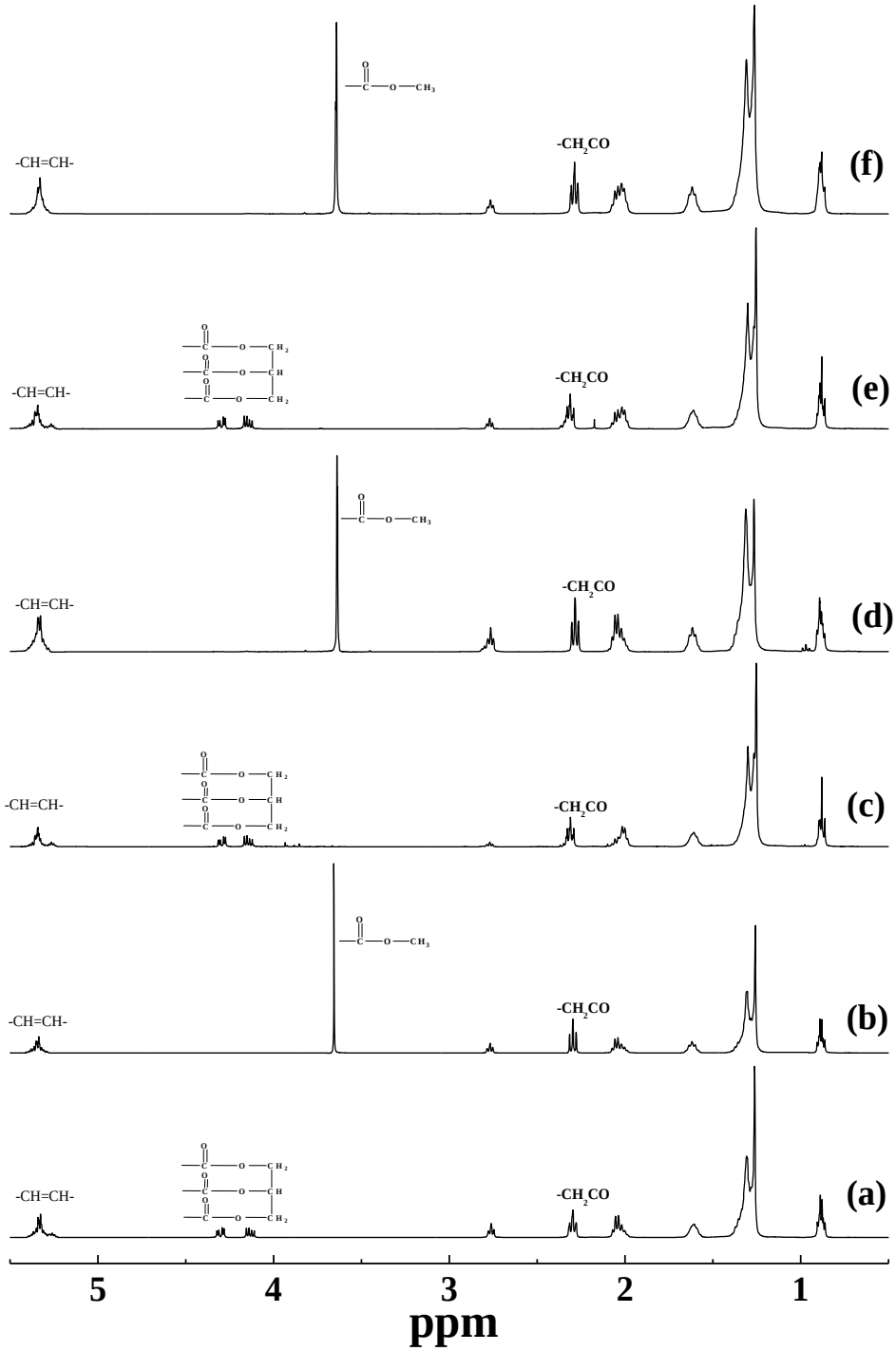

Fig S5. Comparison of ^{1}H-NMR spectra of triglycerides and corresponding methyl ester, (a) cotton seed oil, (b) cotton seed oil derived fatty acid methyl ester, (c) karanja oil, (d) karanja oil derived methyl ester, (e) jatropha oil, and (f) jatropha oil derived methyl ester.

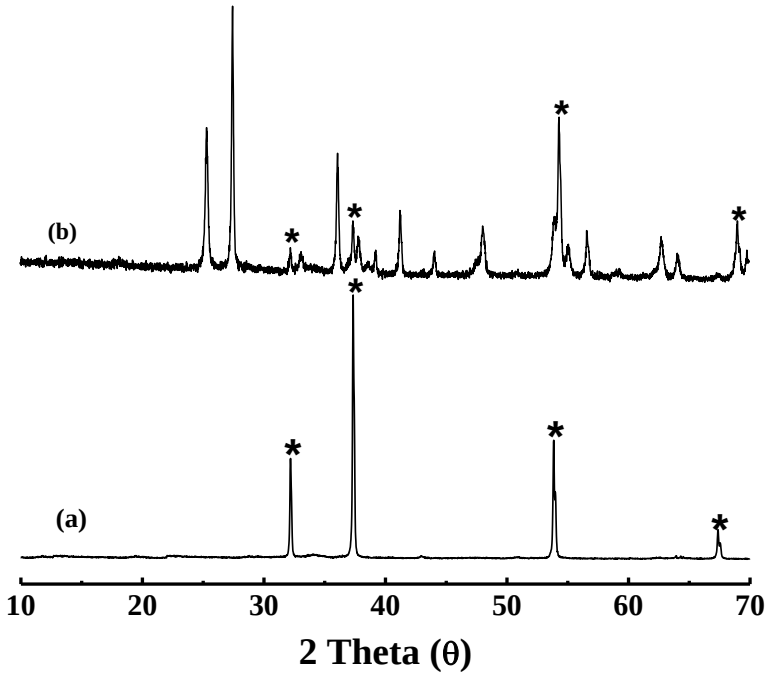

Fig S6. Comparative XRD patterns of (a) fresh and (b) used 0.5-Ni/CaO-650 (* = CaO phase)

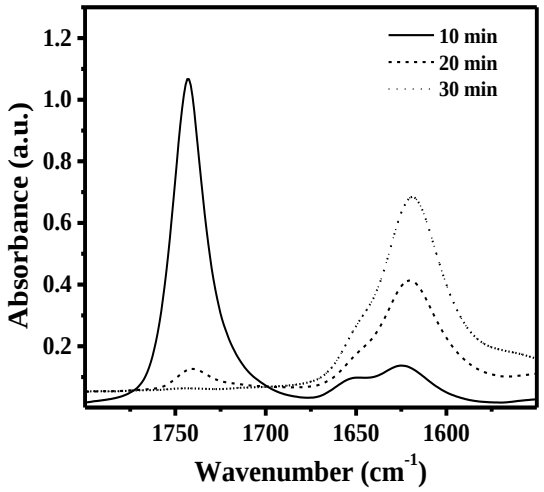

Fig S7. Progress of the aminolysis of WO with diethanolamine monitored by FTIR spectroscopy. Reaction conditions; diethanolamine: cotton seed oil = 5:1 (m/m), catalyst amount = 5 wt%, temperature = 110 ºC.

NMR and FTIR data for FAME and FAA prepared in presence of Ni/CaO catalyst.

WO: ^{1}H-NMR ($CDCl_3$, δ ppm): 5.34 (m, -C**H**=C**H**-), 4.28-4.32 (dd, -C-CH_2-O-CO-C), 4.10-4.15 (dd, -C-CH_2-O-CO-C), 2.76 (m, -CH=CH-C**H₂**-CH=CH-), 2.31 (m, -C**H₂**-CO-), 2.02 (m, -C**H**$_2$-(CH_2)$_n$-), 1.6-1.25 (m, -(C**H₂**)$_n$-), 0.88 (m, -CH_2-C**H₃**).

WO derived FAME: Yield > 99%. ^{1}H-NMR ($CDCl_3$, δ ppm): 5.34 (m, -C**H**=C**H**-), 3.6 (s, -OC**H₃**), 2.77 (m, -CH=CH-C**H₂**-CH=CH-), 2.3 (m, -C**H₂**-CO-), 2.03 (m, -C**H**$_2$-(CH_2)$_n$-), 1.6-1.25 (m, -(C**H₂**)$_n$-), 0.88 (m, -CH_2-C**H₃**).

WO derived FAA: Yield > 99%. FTIR (cm^{-1}): 3397 (ν_{OH}), 1615 ($\nu_{C=O}$); ^{1}H-NMR ($CDCl_3$, δ ppm): 5.3 (m, -C**H**=C**H**-), 3.77 (m, -C**H₂**OH), 3.46 (m, -NC**H₂**-), 2.7 (m, -CH=CH-C**H₂**-CH=CH-), 2.31 (m, -C**H₂**-CO-), 2.0 (m, -C**H**$_2$-(CH_2)$_n$-CH-), 1.6-1.25 (m, -(C**H₂**)$_n$-), 0.95 (m, -CH=CH-C**H₃**), 0.87 (m, -CH_2-C**H₃**).

FAA derivative of WO derived FAME: Yield > 99%. FTIR (cm^{-1}): 3398 (ν_{OH}), 1615 ($\nu_{C=O}$); ^{1}H-NMR ($CDCl_3$, δ ppm): 5.3 (m, -C**H**=C**H**-), 3.78 (m, -C**H₂**OH), 3.48 (m, -NC**H₂**-), 2.7 (m, -CH=CH-C**H₂**-CH=CH-), 2.3 (m, -C**H₂**-CO-), 2.0 (m, -C**H**$_2$-(CH_2)$_n$-), 1.6-1.25 (m, -(C**H₂**)$_n$-), 0.95 (m, -CH=CH-C**H₃**), 0.87 (m, -CH_2-C**H₃**).

MF derived FAA: Yield > 99%. FTIR (cm^{-1}): 3397 (ν_{OH}), 1615 ($\nu_{C=O}$); ^{1}H-NMR ($CDCl_3$, δ ppm): 5.34 (m, -C**H**=C**H**-), 3.77 (m, -C**H₂**OH), 3.46 (m, -NC**H₂**-), 2.3 (m, -C**H₂**-CO-), 2.0 (m, -C**H**$_2$-(CH_2)$_n$-), 1.6-1.25 (m, -(C**H₂**)$_n$-), 0.87 (m, -CH_2-C**H₃**).

FAA derivative of MF derived FAME: Yield > 99%. FTIR (cm^{-1}): 3398 (ν_{OH}), 1615 ($\nu_{C=O}$); ^{1}H-NMR ($CDCl_3$, δ ppm): 5.3 (m, -C**H**=C**H**-), 3.75 (m, -C**H₂**OH), 3.47 (m, -NC**H₂**-), 2.3 (m, -C**H₂**-CO-), 2.0 (m, -C**H**$_2$-(CH_2)$_n$-), 1.6-1.25 (m, -(C**H₂**)$_n$-), 0.87 (m, -CH_2-C**H₃**).

KO derived FAA: Yield > 99%. FTIR (cm^{-1}): 3397 (ν_{OH}), 1615 ($\nu_{C=O}$); ^{1}H-NMR ($CDCl_3$, δ ppm): 5.3 (m, -C**H**=C**H**-), 3.77 (m, -C**H₂**OH), 3.46 (m, -NC**H₂**-), 2.7 (m, -CH=CH-C**H₂**-CH=CH-),

2.31 (m, -C**H**$_2$-CO-), 2.0 (m, -C**H**$_2$-(CH$_2$)$_n$-CH-), 1.6-1.25 (m, -(C**H**$_2$)$_n$-), 0.95 (m, -CH=CH-C**H**$_3$)0.87 (m, -CH$_2$-C**H**$_3$).

FAA derivative of KO derived FAME: Yield > 99%. FTIR (cm^{-1}): 3398 (ν_{OH}), 1615 ($\nu_{C=O}$); ^{1}H-NMR ($CDCl_3$, δ ppm): 5.3 (m, -C**H**=C**H**-), 3.78 (m, -C**H**$_2$OH), 3.48 (m, -NC**H**$_2$-), 2.7 (m, -CH=CH-C**H**$_2$-CH=CH-), 2.3 (m, -C**H**$_2$-CO-), 2.0 (m, -C**H**$_2$-(CH$_2$)$_n$-), 1.6-1.25 (m, -(C**H**$_2$)$_n$-), 0.95 (m, -CH=CH-C**H**$_3$), 0.87 (m, -CH$_2$-C**H**$_3$).

JO derived FAA: Yield > 99%. FTIR (cm^{-1}): 3397 (ν_{OH}), 1615 ($\nu_{C=O}$); ^{1}H-NMR ($CDCl_3$, δ ppm): 5.3 (m, -C**H**=C**H**-), 3.77 (m, -C**H**$_2$OH), 3.46 (m, -NC**H**$_2$-), 2.7 (m, -CH=CH-C**H**$_2$-CH=CH-), 2.31 (m, -C**H**$_2$-CO-), 2.0 (m, -C**H**$_2$-(CH$_2$)$_n$-CH-), 1.6-1.25 (m, -(C**H**$_2$)$_n$-), 0.95 (m, -CH=CH-C**H**$_3$)0.87 (m, -CH$_2$-C**H**$_3$).

FAA derivative of JO derived FAME: Yield > 99%. FTIR (cm^{-1}): 3398 (ν_{OH}), 1615 ($\nu_{C=O}$); ^{1}H-NMR ($CDCl_3$, δ ppm): 5.3 (m, -C**H**=C**H**-), 3.78 (m, -C**H**$_2$OH), 3.48 (m, -NC**H**$_2$-), 2.7 (m, -CH=CH-C**H**$_2$-CH=CH-), 2.3 (m, -C**H**$_2$-CO-), 2.0 (m, -C**H**$_2$-(CH$_2$)$_n$-), 1.6-1.25 (m, -(C**H**$_2$)$_n$-), 0.95 (m, -CH=CH-C**H**$_3$), 0.87 (m, -CH$_2$-C**H**$_3$).

ML derived FAA: Yield > 99%. FTIR (cm^{-1}): 3397 (ν_{OH}), 1615 ($\nu_{amide\text{-}C=O}$); ^{1}H-NMR ($CDCl_3$, δ ppm): 3.8 (m, -C**H**$_2$OH), 3.5 (m, -NC**H**$_2$-), 2.3 (m, -C**H**$_2$-CO-), 1.6-1.25 (m, -(C**H**$_2$)$_n$-), 0.87 (m, -CH$_2$-C**H**$_3$)