

Supporting Information for:

Hydrothermal synthesis of rutile TiO₂ nanoflowers using Bronsted Acidic Ionic Liquid [BAIL]: Synthesis, characterization, growth mechanism

S 1. Synthesis of [CMIM] [HSO₄] Ionic Liquid

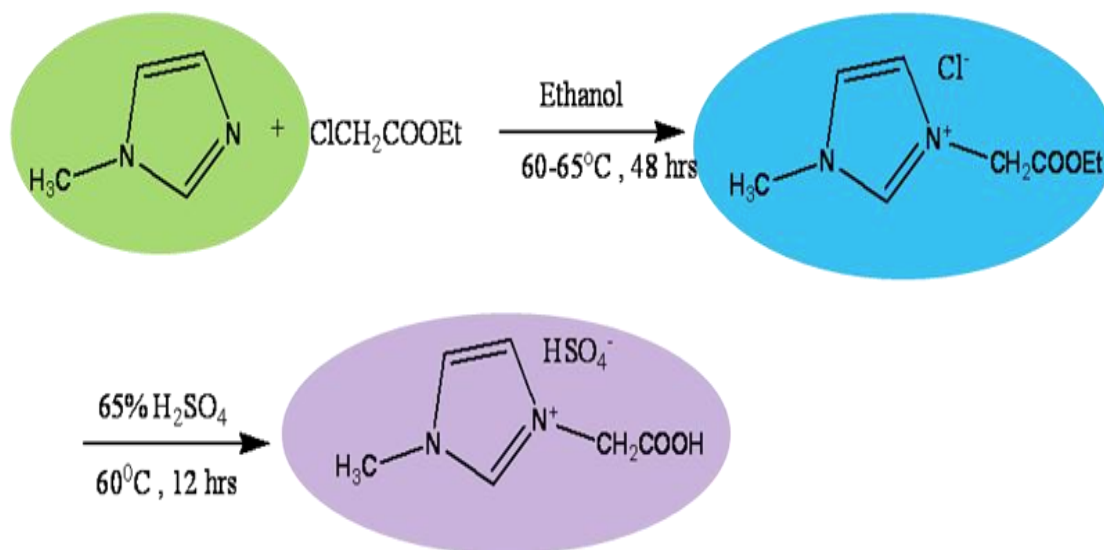
[CMIM] [HSO₄] was synthesized referring to the procedure:

[CMIM] [HSO₄] room temperature ionic liquid (RTIL) was recently synthesized in our group [1]. In brief, 0.1 M Methylimidazole and 0.15 M ethyl chloroacetate were added to a 100 mL round-bottom flask containing anhydrous 25 mL ethanol. This mixture was stirred and heated at 60–65 °C in oil bath for 48 h (TLC monitoring). Then ethanol was removed under reduced pressure by using rotary evaporator; formed viscous product. This product was purified by washing with ethyl acetate (10 X 3) and again dried in vacuum to obtain a pale yellow liquid (referred as **compound 1**: 3-ethoxycarbonyl-ethyl-1-methyl-imidazolium chloride). A stoichiometric amount of 65% sulfuric acid was added to compound 1 and stirred for 12 h at 60 °C. The mixture was evaporated under reduced pressure to remove ethanol and water. Formed product was then washed repeatedly with ethyl acetate to remove nonionic residue and dried in vacuum to give a pale yellow liquid with low viscosity (referred as **compound 2**: 3-carboxymethyl-1-methylimidazolium bisulfate) and this is the final [CMIM] [HSO₄] IL.

NMR data:

¹H NMR (300 MHz, D₂O): δ = 3.626 (s, 3H, N-CH₃), 4.846 (t, 2H, N-CH₂), 7.207 (s, 2H, CH), 8.493 (s, 1H, CH);

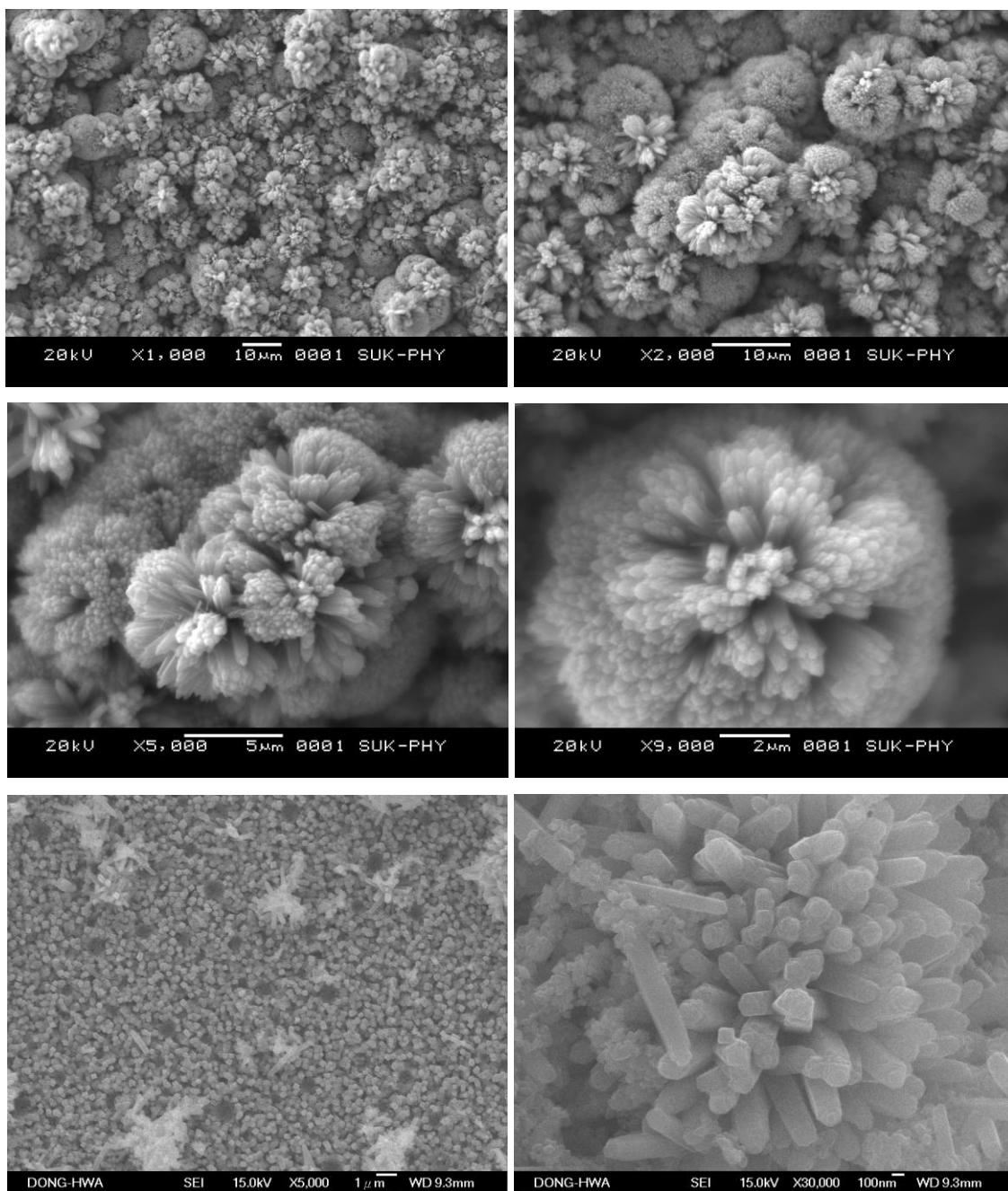
¹³C NMR (d₆ – DMSO): 36.31(CH₃), 50.1 (CH₂), 123.89 (CH), 124.32 (CH), 138.25 (N-CH-N), 168.38 (COOH)



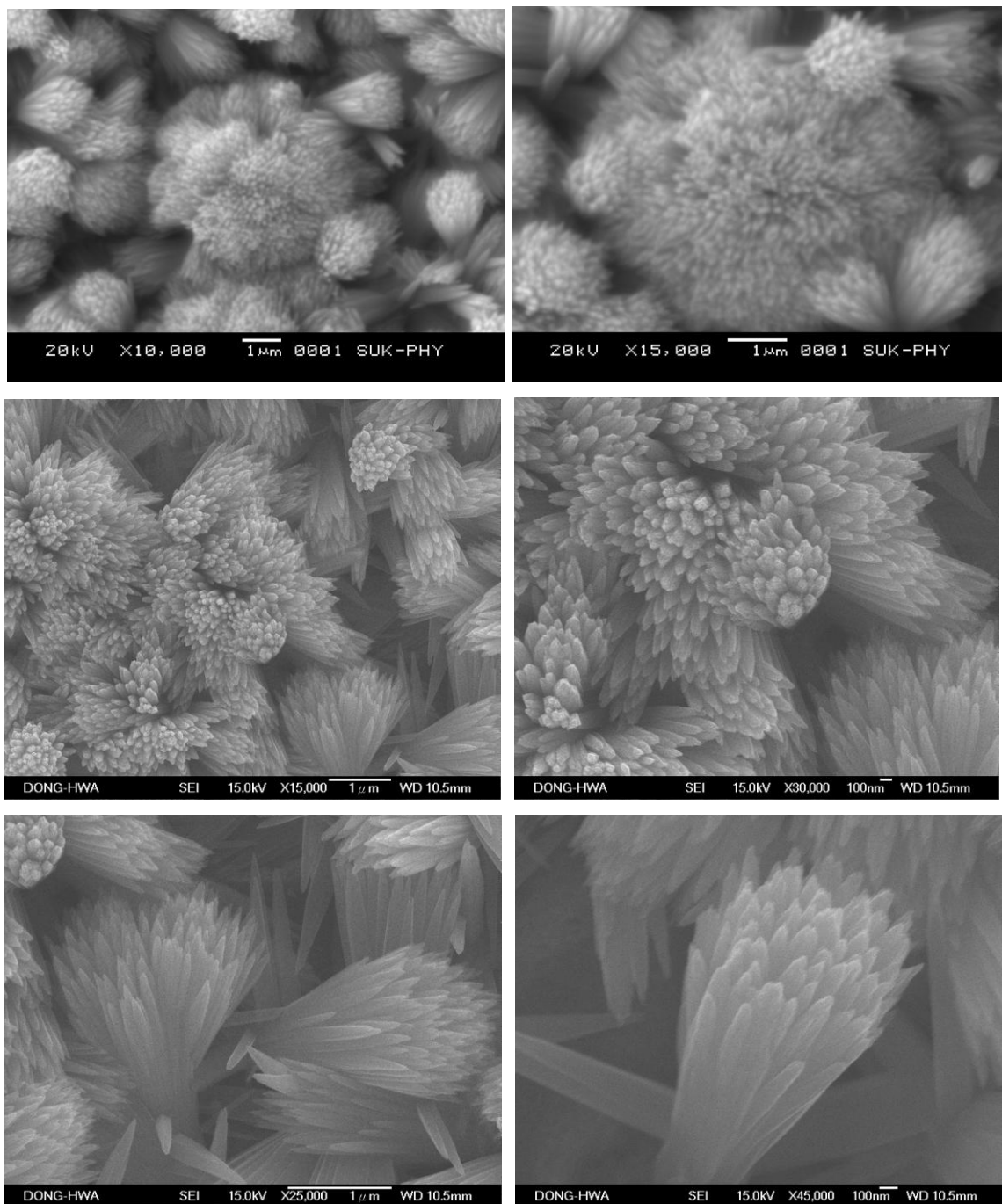
Reference:

- [1] J.S. Shaikh, R.C. Pawar, R.S. Devan, Y.R. Ma, P.P. Salvi, S.S. Kolekar, P.S. Patil, *Electrochimica Acta* 2011, **56**, 2127.

S2 SEM and FESEM images of TiO₂ nanorods structures at varied magnification.



S3 SEM and FESEM images of TiO₂ nanoflowers structures at varied magnification
 (synthesized in Bronsted Acidic Ionic Liquid)



S4 XRD patterns of as-synthesized TNR and TNF samples

