Catalytically dephosphorylate adenosine monophosphate (AMP) to form supramolecular nanofibers/hydrogels

Supplementary Materials

Experimental

Chemical reagents and solvents were used as received from the commercial sources unless otherwise stated. ^{1}H NMR spectra were obtained on Varian Unity Inova 400, CD on a JASCO J-810 spectrometer, LC-MS on Waters Acouity UPLC with Waters MICROMASS detector, TEM on Morgagni 268 transmission electron microscope.

Preparation of 3

To a suspension of **2** (429 mg, 1.5 mmol) in dioxane (20 mL), compound **1** (304 mg, 0.5 mmol) and triethylamine (1.0 mmol) in water (2 ml) were added. The reaction mixture was stirred at room temperature overnight to give a clear solution, which was evaporated by blowing dry nitrogen. Finally, the residue was purified by column chromatography (silica gel; methanol/methylene chloride) to give **3** (257 mg, 60%). ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.39-8.12(m, 5H), 7.83-7.44(m, 7H), 7.23-7.14(m, 10H), 5.88(s, 1H), 4.58-4.49(m, 3H), 4.19-4.13(m, 3H), 3.96(s, 1H), 2.98(m, 2H), 2.85-2.69(m, 6H), 1.75-1.36(m, 6H).

Preparation of 4

In 0.7 mL of trimethylphosphate, 0.1 mmol of **3** was suspended at 0^{o}C. Phosphorus oxychloride (0.3 mmol) was added and the suspension was kept stirring at 0 ^{o}C for 8 h. When the suspension became a clear solution, water was added to hydrolyze the 5'-phosphoryl chloride to 5'-monphosphate. The solution was then neutralized by saturated ammonium carbonate at 0 ^{o}C. The crude products were purified by semipreparative HPLC. ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.25-8.12(m, 5H), 7.78-7.39(m, 7H), 7.17-6.90(m, 10H), 5.86(s, 1H), 4.53-4.45(m, 3H), 4.12-4.01(m, 3H), 3.88(s, 1H), 2.96(s, 2H), 2.76(m, 2H), 2.70-2.61(m, 6H), 1.70-1.31(m, 6H).

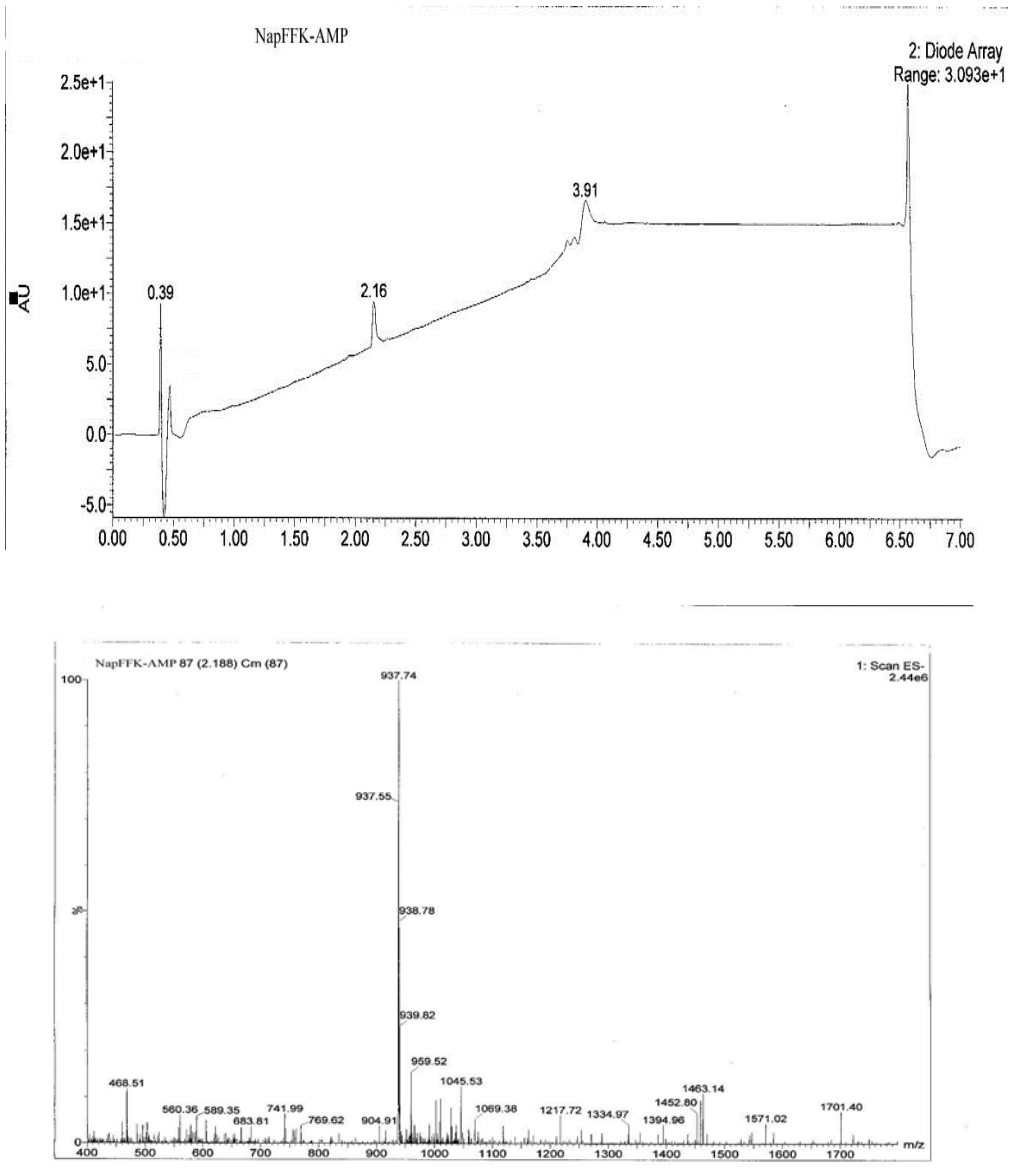

Figure 1. LC-MS of **4**. $[M-1]^-$ showed the peak of 937.74

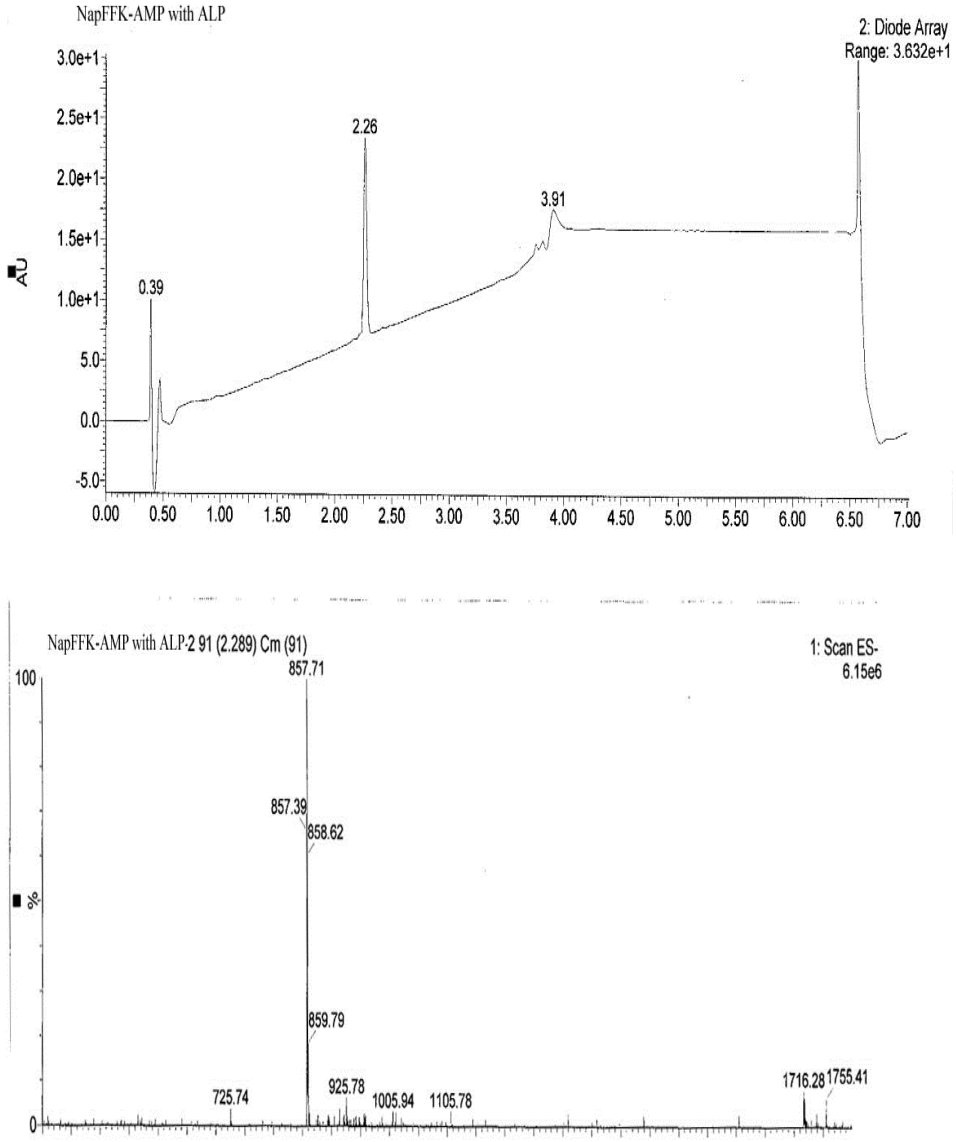

Figure 2. LC-MS of the **4** treated with ALP. The major peak is at 857.71 $[M-1]^-$. The result indicated that **4** was converted to 3.