Supplementary Material (ESI) for Journal of Materials Chemistry

Supplementary Information

Conjugating Methotrexate to Magnetite (Fe_3O_4) Nanoparticles via Trichloro-s-Triazine

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Synthesis of Fe_3O_4 nanoparticles

All reagents were purchased from Sigma-Aldrich and all solvents were purchased from Mallinckrodt Chemicals (Phillipsburg, NJ) unless otherwise noted. 10 ml of benzyl ether, 10 ml of oleylamine, and 0.7064 g (2mmol) iron(II) acetylacetonate ($Fe(acac)_3$) were mixed in a round bottom flask at room temperature. A magnetic stir bar was used was used to stir the solution throughout the reaction. The solution was heated using a heating mantle under nitrogen flow at 120°C for 2 hours and then at 240°C for 1 hour. The nitrogen flow was then reduced to a "nitrogen blanket" and the solution was heated for 1 hour at 300°C. The resulting nanoparticles were dispersed in hexane and washed several times by adding ethanol to the reaction mixture (with a 2:1 ethanol: sample ratio) and centrifuging (Beckman Coulter Allegra 64R Centrifuge) for 5 minutes at 8000 RPM. The nanoparticles (NPs) were stored in hexane.

Synthesis of TsT-PEG-TsT precursor

0.55g of TsT, 3.0 g of 6000 molecular weight PEG (Fluka Chemie, Germany), 0.5 g sodium carbonate (Fisher Scientific, Fair Lawn, NJ), and 100 ml of benzene were added to a round bottom flask and stirred overnight with a magnetic stir bar. The reaction mixture was separated into centrifuge tubes and washed by adding petroleum ether to the reaction mixture (with a 2:1 ether: sample ratio) and centrifuging for 10 minutes at 8000 RPM. After several washings, the solid white material was dried in an oven and stored in the refrigerator.

Conjugation of methotrexate to Fe_3O_4 nanoparticles

2 mg of methotrexate (L-Amethopterin hydrate, 98%) was dissolved in 250 μl of dimethyl sulfoxide (DMSO) and mixed with an 50 μl aqueous solution containing 3 mg of 1-ethyl-3-(3-dimethylaminopropyl)carboiimide (EDAC) and 1 mg of N-hydroxysuccinimide (NHS). After 10 minutes the MTX/EDAC/NHS mixture was added to the NH_2- terminated Fe_3O_4 nanoparticles and stirred overnight under aluminum foil to avoid light exposure.

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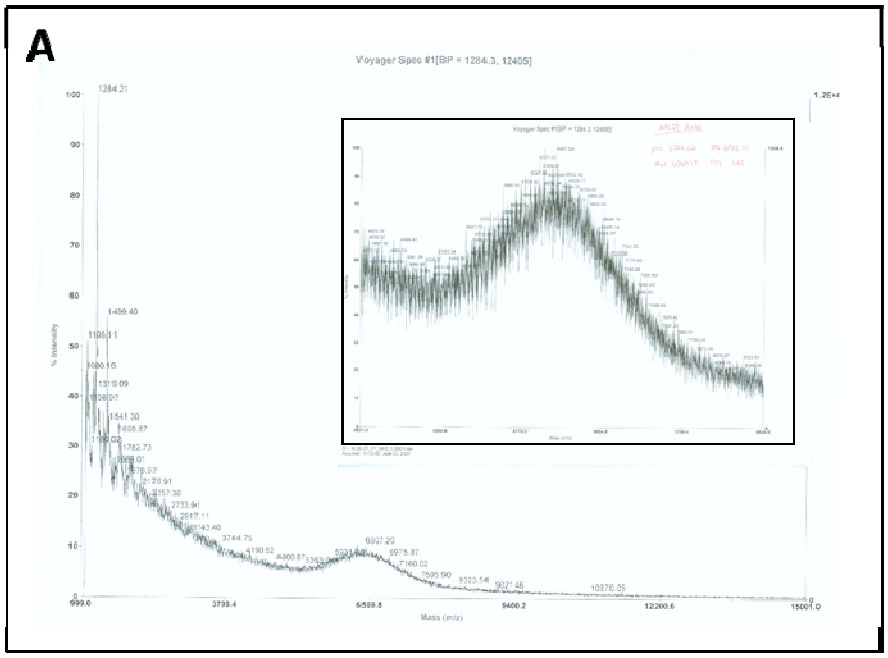

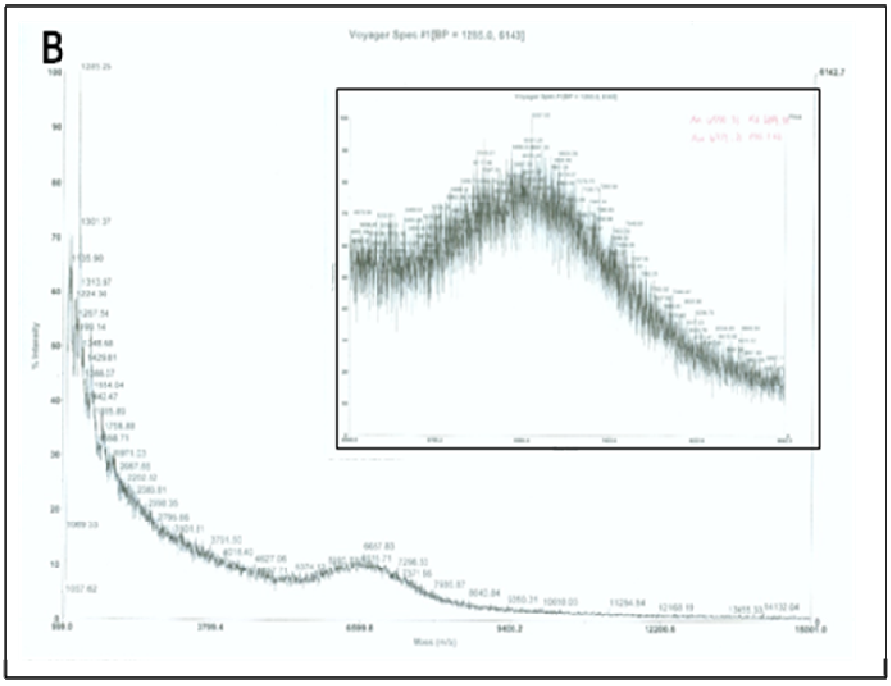

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Fig. S1 MALDI mass spectrometry data for NH_2-terminated NPs (A) and MTX-conjugated NPs (B). The average molecular weight for NH_2-terminated NPs is 6224. The average molecular weight for MTX-conjugated NPs is 6590.