

New Journal of Chemistry

Supporting Information

One-step esterification of nanocellulose in Brønsted acid Ionic Liquid for delivery to Glioblastoma Cancer cells.

L. Cellante,^{a†} R. Costa,^{b†} I. Monaco,^a G. Cenacchi,^b E. Locatelli^{a*}

a. Department of Industrial Chemistry "Toso Montanari", Viale Risorgimento 4, 40136, Bologna, Italy.

b. Department of Biomedical and Neuromotor Sciences – DIBINEM, via Massarenti 9, 40138, Bologna, Italy.

Preparation of N-methylpyrrolidinium hydrogen sulfate [MepyrrH][HSO4]¹

The Bronsted acid ionic liquid was prepared by adding dropwise an equimolar amount of sulfuric acid H₂SO₄ (98%) to the selected 1-Methylpyrrolidine base. The mixture was then stirred for 5 hours at 60 °C. After that, reduced pressure was used to remove water. The result was a pale yellow viscous liquid obtained with quantitative yield.

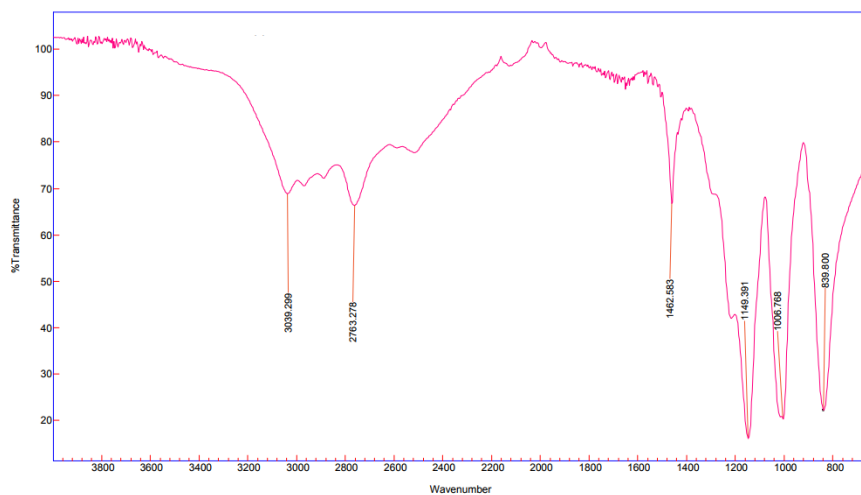


Figure S1. IR-spectra of N-methylpyrrolidinium hydrogen sulfate

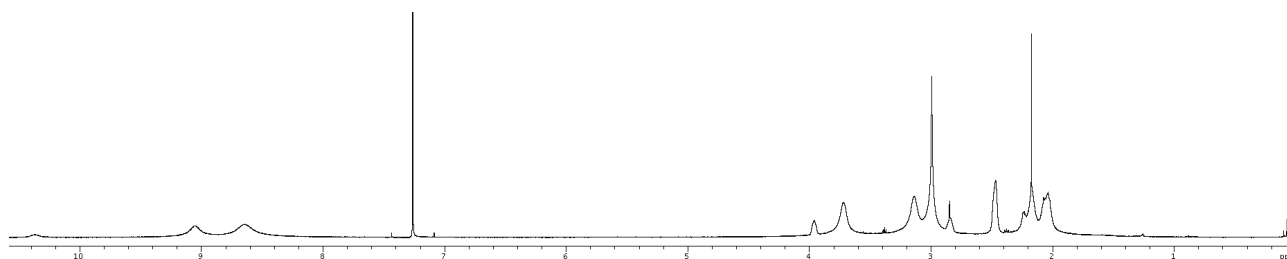


Figure S2: ¹H-NMR of N-methylpyrrolidinium hydrogen sulfate in CDCl₃.

Determination of loaded Cltx-Cy5 onto CNCs.

$^1\text{H-NMR}$ (600 MHz) analysis was performed on washing aqueous solution, dried under vacuum and re-dispersed in D_2O . These showed no signal for Cltx (1.0 ppm, 3.15 ppm, 3.35 ppm), meaning that the conjugation yield can be considered quantitative; other signals can be attributed to ionic liquid remained in the waste.

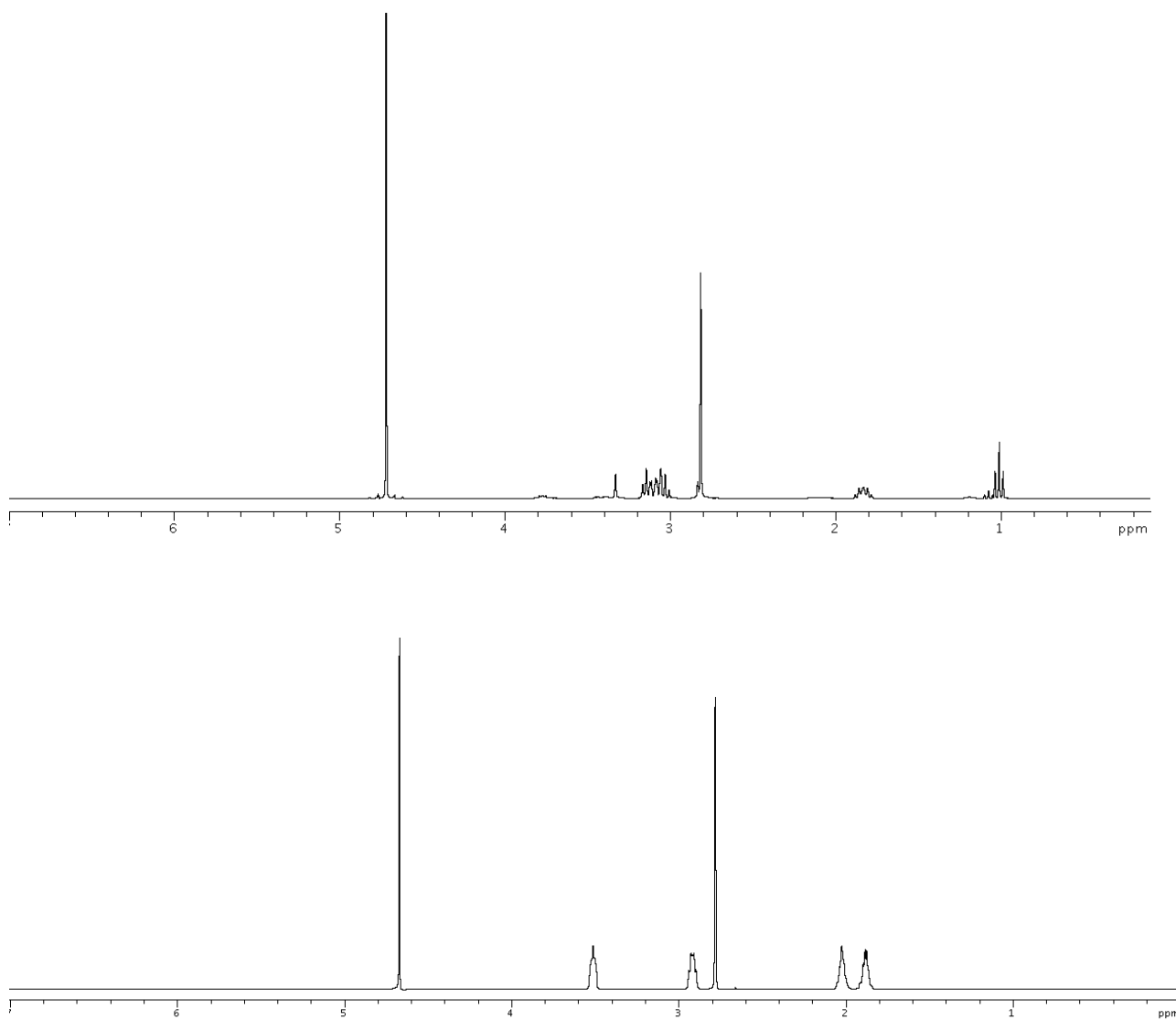


Figure S3: top) $^1\text{H-NMR}$ spectrum of Cltx. bottom) $^1\text{H-NMR}$ spectrum of residual after washings. Reference line for D_2O 4.72 ppm.

References

$^1\text{Chiappe C, Rajamani S, D'Andrea F. A dramatic effect of the ionic liquid structure in esterification reaction in protic ionic media. *Green Chem.*, 2013,15, 137-143$