4th EPNOE International Polysaccharide Conference
POLYSACCHARIDES AND POLYSACCHARIDE-BASED ADVANCED MATERIALS: FROM SCIENCE TO INDUSTRY
October 2015, Warsaw, Poland

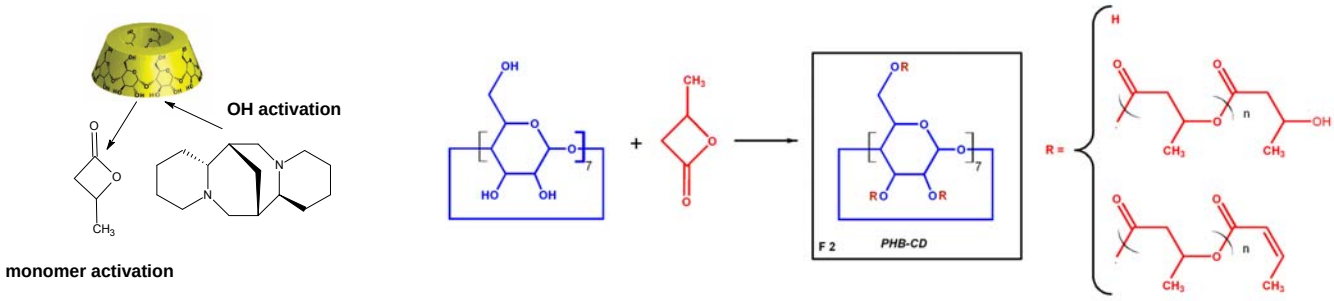
ELECTROSPRAY MASS SPECTROMETRY - INDISPENSABLE TOOL FOR THE EVALUATION OF MODIFIED CYCLODEXTRINS

***Cristian Peptu*, Razvan Rotaru, Valeria Harabagiu**

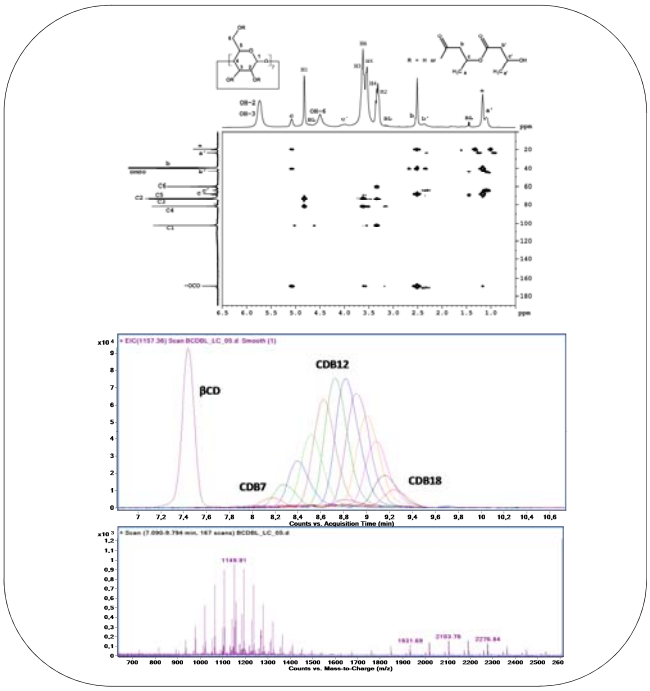
"Petru Poni" Institute of Macromolecular Chemistry, Aleea Grigore Ghica Voda 41A, 700487 Iasi, Romania;

Introduction

Cyclodextrins (CD) ability to form inclusion complexes and hence to alter the physical-chemical properties of guest molecules has been exploited in order to increase the water mixing properties of hydrophobic drugs. To optimize the physical inclusion of specific guests, the polarity of native CD molecules should be adjusted. Generally this is achieved by attaching small alkyl or hydroxyalkyl substituents through ether linkages. Herein, we present the possibility to modify the CD through a simple, one pot reaction, ROP of cyclic esters initiated and catalyzed by CD itself [1-3]. The structural characterization of the obtained products is not a simple task because ***NMR can't provide the average number of monomers connected to a single CD molecule****. On the other hand, mass spectrometry is known to be an easy and fast method to achieve cyclodextrin derivatives characterization. In fact, ESI mass spectrometry has been used to characterize CD derivatives obtained through ring opening oligomerization of β-butyrolactone [1,2], D,L-lactide [3], and other cyclic esters [4].However,* ***ESI MS alone can't provide the average length of a single chain. Herein we show that a combination of both methods, NMR and LC ESI MS, are necessary to achieve structural characterization of CD star polymers.***

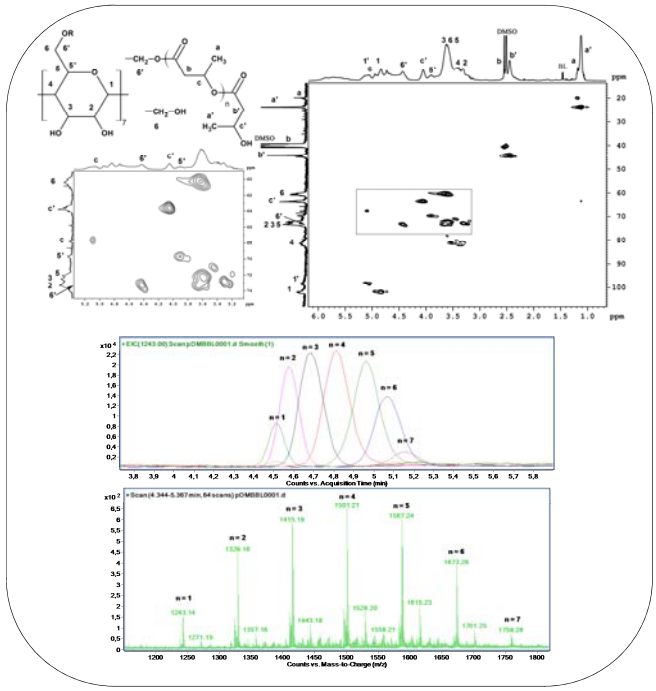


Bulk polymerization



- *Average length of the PHB chains ~* ***3*** *monomer units by NMR and* ***12*** *by LC ESI MS*
- *Cyclodextrins substituted with ~* ***4*** *PHB chains having in average* ***3*** *monomer units*

Solution polymerization



- *Average length of the PHB chains ~* ***1.2*** *monomer units by NMR and* ***4*** *by LC ESI MS*
- *Cyclodextrins substituted with ~* ***4*** *PHB chains having in average* ***1*** *monomer unit*

References
1. Peptu, C., Nicolescu, A., Peptu, C.A., Harabagiu, V. , Simionescu, B.C., Kowalczuk, M. Journal of Polymer Science, Part A: Polymer Chemistry Volume 48, Issue 23,2010, Pages 5581-5592;
2. Peptu, C. , Kwiecień, I. , Harabagiu, V.Cellulose Chemistry and TechnologyVolume 48, Issue 1-2, 2014, Pages 1-10;
3. Shen, J., Hao, A., Du, G., Zhang, H., Sun, H. Carbohydrate Research Volume 343, Issue 15, 2008, Pages 2517-2522;
4. Miao, Y., Zinck, P. Polymer Chemistry Volume 3, Issue 5, 2012, Pages 1119-1122;
5. Peptu, C., Harabagiu, V. Digest Journal of Nanomaterials and Biostructures Volume 8, Issue 4, 2013, Pages 1551-1561.

Acknowledgment. This research has been funded by Romanian MEN-UEFISCDI Program Partnership in priority areas PNII-PT-PCCA-2013, **project nr. 276/2014** .