

Automated Synthesis of Dendritic Core-Shell Architectures with Click Chemistry

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General

Dendrimers are highly ordered, regularly branched globular polymeric molecules composed of multiple monomers that grow radially from a central core to the periphery, like a tree. Their good solubility in various solvents, their high density of functional groups in their periphery and their defined structure render them attractive for many applications, like homogenous complexation and dissolution of guest molecules, light harvesting structures, catalysis, ...

Major drawbacks of those structures are the tedious multistep synthesis, low yields and the problematic purification especially of higher generation dendrimers.

Therefore, hyperbranched polymers (the product of a non-iterative polymerisation therefore possessing irregular architectures with incompletely reacted branched points in their structure) have been developed. Among them, polyglycerols have been found to be ideal candidates for the design of core-shell architectures which present a high potential for host-guest chemistry.

Objective

The objective of the work described herein, is the automated synthesis of a library of different core-shell polymers on an Chemspeed automated Synthesizer, using Click Chemistry (obtention of macromolecules by condensation reactions of small subunits that are connected by carbon-heteroatom bonds).

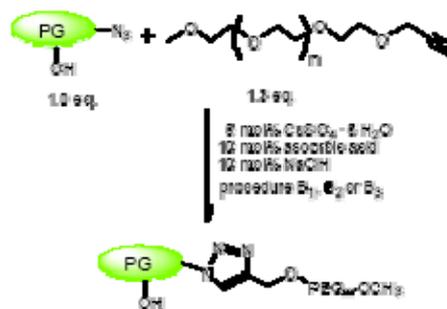
Experimental Set-up



Figure 1: 4x100ml reactor array

The synthesizer is operated with 4x100 mL reactor arrays, filled with Polyglycol azide (PG_x(N₃)_y) in THF. A range of different poly(ethyleneglycol) alkynes (PEG_m-Alk) were added as well as CuSO₄·5H₂O, ascorbic acid, and NaOH solutions. The heating conditions were different in 3 different runs, depending on the procedure:

- Procedure B1: 5 min at 25°C, 720 min at 35°C and 20 min at 20 °C.
- Procedure B2: 10 min at 35°C, 60 min at 45°C, 720 min at 40°C and 300 min at 25°C.
- Procedure B3: 10 min at 35°C, 60 min at 45°C, 900 min at 25°C.



Variations: use of PG₅₀₀₀ and PG₁₀₀₀₀ units
n=6, 10, 14, 22, 43
loading of the PG unit with triazole-mPEG: 0.4, 0.6, 0.5, 0.7 and 0.95

Figure 2: reaction scheme

After the reaction, the solvent was automatically evaporated, and after the addition of methanol, the precipitated copper sulfate, ascorbic acid, and NaOH were removed by online filtration.

Results

Core-shell structures with yields from 26 to 93 % were obtained.

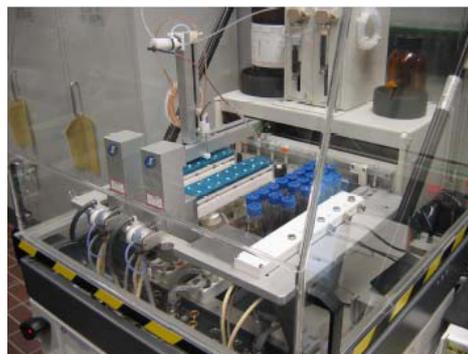
They have been tested for encapsulation and transport capacities of 2 model molecules: Congo red and Rose Bengal. All of them show good encapsulating properties of both dyes. They have also shown to be stable against filtration processes and they can survive a storage for minimum 3 months at room temperature without light.

Summary & Conclusion

Chemspeed's Synthesizer has successfully been used to synthesize a library of core-shell polymers with a variety in the nature of the polyglycerol, the length of the PEG chain, the loading of the PG unit, and the temperature conditions. The one-step click-chemistry is performed in arrays of 4x100 mL reactors, allowing automated liquid handling, shaking, heating, evaporation and filtration of the samples.

Polymers are obtained with up to 93% yield.

Encapsulation and transport tests of the core-shell structures have shown that they were functional and presented interesting capabilities in those two fields. Further investigation are under progress to extend the use of these polymers to other molecules of interest.



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