Physical Characterization of \textit{cis} and \textit{trans} nanostructured diaminophenyl silsesquioxanes and their mixtures

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\section*{Introduction}

The objective of this work was to achieve the separation of a system of 2 and 4 stereoisomers by high-performance liquid chromatography (HPLC), and to propose the separation mechanism in the column.

- Double Deck Polycyclic Oligomeric Silsesquioxanes (DDSO) (Fig 1) increase the chemical resistance, the flame temperature, and thermal and mechanical properties when were included in polymer chains.
- After the synthesis of the DDSQ with a meta or para phenyl amine (PhA) reagent, cis and trans isomers are obtained. Each isomer has different properties such as solubility limits, or melting point.
- The polarity of each molecule could be slightly different based in the positions of the amino groups opening a gate to the separation by liquid chromatography of the DDSQ PhA isomers.

\section*{Materials and methods}

\begin{itemize}
  \item \textbf{Synthesis}
    \begin{itemize}
      \item Developed by the following reaction path. (Schoen, 2013)
    \end{itemize}
  \item \textbf{Preparatory liquid column chromatography}
    \begin{itemize}
      \item For the chromatography techniques, the samples were diluted in CH$_2$Cl$_2$ and gentle injected over the Si support
      \item Dichloromethane as mobile phase (mp)
      \item Si as stationary phase (sp)
      \item Particle diameter (sp) = 5.5\mu.m
      \item Average of 80 fractions collected in 5 ml test tubes after the first analyze came out
    \end{itemize}
  \item \textbf{HPLC chromatography}
    \begin{itemize}
      \item The separation of 2 and 4 isomers was performed in partition and adsorption mode
      \item The calibration curves were constructed for each isomer in adsorption mode
    \end{itemize}
  \item System pressured with N$_2$ to reach an average flow rate of 10ml/min
  \item UV-C detection at 254nm after spotting the sample in a TLC plate

\end{itemize}

\section*{Results}

\begin{itemize}
  \item \textbf{NMR-H$^\circ$} after the preparatory liquid column chromatography
    \begin{itemize}
      \item 7 fractions were collected. However the well defined fractions were the numbers 1.3, and 5.
    \end{itemize}
  \item Fraction 1 – Unreacted species
  \item Fraction 3 – Para-trans isomers
  \item Fraction 5 – Para-cis isomers
  \item Partition HPLC separations
    \begin{itemize}
      \item After the adsorption experiments was proposed the adsorption mechanism described in the figure 2. In this, the cis molecules have more probabilities to form hydrogen bonding than the trans molecules.
    \end{itemize}
  \item Adsorption HPLC separations
    \begin{itemize}
      \item After the sample purification by preparatory liquid chromatography the trans isomers were collected first and the cis isomers in second place with high purities as was described previously by Schoen\textsuperscript{1}.
      \item The partition chromatography in normal phase allowed the separation of the para isomers but it didn’t allow the separation of meta isomers.
      \item The Adsorption chromatography allow the separation of meta and para cis and trans isomers. The adsorption mechanism is a Langmuir isotherm.
      \item The proposed adsorption mechanism is mainly driven by hydrogen bonding but other interactions are not discarded.
      \item The polarity index helped to identify the mp and is helpful to increase the resolution between peaks as was shown in the results of the mp modification.
    \end{itemize}

\section*{Conclusions}

- The authors would like to thank to ERC corporation, Huntsville, AL for the support given to this project.

\section*{Acknowledgments}

References