Building Block Series - Polymer Chemistry Research Experience

July 22 (M) – July 26 (F), 2019

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The goal of this research project is to provide the high school students with the opportunities to (1) have hands-on activities of making samples by 3D printing of plastics and (2) characterize and identify plastic materials using cutting-edge FTIR spectroscopy and thermal analysis instrument in polymer center at RPI, (3) promote their understanding on the thermal properties of polymers, such as glass transition and melting temperatures, which will be the main consideration to dictate the usage of polymers in modern life and (4) ultimately enhance their awareness of plastic recycling issues.

Course Instructors: Dr. Chang Y. Ryu, Professor of Chemistry/Director of Polymer Center, RPI

Student Learning Subjects:

1. Students will learn the fundamental knowledge on polymer chemistry. The faculty instructor will provide a few lectures to expand chemistry learning on periodic table, molecule drawing and monomer/polymer concepts.
2. Students will learn **the 3D printing of polymers** by having hands-on activities of making 3D printed polymer parts.
3. Students will learn the basic knowledge on plastic materials, which includes how different types of plastic materials that they used for soda bottles, soda bottle caps, milk jugs, grocery bags, etc.
4. Students will learn the proper use of thermal analysis equipment to identify the glass transition and melting temperatures of plastic materials. Specifically, during the laboratory sessions, students will acquire hands-on experience to operate and obtain data from **Fourier Transform Infra-Red (FTIR) spectroscopy** and **differential scanning calorimetry (DSC)** equipment. Many different types of plastic materials will be analyzed FTIR with DSC under the supervision of undergraduate/graduate students and faculty instructor.
5. Students will contribute to the research group website of Professor Chang Y. Ryu (http://homepages.rpi.edu/~ryuc/research.html). Plastic sample analysis results will be summarized on this website to acknowledge their excellent contributions in the program. The following format will be used to organize their results.
6. "Student's Name, Affiliated School (City, State), Picture of the plastic sample, FTIR and DSC results, his/her conclusion on which polymer materials are being used" will be archived on the website.

Program Schedule Overview:

: Students need to be in polymer center conference room by 9:10 am and the program starts at 9:15 am each day. Lectures and experiment sessions are arranged to complete the project by 4 pm each day.

- Monday, Tuesday and Wednesday: Experiments
- Thursday: Preparing Figures and Research Paper Outline
- Friday: Finishing Paper and Website posting

About 3D Printing of Plastics

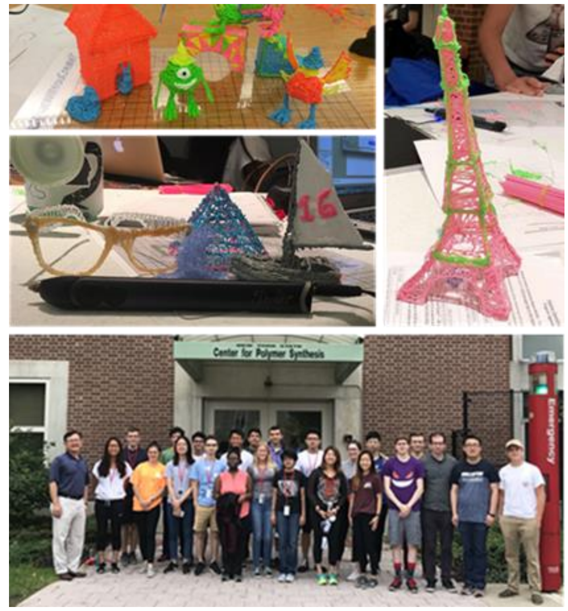
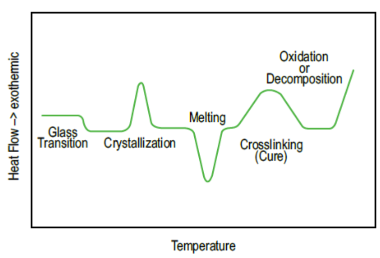


Figure 1. 3D printed plastics made by high school students and group picture from "Research in Polymers" program with TA's and instructors (July, 2018).

The 3D printed polymer technology has the potential to be a disruptive technology in the field of polymeric materials, because it removes a significant technological barrier in making complex polymer parts that cannot be easily made using traditional polymer processing technology.

Each student will use 3D Doodle Pen to create 3D printed plastic objects (**Figure 1**). This is similar to the fused deposition molding (FDM) technique of 3D polymer printing that use the thermoplastics extrusion. Besides that students are making various objects of 3D printed plastics using the 3D Doodle Pens (for learning about FDM), they will also experience the usage of stereolithography (SLA) 3D printing instrument in Prof. Ryu's laboratory. The SLA uses light to polymerize and solidify liquid monomer resins for making 3D printed solid plastics. Photopolymerization will be discussed and hands-on demo will be performed on how we study the reaction in real time using FTIR spectroscopy instrument (aka Real-Time FTIR).

About the Differential Scanning Calorimetry (DSC)



DSC is commonly used in a wide variety of applications to identify melting, crystallization and glass transition temperatures and to study the thermal curing and oxidation behaviors of pharmaceutical compounds, food, liquid crystals and plastics samples. In the general chemistry and/or upper division chemistry laboratory setting, DSC has been also used as a viable alternative method to traditional calorimetry experiments in physical chemistry laboratory module to determine heats of fusion.

DSC is an instrument al thermal analysis technique that measures the heat flow in a sample, subjected to heating, cooling or isothermal temperature programs. A DSC experiment involves (1) placing and sealing a known mass of samples (e.g. 2-3 milligrams) in the DSC sample cell and (2) comparing the heat flow difference between the DSC sample cell and reference cell under the same temperature program (e.g. heating at 10 ^oC/min). The reference cell is prepared by making a DSC cell without containing any samples. Basically, it is an empty DSC cell. Typical DSC data consists of temperature vs. heat flow (or heat capacity) to maintain the same temperature between the sample and reference cells. Finally, researchers prepare a DSC plot to identify the melting (and crystallization) temperatures and the glass transition temperatures of the samples, if there are any.

About DSC in the Polymer Center at RPI

RPI Polymer Center is equipped with a DSC instrument from TA Instruments (Auto Q2000™) having an auto-sampler. (**Figure 2 (a)**). The DSC auto-sampler allows the researchers to load up to 50 sample pans to enable continuous "around-the-clock" sample runs using a robotic unit (**Figure 2 (b)**). This allows the researchers to minimize the instrument down times (sometimes by designing over-night runs) and focus on the DSC data analysis on their samples. Typical temperature window for the sample analysis is -50 °C to 300 °C and the samples are kept under inert nitrogen atmosphere. The typical heating and cooling rates used in the analysis are ± 10 °C/min or ± 20 °C/min. In addition, our DSC is capable of performing much sophisticated thermal analysis called modulated DSC (MDSC), which is applied for characterizing materials with complex transitions and reactions.

(a) (b)

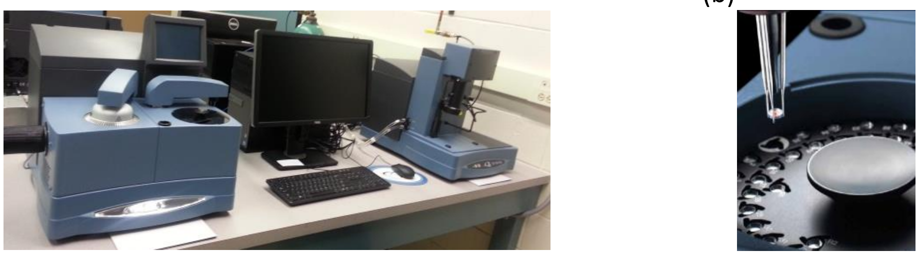
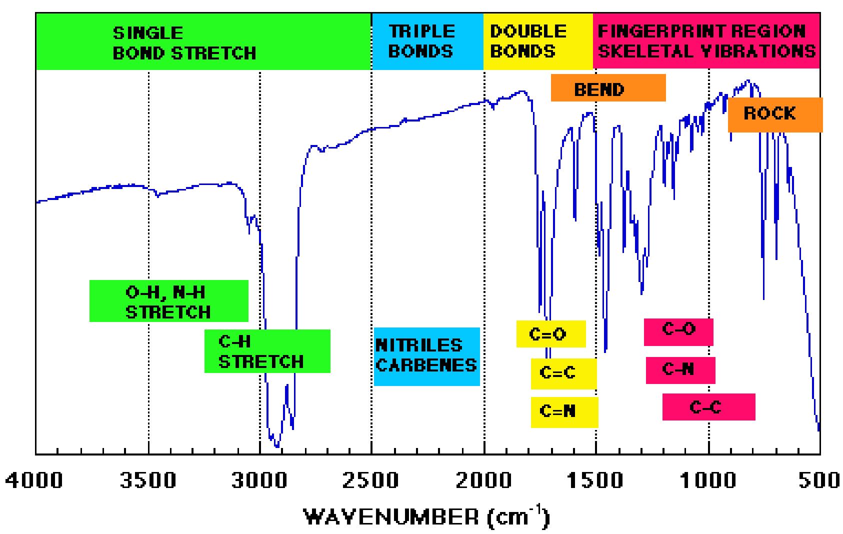


Figure 2. Pictures of (a) Auto Q2000 DSC (left) and (b) DSC auto-sampler in RPI Polymer Center.

About Fourier Transform Infra-Red (FTIR) spectroscopy



FTIR is a spectroscopic method of chemical analysis where IR light spectrum regions are absorbed by different motions (e.g. vibration) of chemical bonds in a molecule. These IR light energy at different wave numbers (wave lengths) are mainly related to vibrational energy of different bonds found with different functional groups in a compound.

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Students will use Thermo-Scientific iS50 FTIR spectrometer in RPI Polymer Center to analyze their polymer samples typically in a form of films. FTIR spectrum will be taken in the wave number range from 4000 to 700 cm^{-1}.

Both transmission mode and ATR (attenuated total reflection) mode will be used to analyze the plastic samples, particularly to address the issue of sample thickness for taking FTIR spectrum.

If time permits, students will perform a series of experiments to measure how fast the UV-initiated polymerization reaction is happening using the Real Time FTIR, under supervision of undergraduate/graduate students and postdoctoral researchers in Professor Ryu's laboratory. This polymer research is related to the SLA 3D Printing of polymers to convert the liquid monomer resins into solid 3D printed plastics. Typical depth of penetration in ATR ranges from about 0.5 μms up to about 5 μm depending up the refractive index of ATR window and sample, and angle of incident IR beam relative to a perpendicular from the surface of the ATR crystal window. The ATR in our lab utilizes diamond as the window crystal and the expected depth length is about 1.7 μm for a sample with a refractive index of 1.5.

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