

**MEMORANDUM**

**DATE: November 11, 2009**

**TO: Laboratory Group E**

**Steven Grant, Kristyl Bently, Michael Burton**

**FROM: Tony Butterfield**

**Engineering Training Supervisor**

**SUBJECT: Batch Polymerization Reaction**

We are looking to develop a model polymerization reaction for our lab. Polyacrylamide happens to be quite important to our research and the work of many others, for its use in creating the hydrogels used in gel electrophoresis. Also, as you will find in the literature, acrylamide’s polymerization is relatively well-understood, and so we will use it as a starting point.

Your task will be to evaluate the use of two very different analytical methods for monitoring the extent of polymerization: 1. falling bead viscometry, and 2. our new FTIR.

You will start off by creating solutions of acrylamide in water at appropriate concentrations. I have done some preliminary work and have samples of fully reacted 5, 6, 8 and 10 wt% polyacrylamide. You will want to take a look at them before you choose appropriate acrylamide concentrations.

You can polymerize acrylamide either photochemically or chemically. In this work we will polymerize chemically, using ammonium persulfate (APS) as an initiator. APS acts as a source of free radicals and, typically, a stock solution of 10 wt% APS is used at a concentration of 0.5 vol% (APS will begin to degrade once it is in water and so it is necessary to freeze the stock solution between use). Once you make up your acrylamide solution and add the APS, the reaction begins slowly. A catalyst, Tetramethylethylenediamine (TEMED), must be used to accelerate the reaction to a reasonable pace. TEMED is typically added at 0.05 vol%, and, once it is added and mixed in, an elevated temperature from the reaction should be noticeable within minutes. For near a complete polymerization, time on the order of an hour or two is needed. Also, keep in mind that the reaction is inhibited by O2. You should remove the oxygen from your acrylamide solutions before use (the O2 in the small APS and TEMED volumes can be assumed to be negligible).

We are only interested in reactions which occur at room temperature. Keep in mind, though, that this reaction is exothermic and you may not have precise control of the average temperature over the time of reaction. You should monitor the temperature and may use it as an indication of reaction rate, but you are not required to control that factor tightly. When acrylamide is used in our lab, we generally just let it react at (and wander above) room temperature anyway.

We are primarily concerned with the effect of initiator concentration on the polymer chain length, and, in turn, viscosity. Please quantify and explain the effect of APS and/or TEMED concentration on chain length and solution viscosity using falling bead viscometry. Determining the effect of initial monomer concentration is not required for this project, but you may take that on if you are feeling ambitious.

Lastly, please determine if the new FTIR can be used to track the rate of polymerization and confirm your falling bead viscometry results.

I will provide you with the acrylamide, APS, TEMED, and the beads. Come to our preliminary lab meeting prepared to discuss possible experimental design, as well as the theory used to inform your decisions.

**IMPORTANT:** While polyacrylamide is reasonably benign, the monomer, TEMED, and APS can be quite dangerous to human health. **All work with these substances *must* be conducted in the fume hood***.* Proper gloves, eye protection and lab coats must be worn at all times. Do not exceed 25 wt% acrylamide monomer, 2.0 vol% of 10 wt% APS, or 0.2 vol% TEMED; the solutions can become too hot if the reaction progresses too quickly. Even after polymerization, your solutions may contain residual acrylamide monomer in need of proper disposal. Please come to our preliminary lab meeting prepared to discuss the related safety issues, and discuss each experimental design with me before you proceed.

Thank you and I’ll look forward to meeting with you on or before Wednesday, November 18.