**Modeling stress relaxation of crosslinked polymer networks for biomaterials
applications: A distance learning module**

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Abstract

Distance learning has become a prevalent method of delivering education to all types of student demographics. Here we present an online materials-related activity, which can be implemented into any course (traditional or online) related to chemical, mechanical, or biomedical engineering. Students use a Matlab graphical user interface (GUI) to predict the stress relaxation behavior of crosslinked biomaterials with three viscoelastic models, the Maxwell, Kelvin-Voigt, and standard linear. By analyzing provided experimental data, students evaluate the impact of crosslinking on the stress-relaxation behavior and learn the underlying molecular mechanisms that cause this behavior. Furthermore, students learn the accuracy of each model prediction by comparison to the experiment data. Overall, the activity highlights the importance of structure-property relationships in the technological advancement of biomedical materials. Our evaluation indicates that the laboratory was effectively conducted online, as it contributed to outcomes set forth for undergraduate Chemical Engineering students by Rowan University and ABET.

Keywords

Biomaterials education, polymers, stress relaxation, viscoelastic models

1. INTRODUCTION

1.1 Pedagogical background

As more colleges and universities begin to offer distance learning formats to their students, it is important to gauge the learning effectiveness of the online format, especially in engineering, a field where students will be expected to apply theory and methodologies in a hands-on design environment. We have developed a materials-related activity that can be integrated to any online course related to chemical, mechanical, or biomedical engineering. In recent years, educational institutions have made increasing use of the internet for delivery of materials education. For instance, MIT developed a 144-credit online program in materials science and engineering (Roylance, 2004). In a reported computer-based activity, students use a Matlab graphical user interface (GUI) to characterize the mechanical behavior of a library of deformable biomaterials (Singh, 2014). In this paper, we report a module where students use a Matlab GUI to analyze the stress-relaxation behavior of crosslinked poly(vinylalcohol) hydrogels using three different viscoelastic models, the Maxwell, Kelvin-Voigt, and standard linear to study. The accuracy of each model prediction is determined by comparison to easy-to-obtain experimental data. Thus, the lab is adaptable to the face-to-face environment, as well. Students relate the results of the lab activity to a biomedical application for the hydrogels, repair of the torn meniscus. The learning objectives are to:

- Describe the manner in which stress relaxation measurements are conducted.
- Describe the underlying molecular mechanisms of stress relaxation in polymers.
- Quantify the impact of crosslinking on the stress-relaxation behavior of a polymer network and describe the underlying molecular mechanisms that cause this behavior.
- To use a mathematical package to fit experimental data to predictive models for viscoelastic behavior.
- Describe key relationships between material science and technological advancement in the field of biomaterials.

We have implemented the activity into an entirely online course entitled *Chemical Engineering Materials*, a junior level requirement at Rowan University. We demonstrate the effectiveness of the learning activity using pre- and post-tests and student surveys.

1.2 Meniscal Tears

The menisci are paired crescent-shaped pads of fibrocartilage in the knees. Knee meniscus injuries are quite common, often resulting from car accidents or sports injuries. Since the blood supply to the meniscus is limited, especially in its central zone, it is susceptible to permanent damage when trauma or degeneration occurs (Arnoczky, 1982; Masouros, 2008). Clinical treatment of meniscus injury is now trending towards replacing, rather than removing or attempting to repair, the injured tissue (Abrams, 2013). It is thought that complete replacement will reduce the risk of patients developing osteoarthritis (Rongen, 2014). The aims of using meniscus replacement are to relieve the symptoms of injury, restore biomechanical function to the knee joint, and prevent development of osteoarthritis in the long term.

1.3 Polymers for Meniscus Replacement

Numerous materials have been investigated for meniscus replacement, most of them polymers. Polymers are used because of their ability to be crosslinked, which imparts resiliency and

rubber-like properties to the materials. The crosslinking also helps to prevent the polymer chains from sliding past one another during deformation, which allows for better dimensional recovery when loads are released (Rongen, 2014). When evaluating polymers as tissue replacements, several properties must be characterized, including biocompatibility, biodegradability, and mechanical properties. The mechanical properties most often studied are compressive, tensile, creep, stress relaxation and fatigue. It is important to have knowledge of the time-dependent behavior of polymers for the development of adequate tissue substitutes. For instance, a stress relaxation test will examine the behavior of a material over time under a constant strain.

1.4 Hydrogels

Hydrogels are special class of polymers. In June 2007, the Society for Biomaterials declared hydrogels “Biomaterial of the Month”. Hydrogels are three-dimensional, water-swollen structures composed of mainly hydrophilic polymers. These materials are for the most part insoluble due to the presence of crosslinks. Hydrogels are superabsorbent (they can contain over 99% water) natural or synthetic polymers. They possess also a degree of flexibility very similar to natural tissue, due to the significant water content.

1.5 Poly(vinylalcohol) Hydrogels as Meniscus Replacements

A candidate material for an artificial meniscus was developed using poly(vinylalcohol) hydrogels (Kobayashi, 2005). Preparation of PVA hydrogels is not only simple and safe, but the methods allow for significant tailoring of mechanical properties. Aqueous solutions of PVA may be physically crosslinked by techniques that introduce hydrogen-bonded regions between adjacent polymer chains, which results in elastic hydrogels. The formation of PVA hydrogels by freeze-thaw (FT) cycles has been studied extensively. From these investigations, it has been established that the crosslink density of PVA hydrogels can be affected by the number of freeze-thaw-cycles (Hassan, 2000b, a; Merrill, 1977). Specifically, using a higher number of freeze-thaw cycles will increase the degree of crosslinking of the hydrogel that is formed.

2. THEORY

2.1 Viscoelastic Properties of Polymers

For perfectly elastic materials, the stress, σ and strain ϵ are related linearly through the modulus, E (Equation 1).

$$\sigma = E\epsilon \quad \text{Equation 1}$$

Elastic behavior is represented by a spring (**Figure 1A**). For an elastic solid, instantaneously increasing the stress to a constant value will cause the solid to deform to a fixed strain, which does not vary with time. If the stress is removed, the material instantly recovers its original shape.

On the other hand, Newton's Law describes the behavior of viscous materials, like silly putty. The stress is linearly related to the strain rate $d\epsilon/dt$ through the viscosity, η , (Equation 2). According to this equation, the stress needed to deform a material depends on how fast the load is being applied. Higher rates of deformation require higher stresses. In addition, at a constant

stress, the strain rate becomes constant. Once unloaded, the material has no tendency to return to its original shape. Viscous behavior is represented by a dashpot (**Figure 1B**).

$$\sigma = \eta \left(\frac{d\varepsilon}{dt} \right) = \eta \dot{\varepsilon} \quad \text{Equation 2}$$

Generally, polymers exhibit viscoelastic mechanical behavior, meaning that they have properties of both viscous and elastic materials when undergoing deformation. On a molecular level, viscoelastic phenomenon is related to the movement of macromolecules in response to external loads. Over long-range time scales, gross rearrangement occurs in the position of the molecules. If the polymer is not crosslinked, chains may slide past one another in response to the load, without returning to their original configuration once the load is released. If the polymer is crosslinked, then chains have more of a tendency to bounce back to their original conformation once the load is released. Over short time scales, both crosslinked and non-crosslinked networks will exhibit small rearrangements in the position of the molecules or the atomic bonds in response to external loads (Wineman, 2000).

In this laboratory activity, we consider the response of a crosslinked polymer to a step increase in strain. The user inputs an instantaneous increase in strain, ε_0 (**Figure 2A**). In response, first the stress instantaneously jumps to a value of σ_0 . Second, the stress needed to maintain the strain ε_0 decreases over time because the chains will rearrange to accommodate the load. The output, a gradual decrease in stress over time (**Figure 2B**), is called stress relaxation (Wineman, 2000).

2.2 Viscoelastic models for stress relaxation

Elastic materials, like a spring, will show no stress relaxation over time, whereas viscous fluids, like a dashpot, show instantaneous relaxation. A viscoelastic response from a polymer is a combination of these two mechanisms, and thus can be predicted by different spring and dashpot combinations. To this end, various viscoelastic models have been developed to mathematically predict the drop in stress that occurs during stress relaxation. Three common viscoelastic models that students studied in this activity were the Maxwell, Kelvin-Voigt, and the Standard Linear.

2.2.1 Maxwell model

The Maxwell model is the simplest model for predicting stress relaxation behavior. It uses a spring with modulus E and a dashpot with viscosity η coupled in series (**Figure 3**) to represent a viscoelastic material (Singh, 2014; Wineman, 2000). The spring and dashpot are being stretched, producing a strain ε_{E1} in the spring and $\varepsilon_{\eta1}$ in the dashpot due to the external stress σ . The total strain on the system ε can be expressed as Equation 3.

$$\varepsilon = \varepsilon_{E1} + \varepsilon_{\eta1} \quad \text{Equation 3}$$

The time derivative of the constitutive relationship for the spring can be written as Equation 4.

$$\dot{\sigma} = E \dot{\varepsilon}_{E1} \quad \text{Equation 4}$$

Substituting Equations 2 and 4 into the time derivative of equation 3 yields Equation 5, a first order differential equation derived from the Maxwell model for predicting viscoelastic behavior.

196 $\dot{\varepsilon} = \frac{\dot{\sigma}}{E} + \frac{1}{\eta} \sigma$ Equation 5

197 The differential equation can be solved to predict stress relaxation behavior, where a material is
 198 subjected to a constant strain ε_0 between time 0 and t. Substituting $\dot{\varepsilon} = 0$ into Equation 5
 199 (Singh, 2014), we then have

200 $0 = \frac{\dot{\sigma}}{E} + \frac{1}{\eta} \sigma$ Equation 6

201 Rearranging Equation 6, solving and integrating yields Equation 7

202 $\sigma(t) = \sigma_0 e^{\left(-\frac{E}{\eta}t\right)}$ Equation 7

203 where the quantity η/E is also referred to as τ , the stress relaxation time constant. Equation 8
 204 can therefore be re-expressed as

205 $\sigma(t) = \sigma_0 e^{\left(-\frac{t}{\tau}\right)}$ Equation 8

206 Note that at long times (as $t \rightarrow \infty$), the Maxwell model predicts that the stress σ will approach
 207 zero.

208 2.2.2 Kelvin-Voigt Model

209 The Kelvin-Voigt model uses a spring with modulus E and a dashpot with viscosity η coupled in
 210 parallel to represent a viscoelastic material (**Figure 4**) (Singh, 2014; Wineman, 2000). Because
 211 of the parallel configuration, the strain, designated as ε , experienced by the spring is the same
 212 as the strain experienced by the dashpot (Equation 9).

213 $\varepsilon_{spring} = \varepsilon_{dashpot} = \varepsilon$ Equation 9

214 The applied external stress σ (**Figure 4**) necessary to stretch spring and dashpot is the sum of
 215 the stresses for the spring and dashpot.

216 $\sigma = \sigma_{spring} + \sigma_{dashpot}$ Equation 10

217 Substituting Equations 1 and 2 into Equation 10, a differential equation results which predicts
 218 the viscoelastic behavior of a material for the Kelvin-Voigt model.

219 $\sigma = E\varepsilon + \eta\dot{\varepsilon}$ Equation 11

220 For stress relaxation experiment, a constant strain ε_0 is imposed, thus $\dot{\varepsilon} = 0$. Equation 11 then
 221 becomes

222 $\sigma(t) = E\varepsilon_0$ Equation 12

223 Upon examination of Equation 12, it becomes apparent that the Kelvin-Voigt models the stress
 224 as a constant. In other words, it does not predict stress relaxation.

225 1.6.3 Standard Linear model

226 In order to capture the response more accurately, the standard linear model is used, which
227 essentially combines the Maxwell and Kelvin-Voigt models. It consists of a spring placed in
228 series with the Kelvin-Voigt configuration (**Figure 5**) (Singh, 2014; Wineman, 2000).

229 The total stress σ on the Kelvin-Voigt element, shown in Equation 13 is the sum of the stresses
230 on each element.

231
$$\sigma = \sigma_1 + \sigma_2$$
 Equation 13

232 The total strain on the model can be expressed with Equation 14.

233
$$\varepsilon = \varepsilon_1 + \varepsilon_2$$
 Equation 14

234 Taking the time derivative of Equation 15 we have

235
$$\dot{\varepsilon} = \dot{\varepsilon}_1 + \dot{\varepsilon}_2$$
 Equation 15

236 Note that the time derivative of the stress strain relationship for spring E_1 is

237
$$\dot{\sigma} = E_1 \dot{\varepsilon}_1$$
 Equation 16

238 and the dashpot behavior is represented by Newton's Law.

239
$$\sigma_2 = \eta \dot{\varepsilon}_2$$
 Equation 17

240 Substituting Equations 16 and 17 into Equation 15, we obtain

241
$$\dot{\varepsilon} = \frac{\dot{\sigma}}{E_1} + \frac{\sigma_2}{\eta}$$
 Equation 18

242 Substituting Equation 13 into 18:

243
$$\dot{\varepsilon} = \frac{\dot{\sigma}}{E_1} + \frac{(\sigma - \sigma_1)}{\eta}$$
 Equation 19

244 The spring in the Kelvin-Voigt model can be represented by

245
$$\sigma_1 = E_2 \varepsilon_2$$
 Equation 20

246 Substituting Equation 20 into 19, a differential equation is obtained for the standard linear
247 model.

248
$$\dot{\varepsilon} = \frac{\dot{\sigma}}{E_1} + \frac{\sigma}{\eta} + \frac{E_2 \varepsilon_2}{\eta}$$
 Equation 21

249 For stress relaxation experiment, a constant strain ε_0 is imposed between time 0 and t. Thus $\dot{\varepsilon} =$
250 0 is substituted into Equation 21 and the differential equation is solved, yielding Equation 22
251 (Singh, 2014):

252
$$\sigma(t) = \varepsilon_0 \left[\frac{E_1^2}{E_1 + E_2} e^{-\left(\frac{E_1 + E_2}{\eta}\right)t} + \frac{E_1 E_2}{E_1 + E_2} \right]$$
 Equation 22

253 The following activity, all three models are simulated in a MATLAB graphical user interface

(GUI) to predict the stress relaxation behavior of PVA hydrogels as a function of FT. The accuracy of each model will be determined by comparison to experiment data.

3. MATERIALS AND METHODS

3.1 Generation of Raw Data

Stress relaxation testing was performed on an Instron mechanical testing system (Model 3362, Park Ridge, IL) by the course developers. PVA hydrogels were formed in 96-well plates from 15 wt% aqueous solutions of PVA (molecular weight 89,000, 99%+ hydrolyzed, Sigma Aldrich). Solutions that underwent 2, 4, 6 or 8 freeze-thaw cycles were compressed with an anvil to 15% strain. Load and displacement data were captured by Instron BlueHill 1.2 software and converted to stress-strain values. Students in the online course received raw data (stress versus time) files in “*.csv” format for the PVA hydrogels. Raw data files are included with this publication as supplementary documentation.

3.2 Matlab™ for Data Analysis

Custom-written MATLAB routines for each of the three viscoelastic models were used to process and fit the experimental stress and time data. In each model case, a nonlinear least squares fit routine minimized the root-mean-squared error between the experimental data and the computed results using the stress relaxation formula for each model. A graphical user interface (GUI) was created to facilitate the students' investigation into each model. The GUI files and instructions for use are included with this publication as supplementary documentation. The GUI allows the user to browse for a specific file of raw experimental data in csv format and then plot it on a set of axes within the GUI. Matlab will plot a best fit of the data to each model by manipulating the parameters E or N, which represent the spring modulus E and viscosity η , respectively. In addition, models can also be plotted with values for the parameters E and N that are entered manually. Students are guided through the following data analysis questions:

- Choose any one of the sample data files and plot it on the axis using the GUI interface. Fit the data with the Maxwell, Kelvin-Voigt, and standard linear models. Comment on the suitability of each model.
- Choose any one of the sample data files and fit it to the Maxwell model. What are the values for the model parameters, E and η for the best fit? Comment on how each of the parameters affects the model when varied independently.
- For the Maxwell model, make a histogram plot of E versus number of FT. Now, make a histogram plot of viscosity η versus number of freeze-thaw cycles. What trends are observed in the data? Explain these trends, based on what is known about the effect of crosslinking on polymer structure.
- For the standard linear model, plot 3 histograms: E_1 versus FT, E_2 versus FT, and η versus FT. What trends are observed in the data? Explain these trends, based on what is known about the effect of crosslinking on polymer structure.

4. RESULTS AND DISCUSSION

Plotted in **Figure 6** is experimental data for hydrogels prepared with 2 FT cycles. The best fit for Maxwell, Kelvin-Voigt and Standard Linear are shown on the same plot. The Kelvin-Voigt clearly does not predict stress relaxation, **only a constant stress for step displacement**. The R^2 value for the Maxwell and Standard Linear models are 0.87 and 0.97, respectively, indicating that the standard linear is the most suitable model **of the three**.

Table 1 gives the best fit E and η values for the Maxwell model and the E_1 , E_2 , and η for the standard linear model. For the Maxwell model, changing the value of E will shift the curve up or down the y-axis. Changing the value of η will affect how quickly the curve decays.

Table 1. Best fit values for E and η for the Maxwell model.

	Maxell		Standard Linear		
	E (Pa)	η (Pa.S)	E_1 (Pa)	E_2 (Pa)	η (Pa.S)
2 FT	5.56×10^4	2.04×10^8	5.63×10^4	1.15×10^6	6.03×10^7
4 FT	1.10×10^5	3.22×10^8	1.11×10^5	1.79×10^6	1.15×10^8
6 FT	1.66×10^5	3.35×10^8	1.69×10^5	1.84×10^6	1.28×10^8
8 FT	1.89×10^5	4.37×10^8	1.92×10^5	2.4×10^6	1.66×10^8

Shown in **Figure 7** is a histogram plot of E versus number of FT cycles for the Maxwell model. The trends in the data show increasing modulus E with increasing FT. Increased degree of crosslinking between the chains generates a stronger three-dimensional network, making the modulus value of the polymer increase.

Shown in **Figure 8** is a histogram plot of viscosity η versus number of freeze-thaw cycles for the Maxwell model. The trend in the data shows increases in viscosity η with increasing number of freeze-thaw cycles. Increased degree of association between the chains **limits chain reorientation** (Wineman, 2000), so viscosity will increase.

To evaluate the impact of this experiment on learning, a quiz was administered to students before and after the lab. The quiz was comprised of 10 multiple-choice questions, which were mapped to student outcomes set forth by the Accreditation Board for Engineering and Technology (ABET) for undergraduate chemical engineering students. **ABET is an accrediting agency for programs in applied science, computing, engineering, and engineering technology** (Milligan, 2014). The ABET outcomes relevant to this activity, as well as measurable skills that are associated with each, are summarized in Table 2. **The complete assessment quiz is included in the supplementary documents**. The average scores on the pre- and post-tests were analyzed with a t-test using a 95% confidence interval ($\alpha = 0.05$).

Table 2. Pre- and post-test assessment questions were aligned to ABET and Rowan student outcomes for undergraduate chemical engineering students.

Outcome	Reinforced in assessment quiz questions	Example skills categorized within this outcome:
Student demonstrate an ability to apply knowledge of mathematics, science, and engineering (ABET - A)	1-8, 10	Students will 1) describe the underlying molecular mechanisms of stress relaxation in polymers, 2) use three viscoelastic models to predict the stress-relaxation behavior of crosslinked hydrogels, 3) describe key relationships between materials science and technological advancement in the field of biomaterials.
Students demonstrate the ability to design and conduct experiments as well as to analyze and interpret data (ABET-B)	1,4, 5	Students will 1) demonstrate an understanding of how stress relaxation tests are conducted, 2) analyze data on the effects of crosslinking on the stress-relaxation behavior of a polymer network and 3) describe the underlying molecular mechanisms that cause this behavior.
Students demonstrate a knowledge of contemporary issues relevant to the field of chemical engineering (ABET - J)	7, 9	Students demonstrate an awareness of how the principles of math, science, and engineering are applicable to current medical device technology.

The average score on the pre-test (n=53) and post-test (n=49) was 51% and 80%, respectively (**Figure 9**). The average score on the post-test was significantly higher than that of the pre-test ($p < 0.05$). Between the pre-test and the post-test, the percentage of correct responses increased by 31, 34, and 31% for ABET-A, B, and J, respectively (**Figure 10**).

5. CONCLUSIONS

Students used a Matlab graphical user interface (GUI) to predict the stress relaxation behavior of crosslinked biomaterials with three viscoelastic models, the Maxwell, Kelvin-Voigt, and standard linear. By analyzing the provided experimental data, students evaluate the impact of crosslinking on the stress-relaxation behavior of a polymer network and learn the underlying molecular mechanisms that account for the results. Furthermore, students learn the accuracy of each model prediction by comparison to the experiment data. Overall, the experiment highlights the importance of structure-property relationships in the technological advancement of biomedical materials. Our evaluation indicates that the laboratory was effectively conducted online, as it contributed to outcomes set forth for undergraduate Chemical Engineering students by ABET. This flexible, cost-effective distance-learning module could easily be adapted to face-to-face environment. **The activity can be extended by using the Maxwell, Kelvin-Voigt, and standard linear models to predict creep response by applying constant load, measuring deformation, and then model fitting the stress-strain data. In addition, nonlinear models can also be explored to capture the stress-strain behavior of hydrogels (Wang, 2013).**

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CAPTIONS

Figure 1. Elements of rheological models. (A) A spring, representing elastic behavior and (B) A dashpot, representing viscous behavior.

Figure 2. Summary of a stress relaxation test. (A) The input for a stress relaxation test is a step increase in strain; (B) The output is a gradual decrease in stress over time.

Figure 3. The Maxwell model is developed using a spring and dashpot coupled in series.

Figure 4. Elements for the Kelvin-Voigt model are coupled in parallel.

Figure 5. The standard linear model combines a spring placed in series with the Kelvin-Voigt configuration.

Figure 6. Stress versus time data for hydrogels prepared with 2 FT cycles. The best fit for Maxwell, Kelvin-Voigt and Standard Linear are shown.

Figure 7. The spring modulus E for the Maxwell model as a function of number of FT cycles.

Figure 8. The viscosity η for the Maxwell model as a function of number of FT cycles.

Figure 9. Average score on the pre and post-test ($n=53$ and 49 , respectively). The average score on the post-test was significantly higher than that of the pre-test ($p < 0.05$). Error bars represent the 95% confidence interval.

Figure 10. Percentage of correct responses for each learning outcome as described in Table 1. The percentage includes responses for questions mapped to a particular outcome.

Figure 1

(A)

(B)

Figure 2

(A)

(B)

Figure 3

Maxwell	$\sigma(t) = \sigma_o \exp\left(-\frac{E}{\eta}t\right)$
Kelvin-Voigt	$\sigma(t) = E\varepsilon_o$
Standard Linear	$\sigma(t) = \varepsilon_o \left[\frac{E_1^2}{E_1 + E_2} \exp\left(-\frac{E_1 + E_2}{\eta}t\right) + \frac{E_1 E_2}{E_1 + E_2} \right]$

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

Figure 10