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Compression of Fluid-Filled Polymeric Capsules and Inverse Analysis to Determine Nonlinear Viscoelastic Properties

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Abstract

Polymeric capsules are widely used in the pharmaceutical industry for drug delivery. The mechanical properties of the capsule play an important role in the drug release process. For example, as soon as the capsules are swallowed, they can undergo large deformation due to the interaction with the surrounding tissue environment. Hence, it is necessary to study the mechanical responses of polymeric capsules under such conditions.

This work considers an experiment in which polymeric capsules filled with fluid are compressed between two flat, rigid, parallel plates. The bottom plate is stationary while the displacement of the top plate is controlled. The bottom plate is a transparent prism through which the contact area can be monitored by means of an optical arrangement. The reaction force on the capsule is recorded and the force-displacement-time data is used in an inverse analysis to determine the capsule wall material properties. This inverse analysis is built upon comparing the simulated force-displacement- time response obtained from a finite element (FE) model of the experiment, with the experimental data. Since the capsule is subjected to large relative displacements of the plates, material and geometric non-linearity are incorporated in the finite element model. Also, as observed from experiments, the capsule material exhibits time-dependent characteristics. Therefore, in this study, a nonlinear viscoelastic constitutive relation for modeling the capsule material is used. The enclosed fluid is assumed to be incompressible and in a steady state. Information obtained from the experiment and the inverse analysis is used to determine the nonlinear viscoelastic material parameters for the polymeric capsule enclosing incompressible fluid.

Details of Experiment

Commercially available polymeric capsules enclosing a fluid were compressed between two flat, rigid, parallel plates with the use of an Instron testing machine. Displacement control tests were conducted by specifying the displacement for the top plate while the bottom one remains stationary. After reaching a specified value, the applied displacement of the top plate was kept constant to study the relaxation behavior of the polymeric capsules. The time variation of the capsule's reaction force during the loading and holding processes was measured through a load cell attached to the top plate. The contact area between the capsule and the plates was also recorded by using a clear cube beam splitter as the bottom plate. Images of the contact area (after reflection through the beam splitter) while the capsule was in compression as well as relaxation were captured through this optical arrangement and with the use of a high speed camera.

Contact Area and Force-Displacement Data

Applied displacement input, contact area and force-time data for a typical test on an ellipsoidal capsule are shown in Figure 1a, 1b.

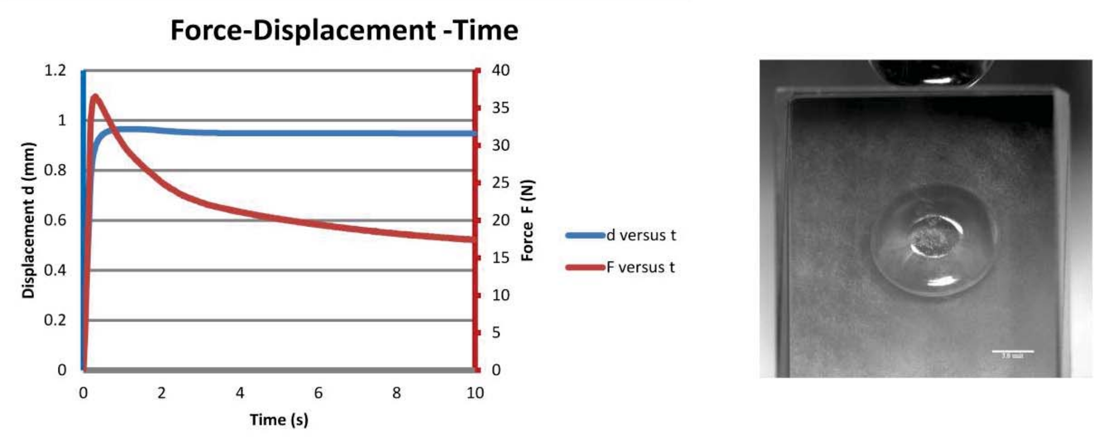

Figure 1a: Applied displacement to the top plate and the force-time response

Figure 1b: Contact area between the capsule and the bottom plate (time t>2s)

Figure 1. Experiment Setup and Output

The contact area is observed to be close to elliptical and increases with increasing deformation during the loading part. The force-time data shows relaxation behavior of the polymer capsule, as expected.

Modeling

The compression and relaxation stages of the experiment are simulated through an FE analysis using the commercial FE code ABAQUS. The polymeric capsule is modeled as a deformable ellipsoidal shell with the major axis dimension of 9.3 mm and minor axis dimension of 7.2 mm. The enclosed fluid is assumed to be incompressible. The thickness of the capsule was determined by slicing the capsule in half, embedding it in epoxy and measuring the wall thickness using an optical microscope. The variation of the thickness is found to be from 0.3 to 0.5 mm. For the simulation here, a thickness of 0.4 mm is used, however, incorporating realistic thickness variations is also being studied. The two contact plates are modeled as rigid bodies. The actual displacement-time history of the top plate from the experiment is used to specify the input for finite element simulations. Reaction forces and contact area are extracted from finite element simulations to compare with the experimental data. Two different constitutive models for the capsule's material are employed in the simulations. The built-in nonlinear viscoelastic model in ABAQUS where the elastic part is described by nonlinear elastic material [1, 2], and the viscoelastic part is described by a Prony series in which the $i-th$ term has material constants g_i and τ_i as the amount of relaxation and relaxation time respectively [3]. The second model is based on the Pipkin-Rogers model [4, 5, 6] and is implemented through a UMAT subroutine. Elasticity and relaxation parameters need to be determined for both models.

Inverse Analysis

To extract unknown material properties described above, an inverse analysis is applied to fit the experimental force-time response. Due to the computational cost of the FE simulation which is a full 3D model including nonlinearity in material and geometry as well as contact features, using a sequential inverse analysis approach is time-consuming. In addition, the need to determine many unknown material parameters adds more difficulty to this approach. To overcome these problems, an inverse analysis approach based on establishing a meta model and a Kriging estimator [7, 8] is utilized. A preliminary fit of the force-time curve using the built-in nonlinear viscoelastic model in ABAQUS is shown in Fig.2. The nonlinear elastic part is described by a Mooney-Rivlin model and the viscoelastic part is captured by a two-term Prony series. Five unknown materials parameters were extracted: two parameters for the elastic part C_1, C_2, and three for the viscoelastic part g_1, τ_1, g_2. For simplicity, the second relaxation time τ_2 is assumed to be $\tau_2 = 10\tau_1$ and the capsule material is assumed to be incompressible. A similar approach will also be applied to the Pipkin-Rogers model.

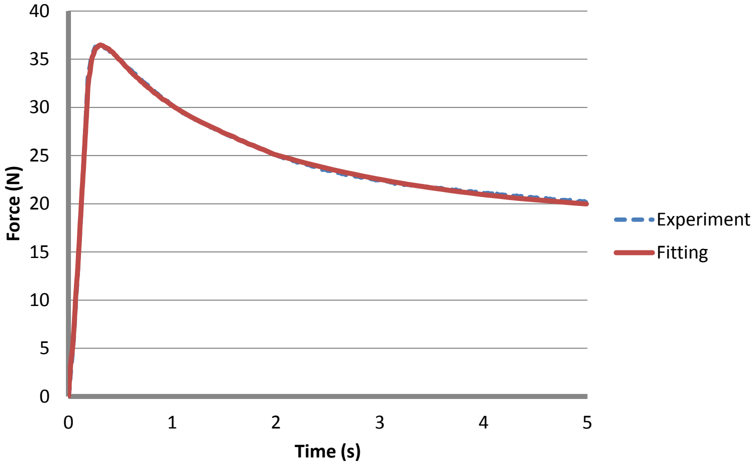

Figure 2. Fitting force curve using inverse analysis: $C_1 = 127.983$ Mpa, $C_2 = 21.33$ Mpa, $g_1 = 0.31173$, $\tau_1 = 0.2$ s, $g_2 = 0.33642$

Results of using both, force versus time ($F - t$), and contact area versus time ($A - t$), to determine material parameters will be presented.

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