Effective Young's Modulus of Rigid Particles in Gelatin Composites

by

Kamyin Cheng

Submitted to the Department of Mechanical Engineering
in Partial Fulfillment of the Requirements of the
Degree of

Bachelor of Science

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Abstract

In many biological systems, small rigid parts are embedded in deformable tissues to perform different biological functions. This study examines the effects of adding rigid filler particles inside deformable material. More specifically, a series of experiments led to eventual understanding of the relationship between effective Young's Modulus of material and volume fraction of rigid particles. The deformable material used in this study is gelatin, a readily available consumer product. It was found that the higher the volume fraction, the higher the Young's Modulus value for the composite material. In addition, it was found that cyclic loading with high strain and high volume fraction may cause stress stiffening or stress softening, while cyclic loading with small strain and small volume fraction yields linear elastic behavior. Furthermore, the effect of strain rate on material behavior was examined. Unfortunately the sample size was too small to draw definite conclusion. Finally, the reusability of particles was explored, and the results suggested that particles in composites are reusable so long as the composite did not undergo high strain compression.

Thesis Supervisor: Anette Hosoi

Title: Associate Professor of Mechanical Engineering

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1. Introduction

Young's Modulus is a measure of stiffness in materials. It is an important property in material science to predict mechanical behaviors. In this study, small rigid filler particles were mixed with soft deformable material to form uniform composite materials. The relationship between effective Young's Modulus and volume fraction of rigid particles was examined. This study was motivated by biological system with small rigid materials in soft tissues. To mimic the biological system, gelatin was used in this study as the soft material base chosen for convenience.

Throughout the study, many factors that potentially affect the material properties of the resulting composite were investigated. The manufacturing process, various loading and unloading rate, cyclic loading, used and new particles, and random and ordered particle packing were the experimental parameters that we varied to gain better understanding of how the composite material behaves.

2. Background Information

2.1. Inspiration

The SquishBot team is currently working on finding solutions to a compliant robot that will be able to squeeze through tight spaces. Many technologies have been proposed to allow soft bodies to morph. This study is aimed to further understand the mechanical effects of rigid fillings in deformable material. In many biological systems, this type of composite is very common. A study by M. A. R. Koehl in 1982 discussed the tensile behaviors of spicule-reinforced connective tissues from various cnidarians and sponges. Published in 1982, *Mechanical Design of Spicule-Reinforced Connective Tissue: Stiffness* explores whether or not spicules play any role in determining the effective mechanical properties while embedded in biological tissues. It also examined the mechanical effects of spicule size, shape and packing within connective tissues.

The results from this study indicate that spicules increase the stiffness of pliable connective tissues. The greater the volume fraction of spicules, the stiffer the sample will be. In addition, Koehl found that with a greater surface area to spicules per volume of tissue ratio, the stiffer the material will be. These are the main findings from the paper that inspired this thesis project.

To mimic the spicule behavior in connective tissues, the experiments in this thesis study used small 3D printed rigid particles in gelatin, a deformable material that resembles biological tissues. The purpose of this project is to examine the relationship between the effective Young's Modulus of the composite and volume fraction of rigid particles. A further purpose of this study was to explore the mechanical effect of strain rate, cyclic loading, and small and large strain compression.

2.2. Fundamental Principles

The objective of this study is to explore the underlying theories of stress for various composites. A uniaxial loading was applied to various specimens of materials in order for deformation to take place. Stress is a measure of loading per unit area, and strain is the measure of deformation. The physical constants that arise in relating the two define the material properties of the substance.

All the experimental data derived in this study were processed to derive engineering strain and engineering stress. These engineering parameters measured are based on initial referenced conditions:

$$\sigma_{eng} = \frac{F}{A_0} \quad (1)$$

$$\varepsilon_{eng} = \frac{L - L_0}{L_0} \quad (2)$$

Equation 1 shows the engineering stress, σ_{eng}, as a function of force, *F,* per original specimen area, A_0. Equation 2 describes engineering strain. It is the change in length with respect to original length, L-L_0, divided by the original length, L_0.

Stress and Strain and provide information on the material stiffness, or the Young's Modulus, *E*. It is given by equation 3 below. It is essentially the slope in a stress-strain curve, as shown in figure 1 below. The steeper the slope, the stiffer the material.

$$E = \frac{\sigma}{\varepsilon} \quad (3)$$

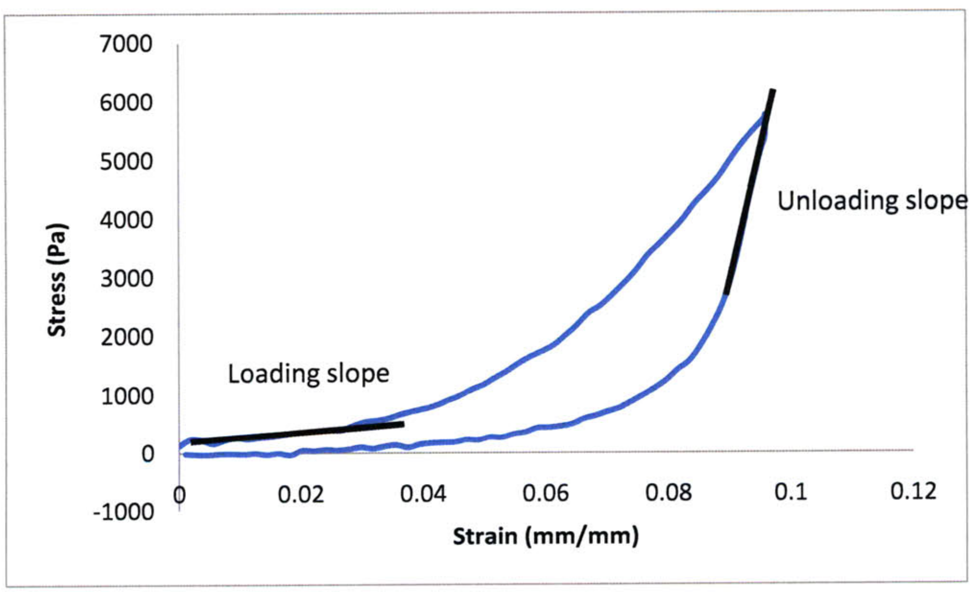
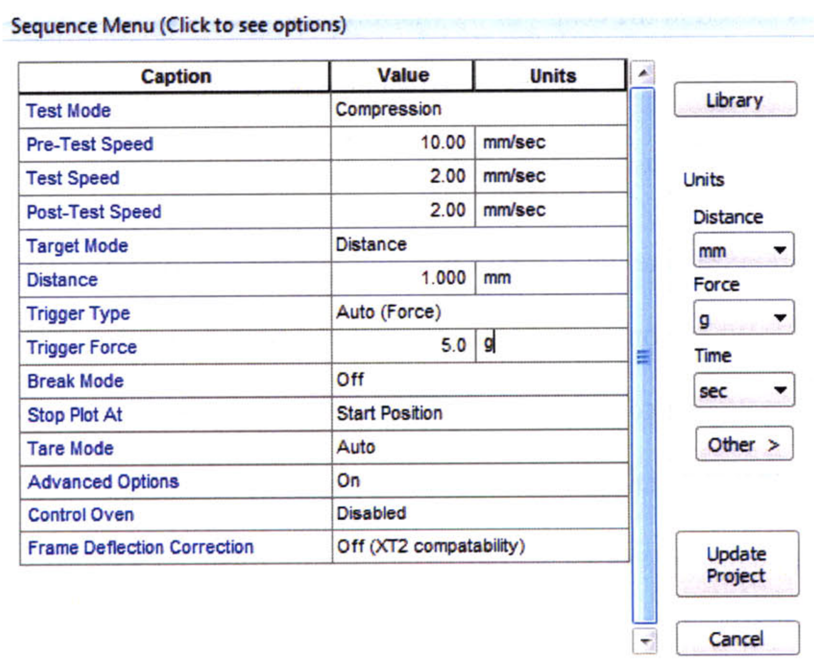


Figure 1: A typical stress-strain curve showing where the loading and unloading slopes are measured.

3. Experimental Setup

3.1 Texture Analyzer

The equipment that was used to compress samples is a TA.XT.plus Texture Analyzer from Stable Micro Systems. It comes with software to control experimental parameters and to collect compression data. In order to run a typical experiment, the user first needs to calibrate the machine to make sure it has an accurate reading in force and displacement. After resetting those values, one can go to the TA settings to select the desired experimental parameters, including test mode, loading speed, unloading speed, and trigger type. Figure 2 below displays a typical TA setting window.


Sequence Menu (Click to see options)

Caption	Value	Units
Test Mode	Compression	
Pre-Test Speed	10.00	mm/sec
Test Speed	2.00	mm/sec
Post-Test Speed	2.00	mm/sec
Target Mode	Distance	
Distance	1.000	mm
Trigger Type	Auto (Force)	
Trigger Force	5.0	g
Break Mode	Off	
Stop Plot At	Start Position	
Tare Mode	Auto	
Advanced Options	On	
Control Oven	Disabled	
Frame Deflection Correction	Off (XT2 compatability)	

Figure 2: TA Setting interface within the Texture Exponent 32 program. It allows users to select experimental parameters.

All the samples were examined under compression. The loading and unloading speeds were different throughout the experiments in order to compare the effect of strain rate on material properties. Compression distances also vary throughout the experiments, ranging from 1mm to 5mm.

It is important to note the difference between 'Button Trigger' and 'Force Trigger'. 'Button Trigger' starts collecting data at the starting position, thus it is easy to precisely detect when the probe contacts the sample and if there is any sample shrinkage after cyclic loading. However, it is difficult to control the compression distance. In contrast, force trigger starts collecting data points when the probe hits the composite sample. This is useful in order to control the compression distance, yet the disadvantage of using such trigger is the inability to detect if the sample has shrunk.

The Sample is placed in the center of the platform and the machine starts collecting data by pressing 'run test' from the software. The user can then collect the force and distance data and convert this into a stress-strain relationship with further data processing.

It is also important to note the system compliance of the machine. The texture analyzer itself is not infinitely stiff. When compressing an object, the machine itself will have some internal deflection. This is analogous to the effect of deflection on a cantilever beam, where the force sensor is measuring the deflection on the fixed end, but the desired measurement is on the free end. System compliance, k, is calculated by dividing the Force with displacement in a controlled force test. To obtain the effective displacement, the displacement value collected from the sensor is reduced by the calibrated compliance value and the loading force.

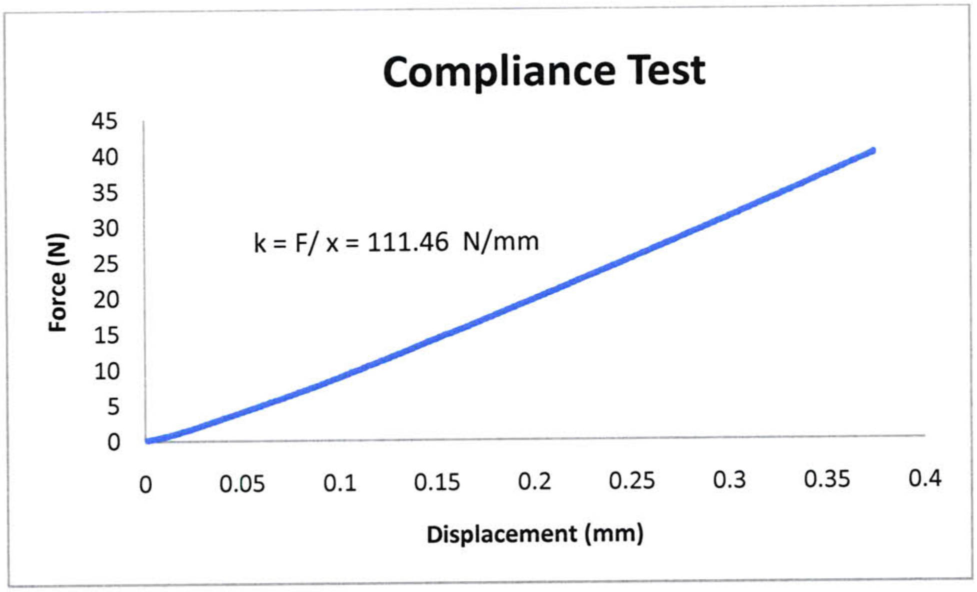


Figure 3: Results from a system compliance test.

Figure 3 above shows a typical curve for a compliance test. The compliance value, *k*, is the slope of force over displacement. To obtain a truly accurate result, one should consider subtracting the system compliance from the measured results. However, it is important to note that the results from this study did not take this compliance adjustment into account. To justify the results without compliance test, figure 4 shows a set of data adjusted with compliance and compared to the same data without adjusting for machine compliance.

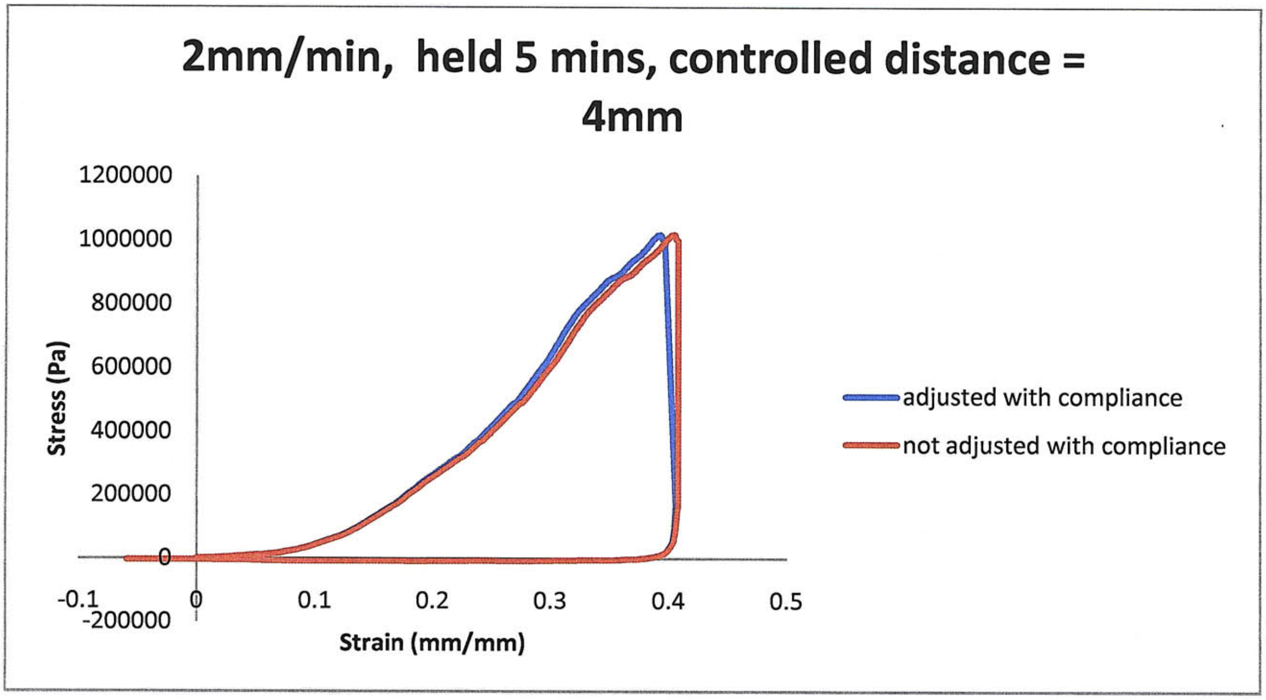


Figure 4: Comparison of results with compliance adjustment and without compliance adjustment

The results with and without compliance adjustment display mirror inconsistencies for large strain. The loading Young's Moduli (initial slope) resemble each other. Due to the large amount of data collected throughout the study, all the data were not processed with compliance adjustment to find the effective modulus. However, this should not change the qualitative conclusion drawn. Nevertheless, for future exploration, this is something that should be considered in order to obtain true accuracy on data collection.

3.2 Particles

The rigid particles that are embedded in each composite sample were printed with the Objet PolyJet-based 3-Dimensional Printing Systems. The material used is FullCure®720. This material has a Young's Modulus value of 2870 MPa according to the data sheet from the company's website. The dimensions of an individual particle are shown in figure 5.

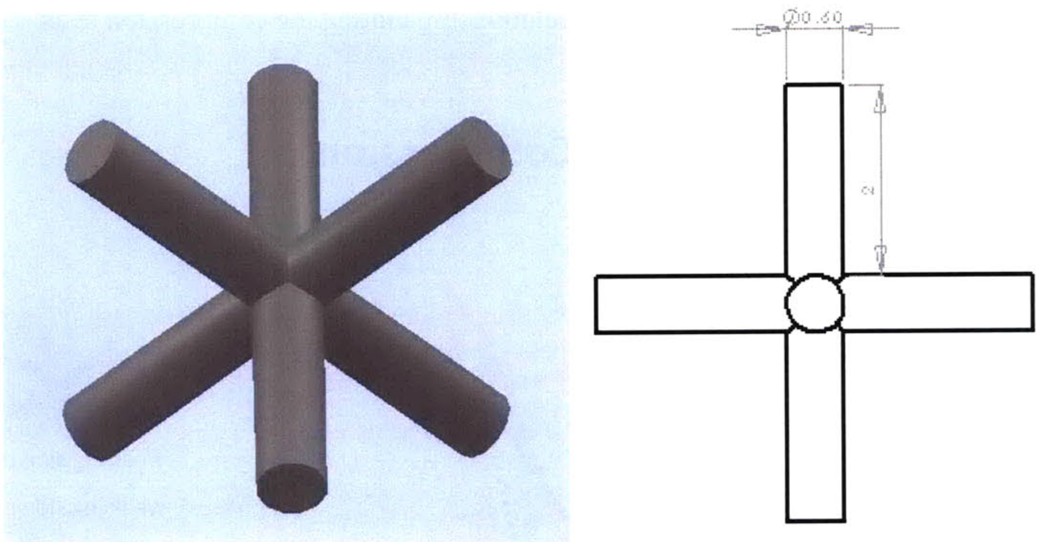


Figure 5: Particle used in all experiments. Dimensions are in mm.

The geometry was previously explored and designed by Sarah Bates. It is an interesting shape to work with especially when the particles start interacting and interlocking with each other. Each particle has a volume of 3.563×10^{-9} m^3. Such geometry exhibits a higher volume to surface area ratio than a regular sphere sharing the same volume of materials. This shape also better distributes the weight of the particle to make the results more interesting.

After the particles are 3D printed, they were first embedded in a sheet of support material. They are then individually picked out from the support material and transferred to a meshed bag.

After the meshed bag is filled with particles, it is soaked in a 2M NAOH solution for 2 hours. Lastly, the meshed bag was rinsed under water. The particles are then dried and ready to be used.

To further explore the properties of these particles, a series of individual particle compression tests were performed using the texture analyzer. While printing the particles, 1 leg was embedded in the support materials, and it appears to be the weakest of the 6 legs. Two compression tests were done with weak leg side up and 2 compression tests with weak leg side down. Figure 6 below shows the stress-strain relationship among the compression tests.

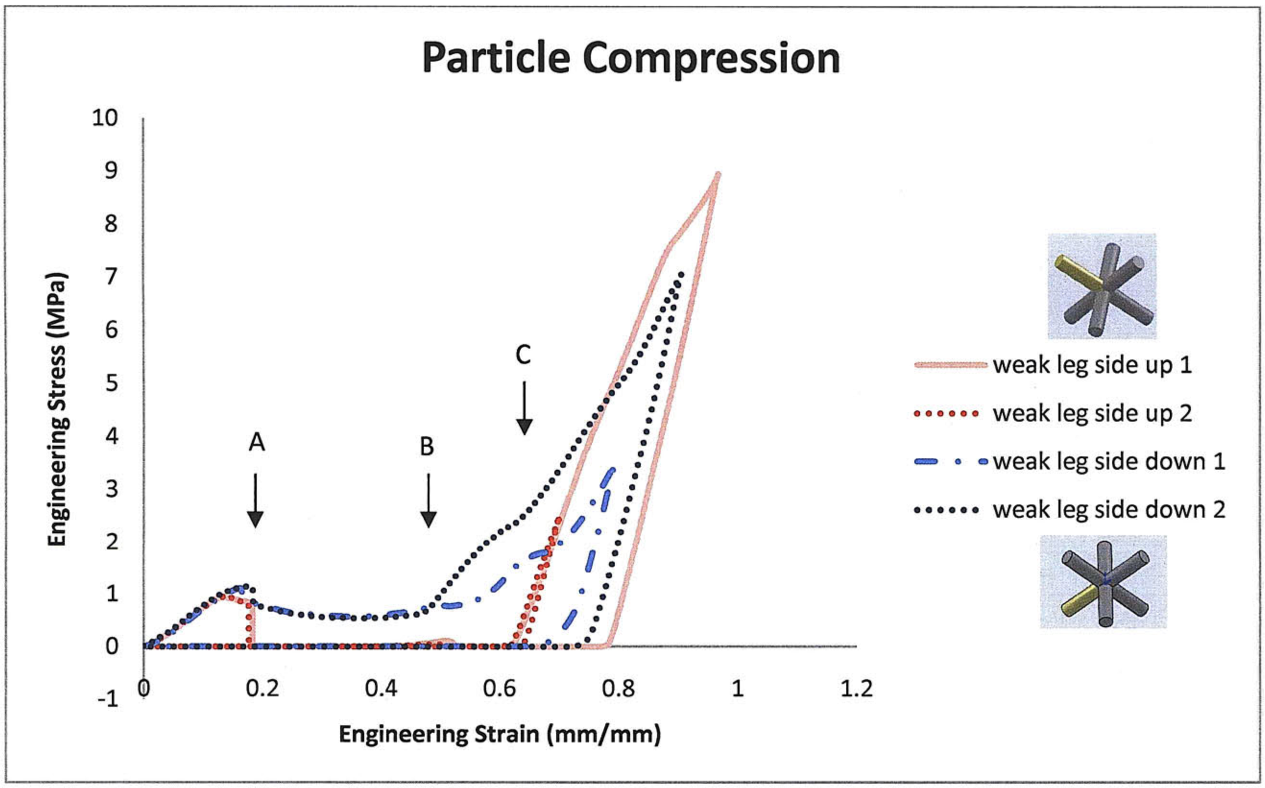


Figure 6: Stress strain relationship of compressing 1 particle

Figure 6 illustrates the stress strain relationships of the 3D printed particle of both weak leg side up and weak leg side down. For the weak leg side up compression tests, when the probe hits point A, weak leg fractures. However, since the fracture occurs on top, while the bottom 3 legs were still stable, the particle did not rotate itself to adjust. Following the fracture, there were no force to push back the sensor, thus between point A and B there is practically no stress. At point C, the compression hits the point there is barely enough physical space for the particle, hence the steep slope after point C.

On the other hand, for weak leg side down compressions, when the compression hits point A, the weak leg probably fractured, and then the particle rotated to adjust itself to withstand more stress. Up until point B the particle has no more room to 'move around', thus stress increased dramatically.

3.3 Gelatin

Gelatin is another key component to the composite samples. To prepare the gelatin solution, 85g of JELL-O packages were purchased and the 'Jiggers' recipe from the back of the packages were followed. Since the 'Jiggers' recipe requires 4 packages of gelatin powder, the amount of water added was reduced. For 85g of gelatin powder, 156mL of boiling water was added and stirred for 3 minutes. The gelatin solution is then added to the composite mold, to be refrigerated for at least 3 hours.

3.4 Mold

Various methods of molding were explored in designing the manufacturing method of composite samples. Originally the composite samples were prepared and put into a 3D printed cylindrical mold. However, it is very difficult to get the samples out once the gelatin solution stiffens. Several ideas were proposed such as a bottomless mold to be mounted to a flat surface; lining the interior with wax paper, plastic wraps, adding a layer of flour to prevent the sample sticking to the inner wall of the molds; or even making the samples in a big tray and using a cookie cutter method to obtain a certain shape. A preliminary experiment was performed to compare the efficiency of proposed methods.

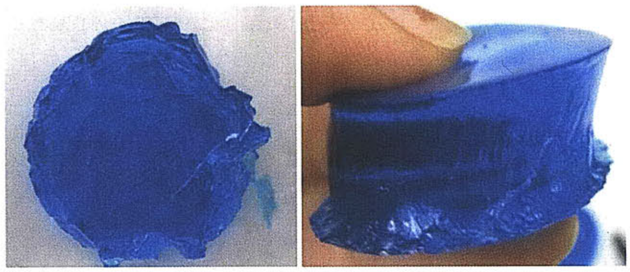


Figure 7: Samples made with plastic wrap lining (left) and cookie cutter method (right).

The Jell-O mold without bottom was a failure. The intention was to mount the sides of the mold to a flat surface via duck tape. However, Jell-O solution has a very low viscosity thus it started leaking through the duck tape.

The wax paper technique resulted in the best surface finish. Plastic Wrap gives very rough edges, as seen in figure 6 above. Gelatin samples in floured molds are still very difficult to take out, and cookie cutters give uneven cross-sectional areas (figure 7). Therefore, all the samples throughout the experiments were prepared with wax paper lining.

The molds explored in the preliminary testing have a size of 30mm diameter. Another element explored in terms or making the mold is the size. Ideally the smaller a mold would be better, since the rigid particles are difficult to process and it would be nice to use fewer. A 20mm diameter mold was used to compare to the 30mm diameter results. All the molds were made out of Tango Grey Materials and the dimensions of each mold are shown in figures 8 and 9.

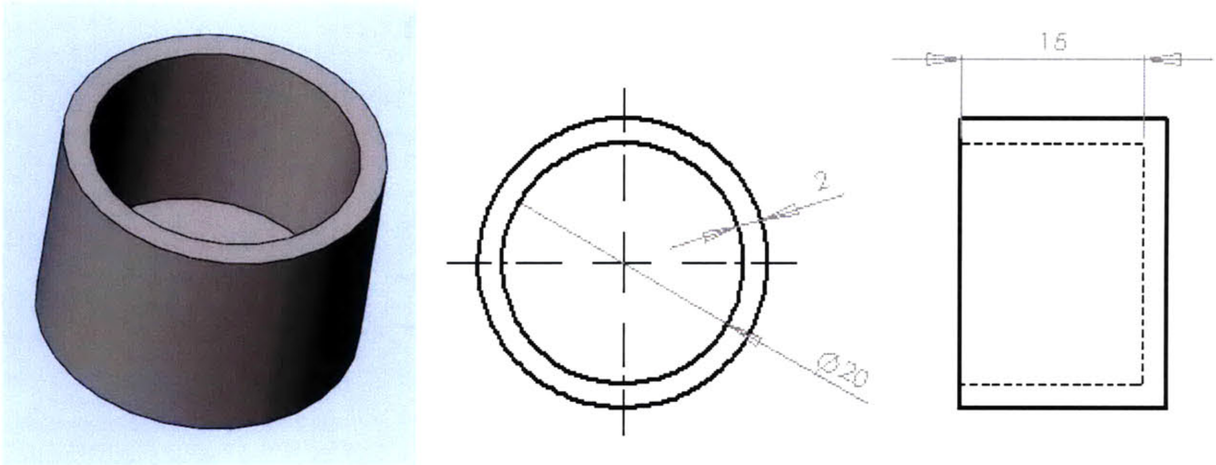


Figure 8: Dimensions (mm) for the small JELL-O mold.

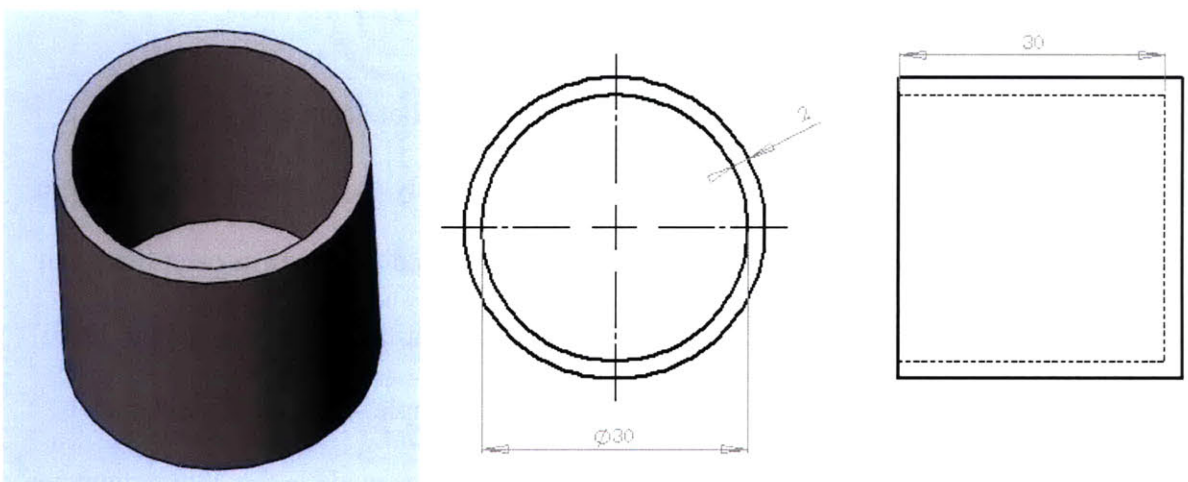


Figure 9: Dimensions (mm) for the big JELL-O mold.

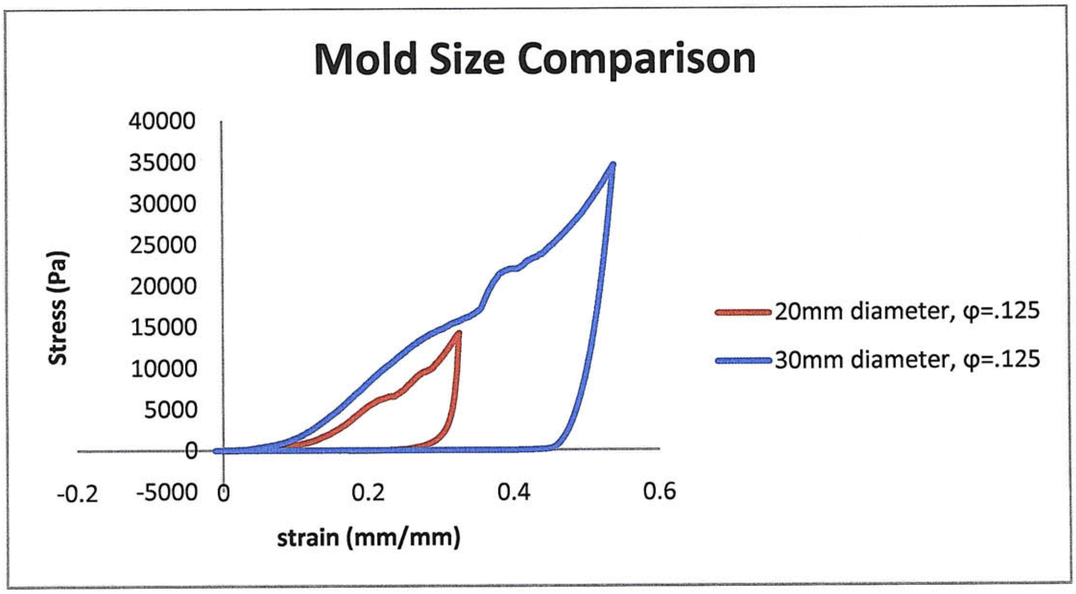


Figure 10: Comparison of gelatin samples with 20mm diameter and 30mm diameter, both sharing the same number of particles and same volume of gelatin solution.

Figure 10 shows the stress-strain relationships of 2 samples, both containing the same number of particles and same amount of gelatin solution. The only difference is that they were made in different mold sizes, namely 20mm diameter and 20mm diameter. It is clear that even with different size mold, the Young's Modulus of the materials still remain comparable. From figure 10 above one can infer that the loading slopes of both samples are parallel. All the composite samples throughout the series of experiments were thus made in the 20mm diameter mold to conserve materials. Additionally, using a smaller sample mold also helps conserve sample heights as well as allowing for smaller strain comparisons.

3.5 Making the Samples

To make the composite samples, the inner wall of the mold is first lined with wax paper. In the earlier experiments, a circle about the size of the mold is also cut out to put in the bottom. To achieve the desired volume, the number of particles is counted and placed in the mold prior to adding the gelatin solution. Almost all of the composite samples contain 2.5 mL gelatin solution, and this volume is pipetted into the mold. The samples are then transferred to a cooling device (i.e. Refrigerator)

Many experiments were performed to explore the material properties of rigid particles in deformable material. Specimens with or without rigid particles were compared to see the effects of adding rigid particles in gelatin. Furthermore, samples made with different volume fractions of rigid particles were examined and compared. Cyclic loading tests were also performed to gain a better understanding of material behavior. In addition, composite samples were compressed under different loading and unloading speeds to determine whether the stain rate plays a role in measuring materials properties. Used particles were also re-used to make samples in order to test the repeatability of the rigid particles. Lastly, the deposition methods of particles within each sample were compared to examine whether a layered distribution of particle in samples would behave differently from random distribution of particles.

4. Results

4.1 Gelatin without Particles

The mold exploration experiments provided some preliminary results in gelatin samples without any rigid particles. Figure 11 below summarizes the results.

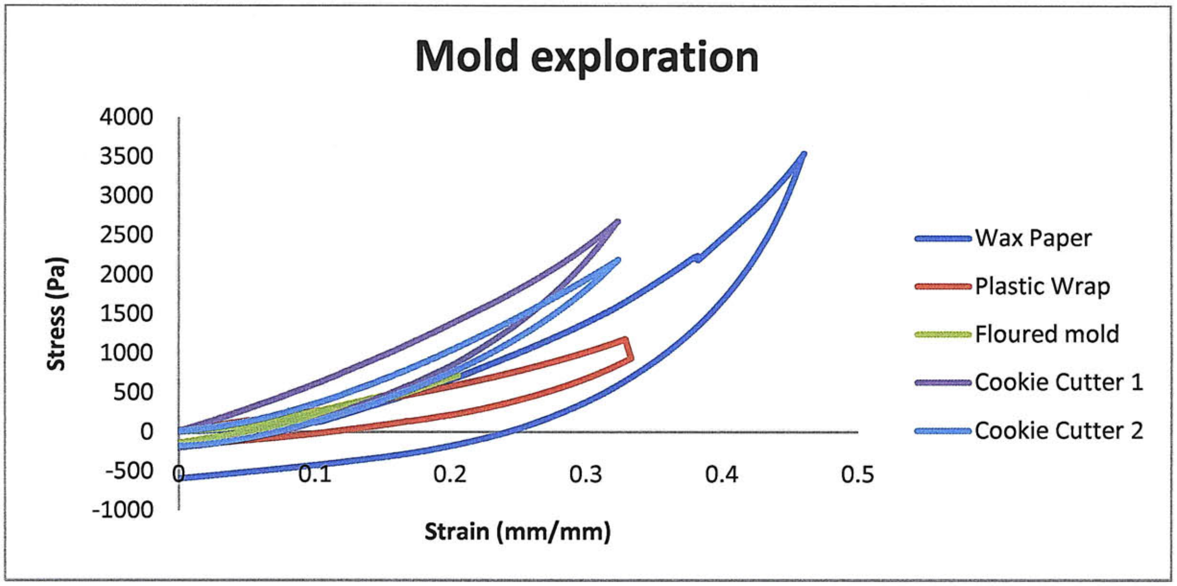


Figure 11: Results from exploring which sample manufacturing method to use.

	mold method	Diameter (mm)	height (mm)	Young's modulus (kPa)
Jello A	wax paper	30	10.8	1 - 5
Jello B	plastic wrap	30	12	2
Jello C	floured interior	30	18.8-20	2
Jello D	cookie cutter	30	12.3	6
Jello E	cookie cutter	30	12.3	2 - 6

Table 1: Results from manufacturing method exploration.

This experiment was performed under a trigger force of 0.3g, loading speed of 2mm/min, and unloading speed of 10mm/min. Table 1 displays the values of the Young's Modulus measured in these tests. Note that without any particles inside the gelatin samples, the Young's Modulii are on the order of 1kPa. The stress-strain curve differences among the samples are likely a result of uneven heights for some samples, imperfect cylindrical shape for some, or difference in height among all samples. The negative stresses for the sample with wax paper lining was a result of sample sticking to the bottom of the probe while unloading, as shown in figure 12 below.

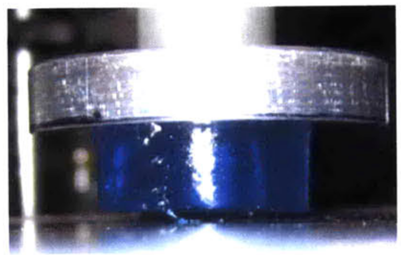


Figure 12: Results from manufacturing method exploration.

The above results were then compared to a composite sample with a rigid particle volume fraction of 0.12 to examine the effect of having particles inside gelatin materials. Figure 13 below displays the comparison between samples with particles and without particles.

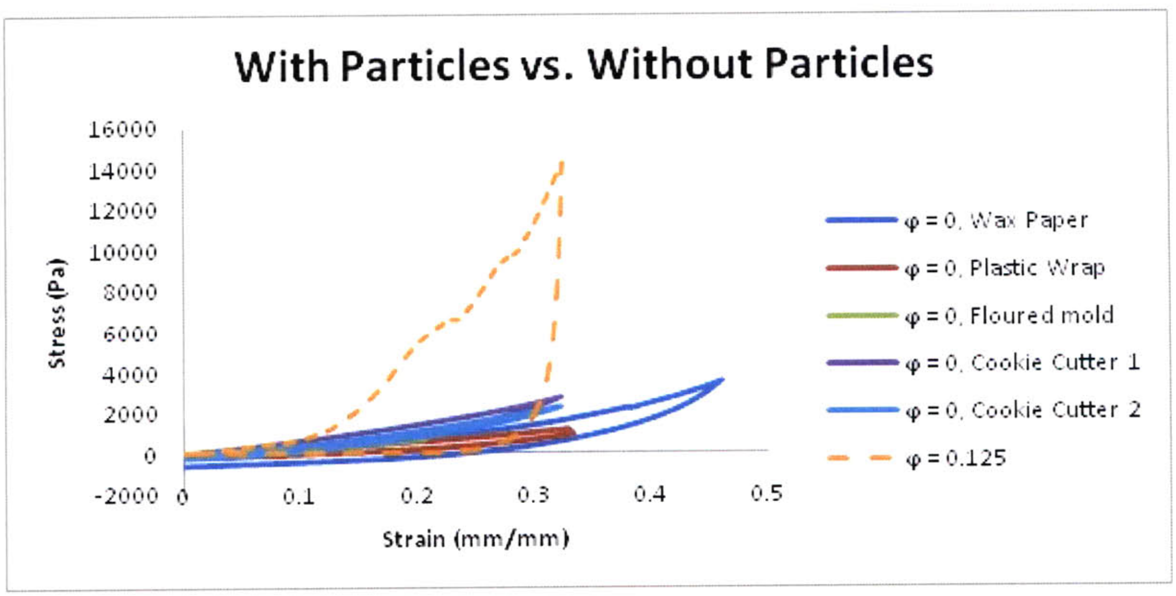


Figure 13: Comparison between samples with particles and without particles.

The sample with $\phi = 0.125$ from the above graphs indicates that composite samples with particles display larger internal stresses at a given strain compared to the samples without particles. This means that with the added rigid particles, the stiffness of the composite increases. Note that the loading and unloading slopes from the above stress-strain relationship are steeper with particles than without particles.

4.2 Volume Fraction vs. Young's Modulus

A series of composite samples were prepared with different volume fractions for comparison. The following diagrams illustrate the compression results from 7 samples, with volume fractions of 0.09 and 0.0125.

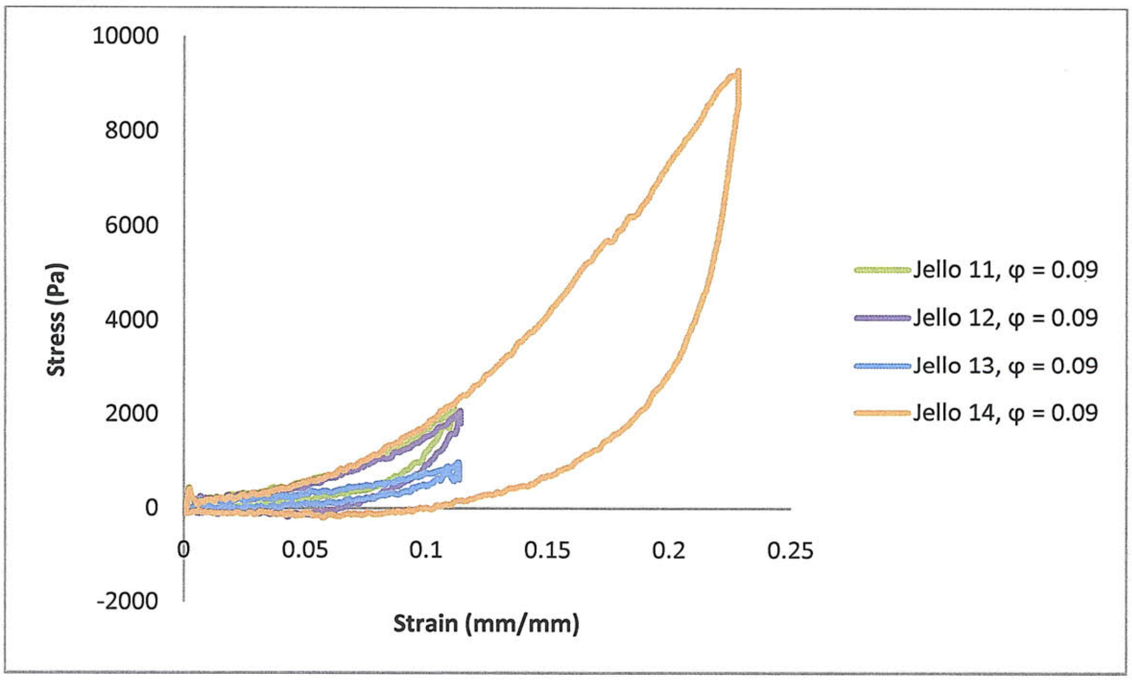


Figure 14: Volume fraction is 0.09, 2mm/min compression, force trigger of 0.5g

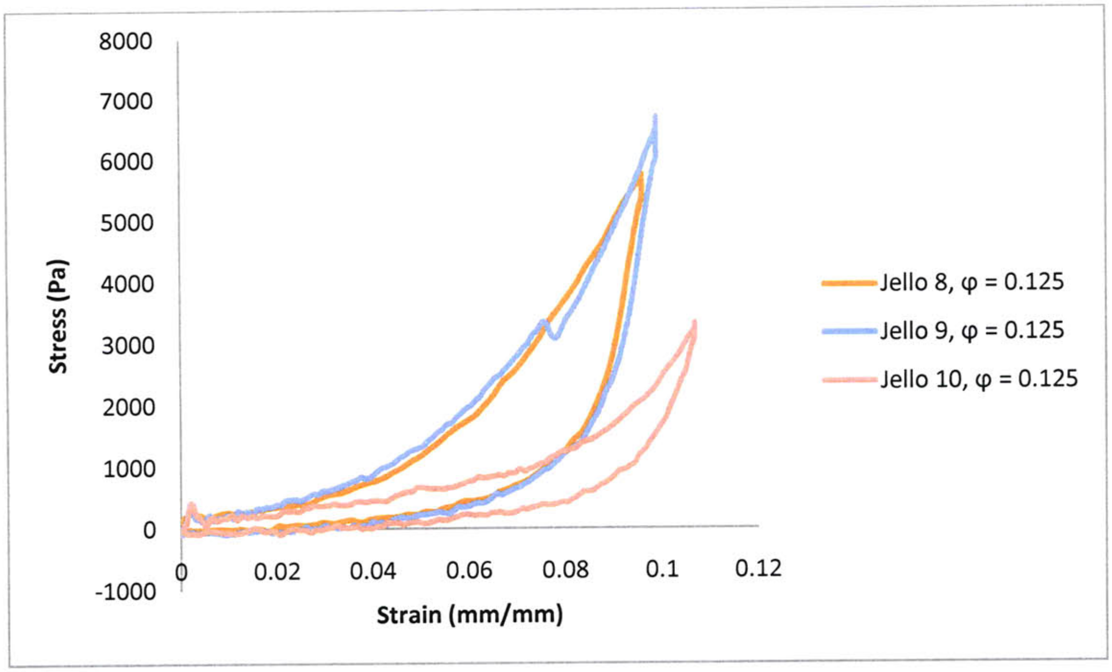


Figure 15: Volume fraction is 0.125. 2mm/min compression, force trigger of 0.5g

All samples from figure 14 and figure 15 above were compressed with loading and unloading speed of 2mm/min. They were also measured with force trigger of 5g. With each respective volume fraction, the loading and unloading slopes were fairly similar. We suspect that the off-shifted curve is a result of sample inconsistency, as discussed in section 5.

As shown in equation 3, the slopes of a stress-strain curve tell us the Young's Modulus of a material. The Young's Modulus characterizes the stiffness of material. The hypothesis of this thesis is that volume fraction of rigid particles positively affects the Young's Modulus of composite structures. A series of experiments with samples that of different Young's Modulus was performed; figure 16 below summarizes the results.

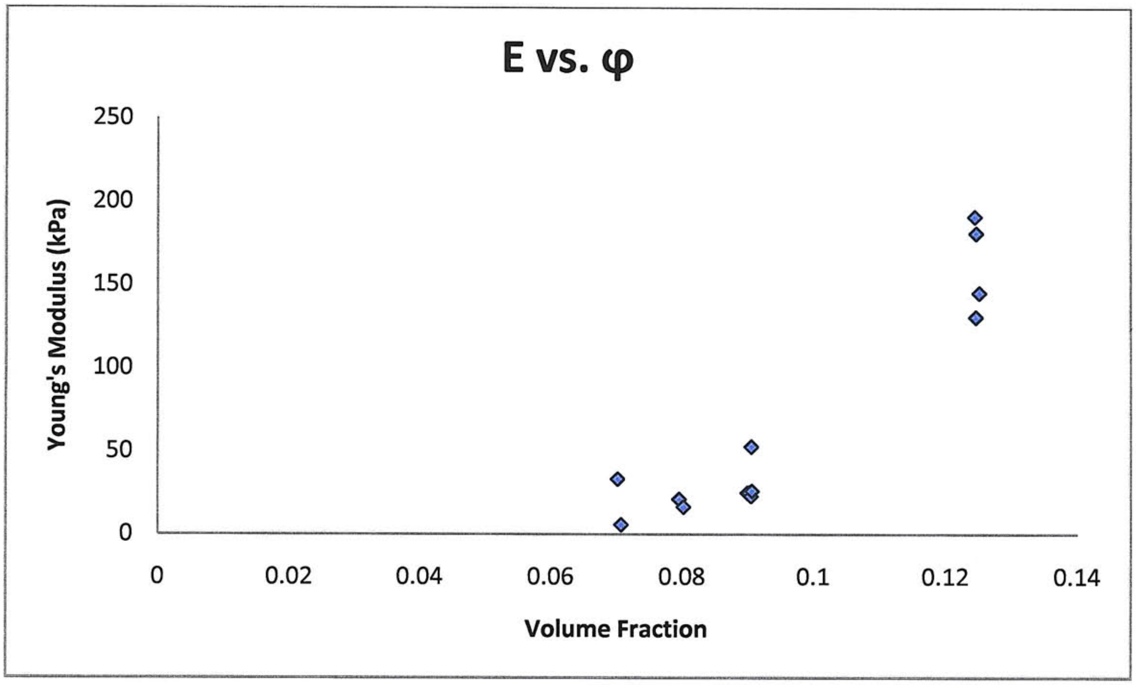


Figure 16: Volume fraction comparing to effective modulus

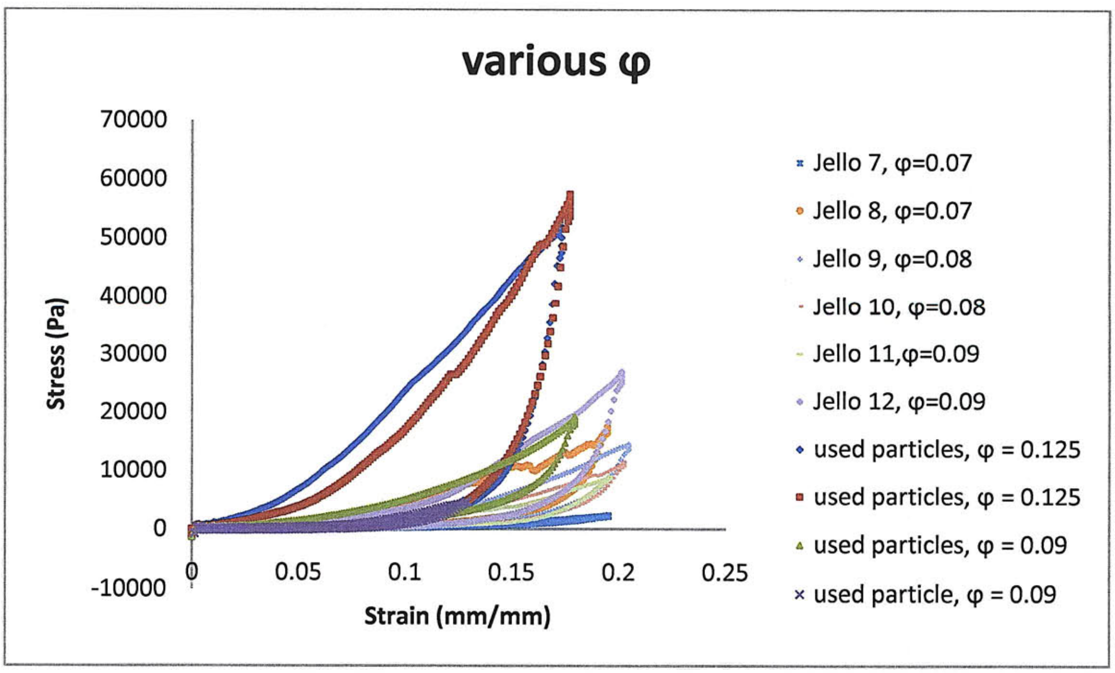


Figure 17: Stress-strain relations of samples with different volume fraction

Table 2: Volume fraction (ϕ) vs. Young's Modulus (E).
These are the values in graphed in figure 14.

ϕ	E (kPa)
0.07	33
0.07	6
0.08	21
0.08	16
0.09	25
0.09	25
0.09	23
0.09	53
0.09	26
0.12	191
0.12	131
0.12	181
0.13	145

As shown in the figure 16, 17 and table 2, there is a positive trend between Young's Modulus and volume fraction. The more rigid particles are in the sample, the stiffer it gets. There is an increasing trend in the effective Young's Modulus values as the volume fraction increases. Nevertheless, there were not enough samples to determine the functional form of this trend. All the samples are very different in their own way and it was challenging to come up with a uniform method to measure the Young's Modulus. Larger sampling size is needed to provide an accurate characterization.

4.3 Strain Rate & Cyclic Loading

As mentioned in the experimental procedure section, the force trigger and button trigger plays an important role in data collection. Force trigger allows data collection starting at a position where the force sensor in the texture analyzer detects a force, while button trigger starts collecting data a distance above the sample surface. Figure 18 is an example of data collected by button trigger.

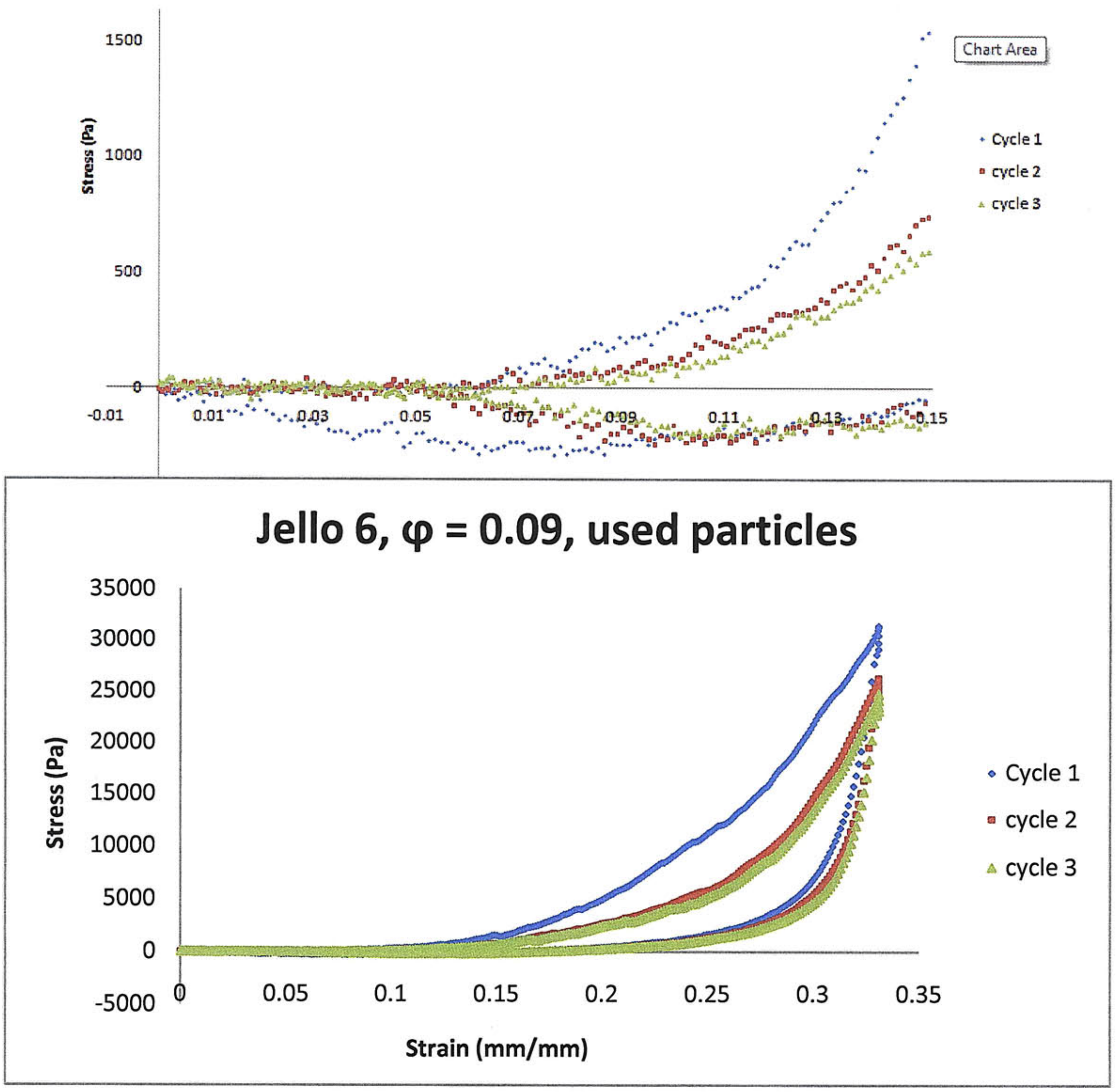


Figure 18: Button Trigger and its shifting.

This sample was prepared with used particles, and it has a volume fraction of 0.09. The loading and unloading speeds are both 2mm/min. The top diagram shows the initial strain in a higher resolution. After the first loading cycle, the sample seems to become slightly shorter as seen in the top diagram. The second and third cycles are shifted to the right. The distance between zero strain and where the slope starts inclining is the distance between the bottom of the probe and top of the sample surface.

These cyclic loading tests performed immediately one after the other. It also seems that with larger strain (> 0.3), the first cycle has the stiffest response compared to the latter cycles. The loading Modulus of Elasticity seems to be greater than those of the 2nd and 3rd cycle. This could be a result of particles rearranging internally after the first large strain compression.

Different compression speeds were explored in multiple samples in order to understand effects of loading speed on material properties. The results are listed in the following diagrams.

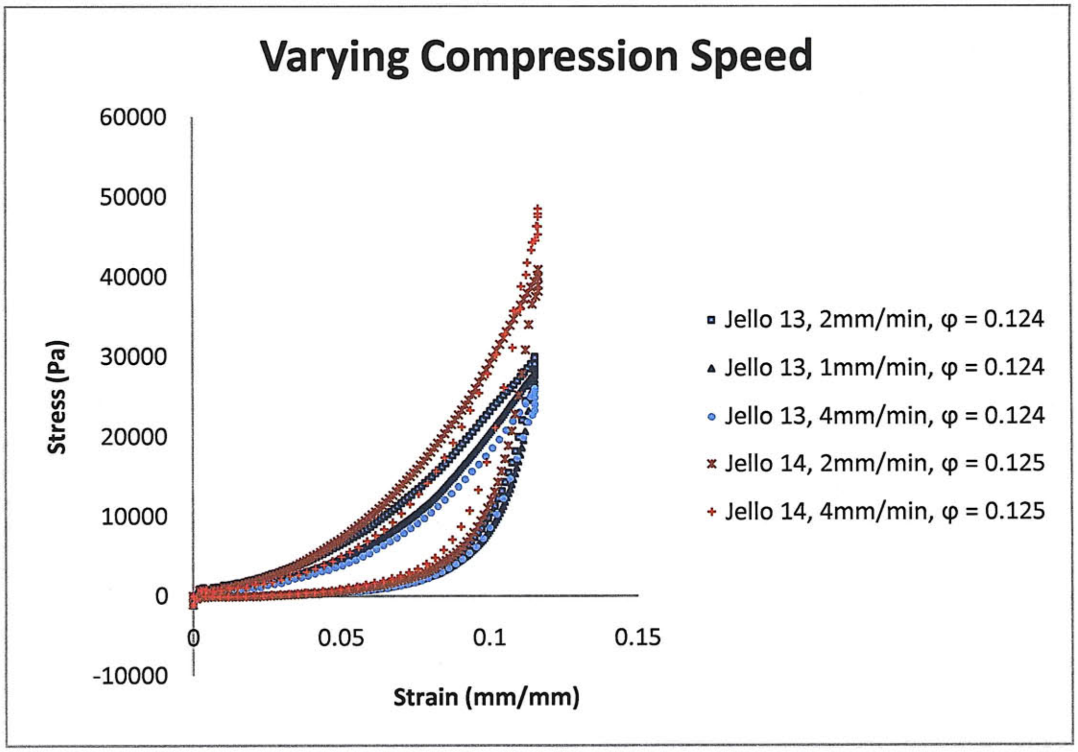


Figure 19: Volume fraction of 0.124/0.125, various strain rates.

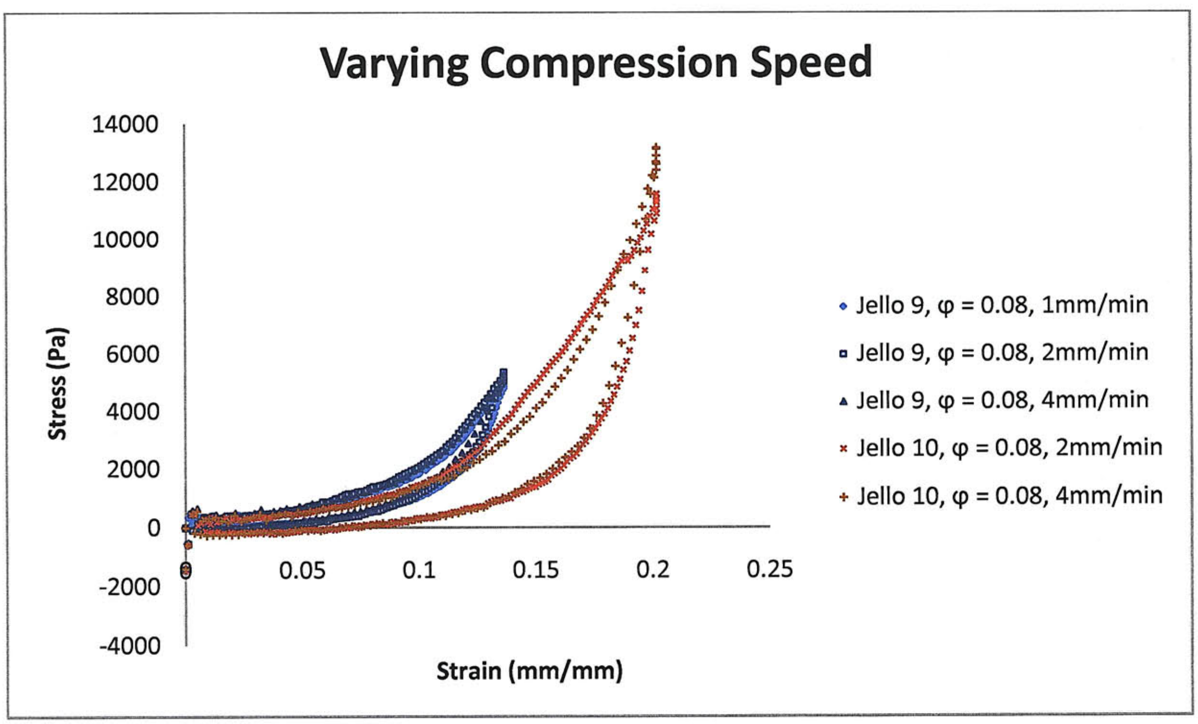


Figure 20: Volume fraction of 0.08, various strain rates.

From the above figures, strain rate does not have a significant effect on the stress and strain relationship in composite samples. Samples with loading speed of 1mm/min, 2mm/min, and 4 mm/min exhibit similar stress-strain behavior. Figure 20 above also shows that with a smaller strain; more repeatable results are obtained.

The following 2 graphs show the cyclic loading results for 2 samples, each with a volume fraction of 0.125. Both data sets were recorded with force trigger. Unlike cyclic loading from figure 19 above, with small strain compression there is not a drastic difference of stiffness between each cycle. All the stress-strain curves resemble each other in this case, whether considering different strain rate or different cycles.

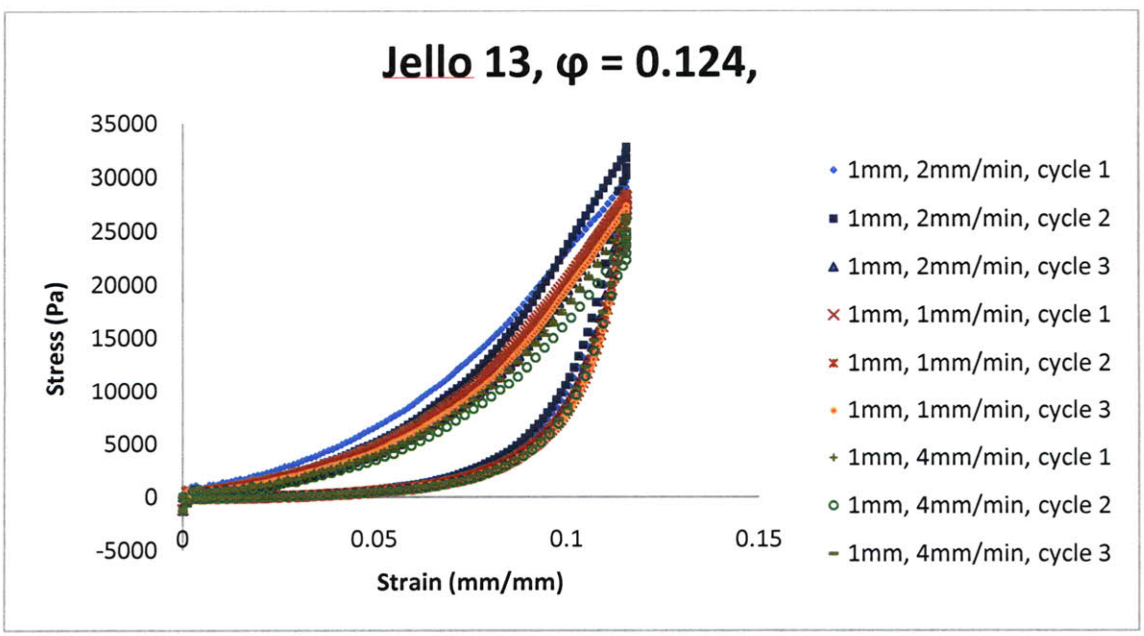


Figure 21: Volume fraction of 0.124, cyclic loading with various strain rates.

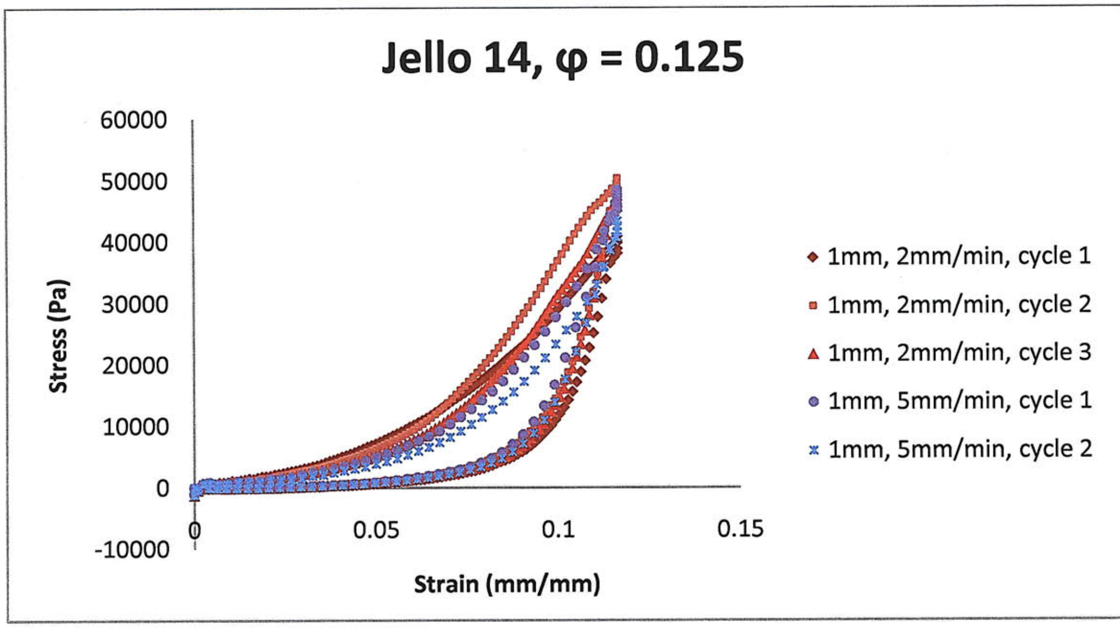


Figure 22: Volume fraction of 0.125, cyclic loading with various strain rates.

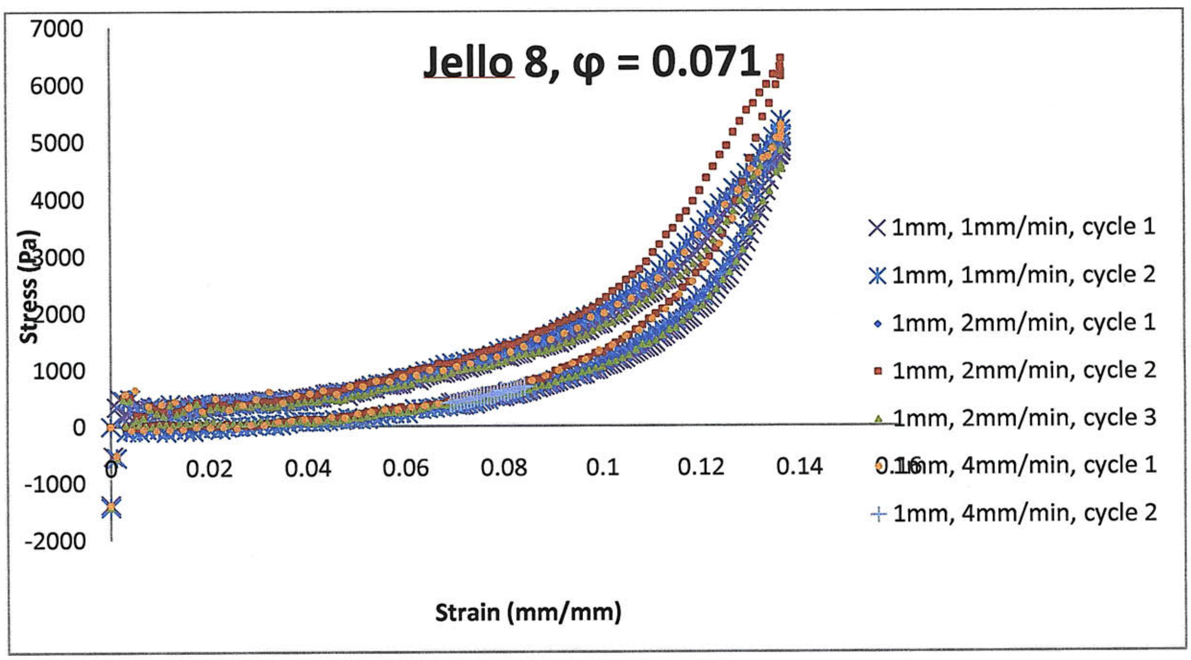


Figure 23: Volume fraction of 0.071, cyclic loading with various strain rates.

Figure 23 above shows a composite sample with a volume fraction of 0.07. The stress-strain curves resembles each other, which shows that strain rate and cyclic loading do not have significant effect for small strain compression.

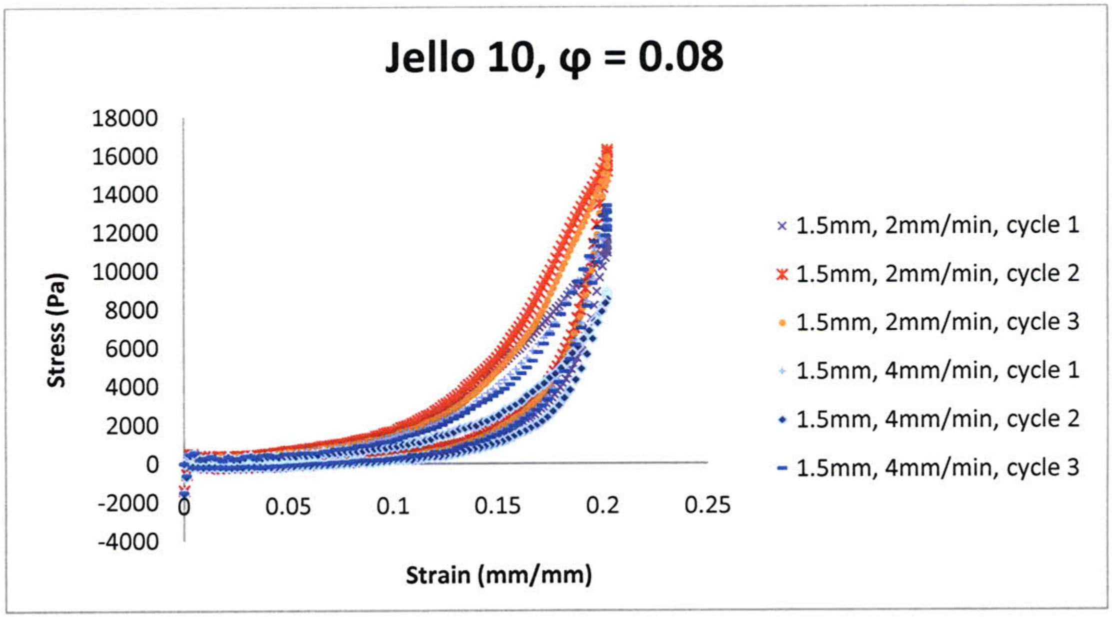


Figure 24: Volume fraction of 0.08, cyclic loading with various strain rates.

Figure 24 shows a composite sample with a volume fraction of 0.08. It is recorded with force trigger as well. This graph, contrary to the previously mentioned experiments, suggests that faster strain rates may slightly vary the stress-strain relationship for materials. The set of data that were loaded and unloaded with 4mm/min seems less stiff in higher strain regions.

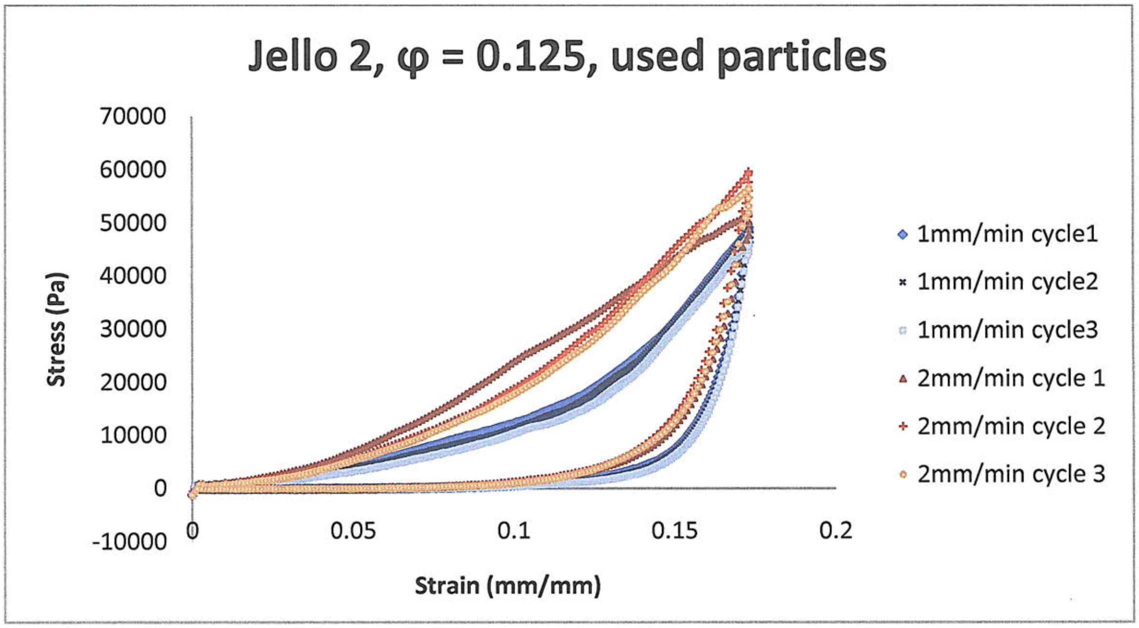
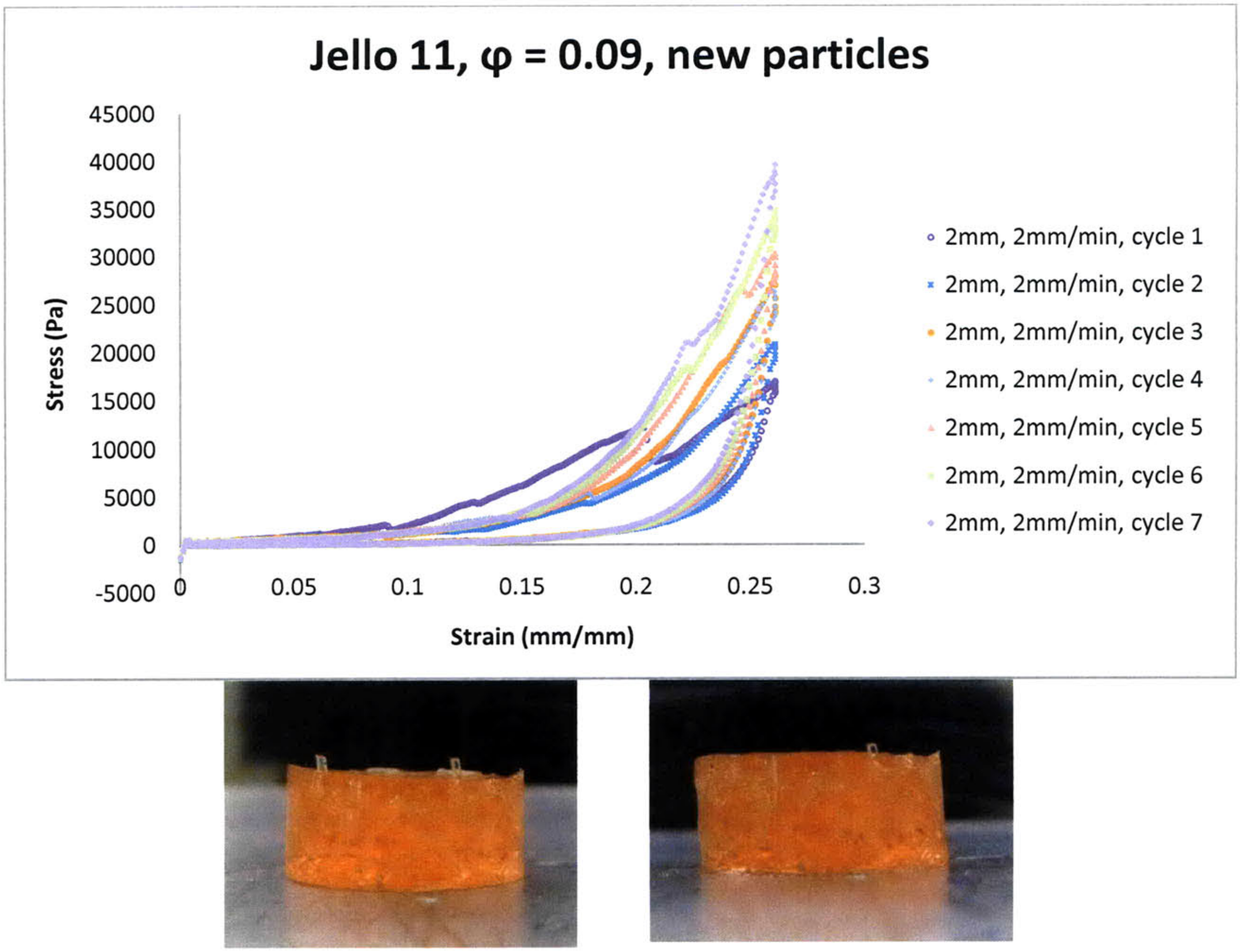


Figure 25: Volume fraction of 0.125, cyclic loading with various strain rates.

The composite sample shown in figure 25 was made from used particles, which will be discussed later to show that they behave just as new particles. This figure also indicates that material properties may depend on strain rate. The loading slopes for 1mm/min sample and 2mm/min sample are slight different, yet each cyclic loading group yields consistent results.

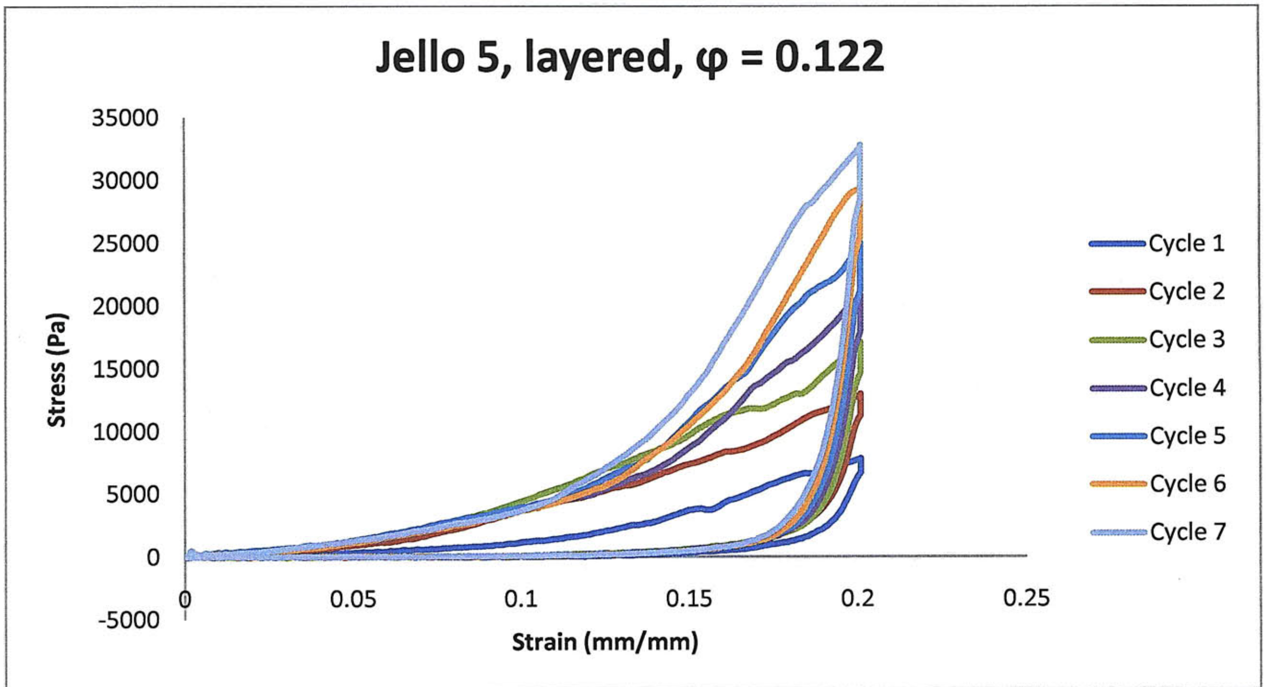
Since there are many different behaviors we observed for different individual composite samples, the results from this experiment are inconclusive. Whether the strain rate affects the material properties of composite samples cannot be concluded at this time, and this is a direction that can be explored in the future. There were not enough samples that behave consistantly due to the discrepancy discussed in section 5 to make a valid conclusion.

One surprising finding throughout the study is the phenomenon of stress stiffening in high volume fraction or high strain samples. During cyclic loading tests, it was found that the composite samples get stiffer after every compression cycle for some composite samples. Figure 26 below is one of the examples.



Before After

Figure 26: Volume fraction of 0.09, cyclic loading (top). Pictures of the composite sample before and after the cyclic loading test is shown at the bottom



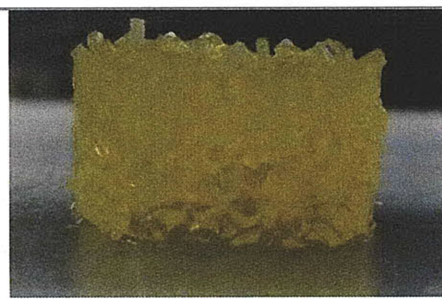
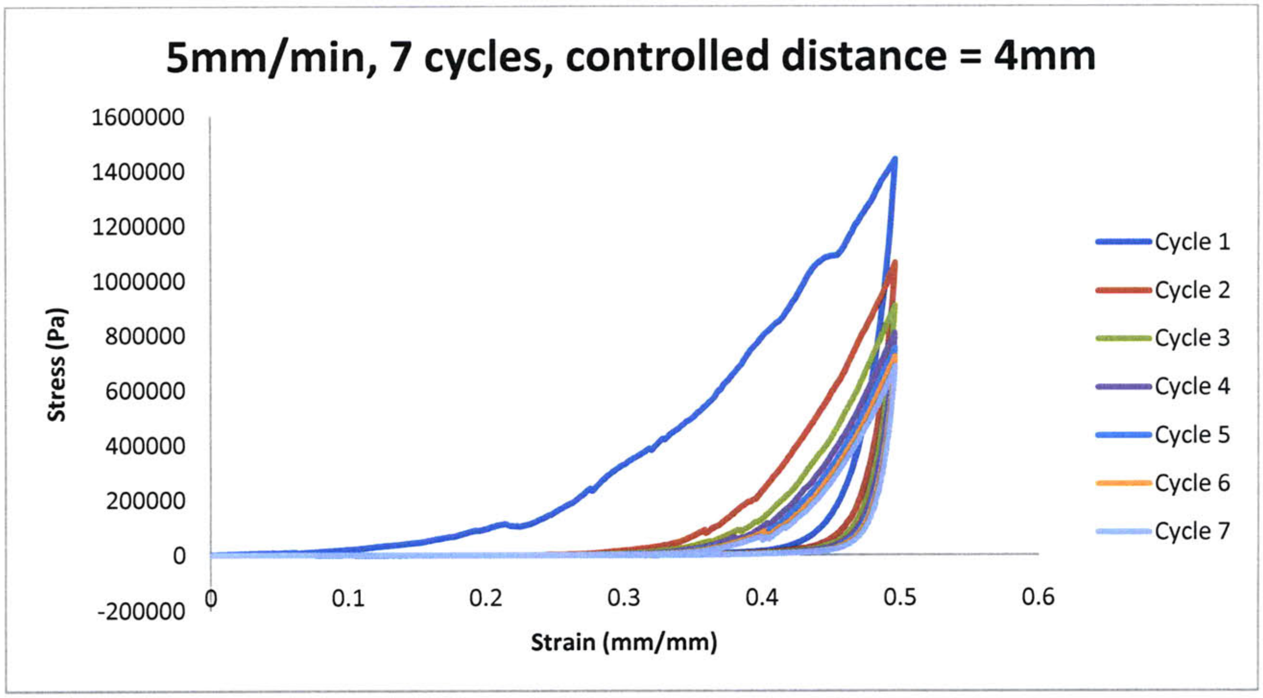


Figure 27: Volume fraction of 0.122, cyclic loading (top). Picture of composite sample for this test is displayed in the bottom.

The before and after pictures in figure 26 explain the stress stiffening phenomenon indicated in the graph. Before the compression begins, there were particles sticking out from the surface. This also applies to figure 27 above where many particles stick out. After each loading cycle, the probe compresses the particles so much that the legs got pushed into the gelatin bedding. The particle rearrangement and interlocking thus stiffen the sample after each loading cycle.



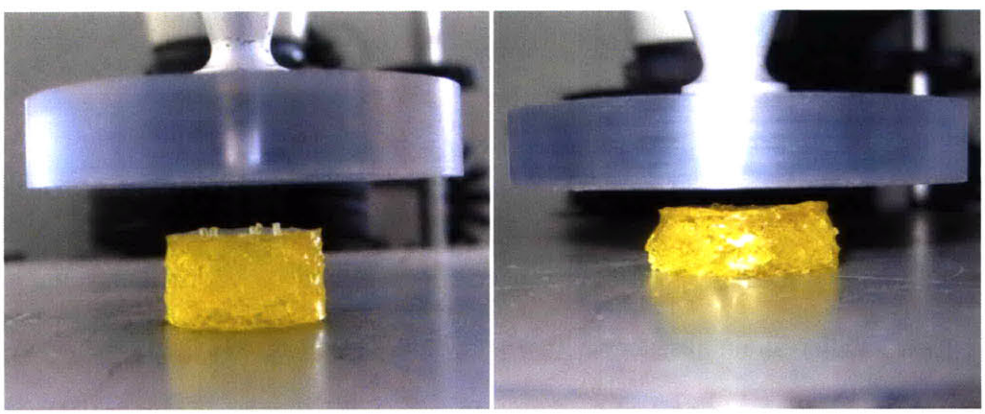


Figure 28: This Volume fraction of 0.09, cyclic loading (top). Pictures of the sample before and after the cyclic loading test are displayed in the bottom.

On the other hand, for large strain compression the composite material exhibits stress softening. As seen in the graph in figure 28, with every cycle of compression the maximum stress decreases. This means that the material is getting softer or less stiff after each cycle. A possible cause for this phenomenon can be that due the high strain, the particles start breaking internally after each cycle, thus become weaker overall. Furthermore, as seen in the pictures taken before and after the 7 cycles in figure 28, it is clear that some particle legs stick out before the first loading, after the first cycle excess particles were compressed inside the Jell-O. There are also buckling effects post-loading.

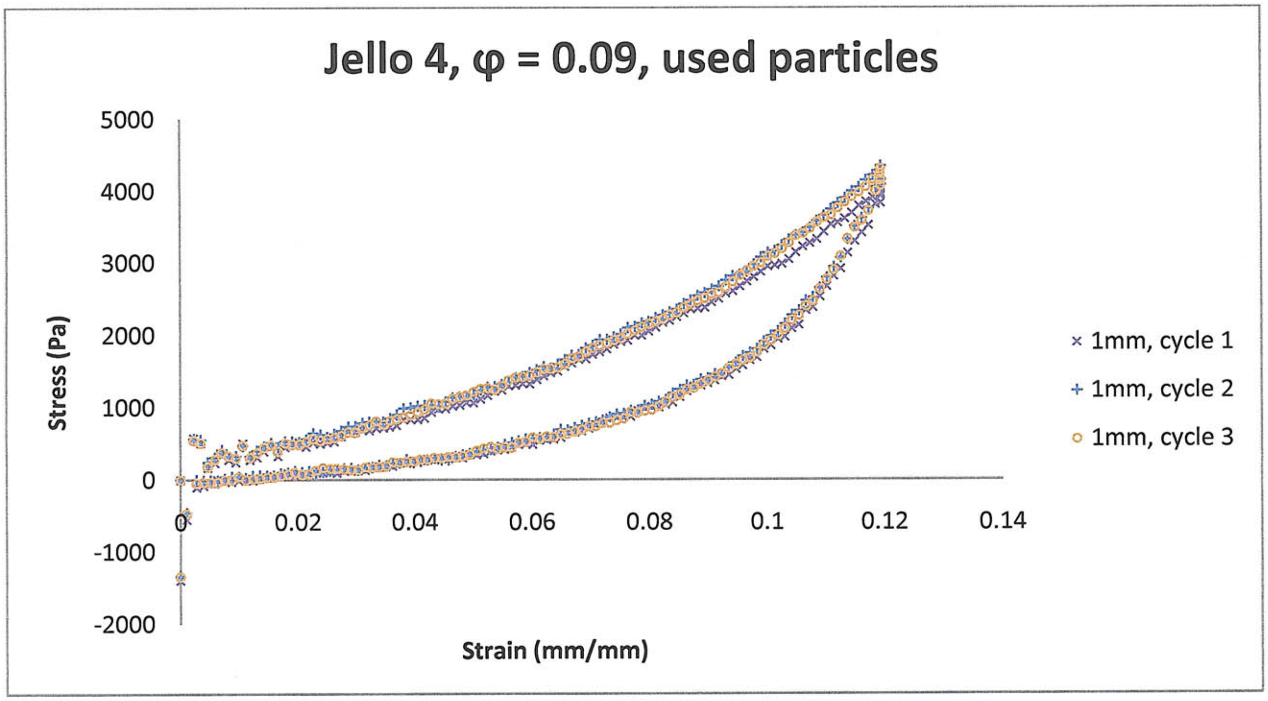


Figure 29: Volume fraction of 0.09, cyclic loading with used particles.

Unlike the samples in figure 26, 27 and 28, composite samples with smaller volume fraction and compressed to a smaller strain (as shown in figure 29) shows repeatable results in cyclic loading. Therefore, stress hardening and stress softening only occurs in large strain compression, large volume fraction, or both.

4.3 Used vs. New Particles

The cleaning process for these particles is long and tedious, and it would be ideal to be able to conserve the particles and reuse them. To validate the use of old particles, few samples were made with particles in previously compressed composite samples to compare to the samples made with new particles.

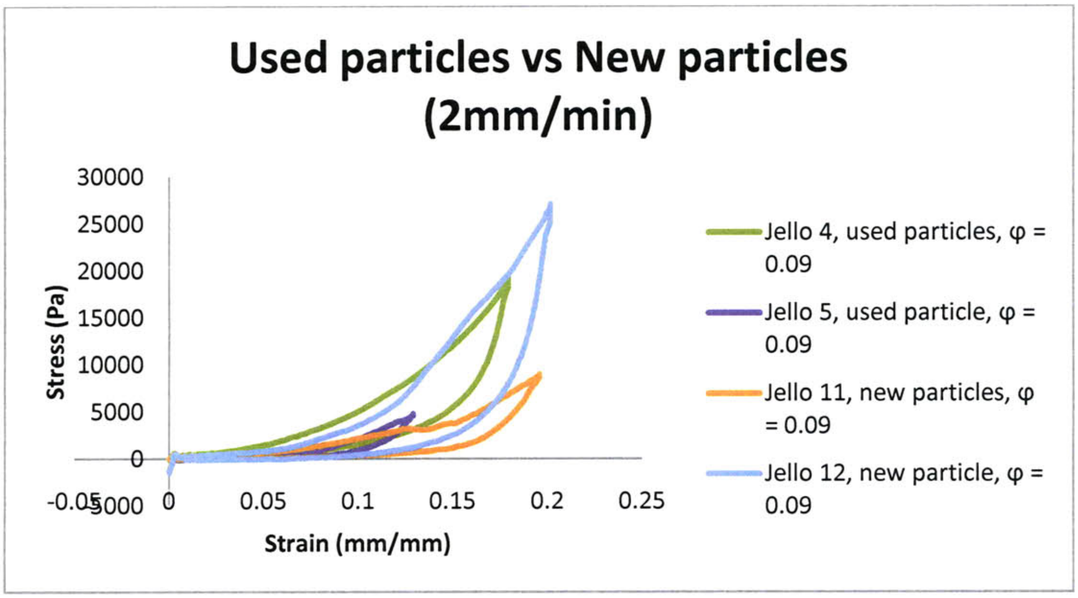


Figure 30: Volume fraction of 0.09, used particles vs. new particles.

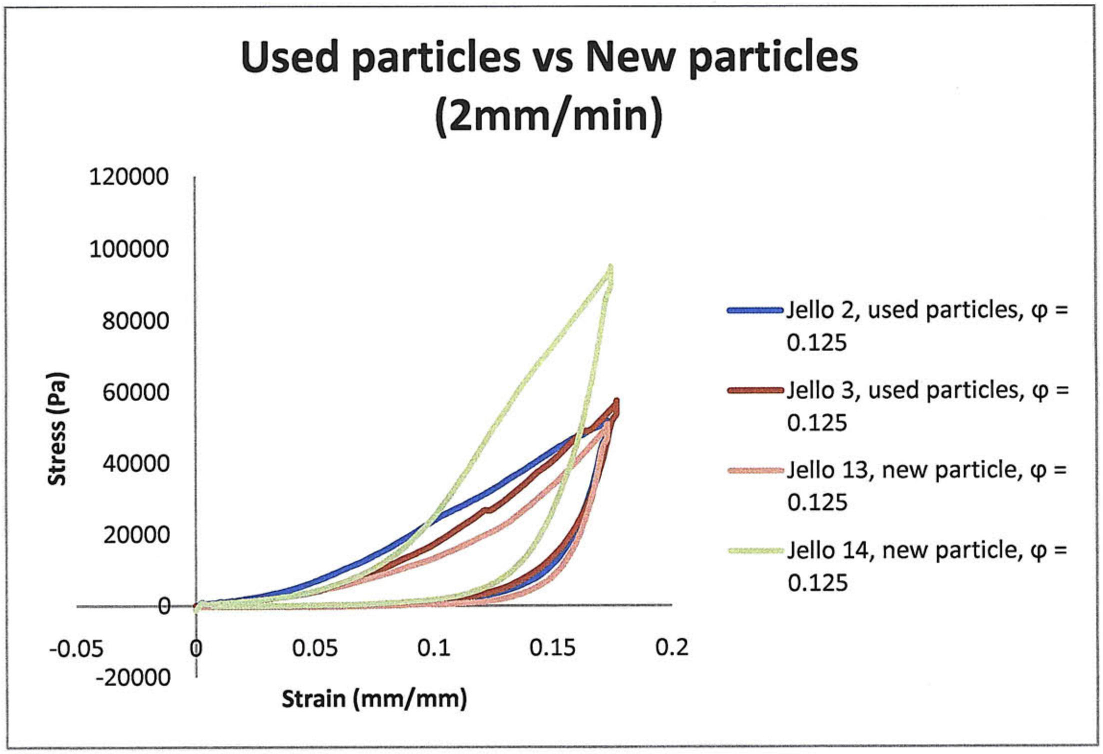


Figure 31: Volume fraction of 0.125. Used particles vs. new particles.

Figure 30 and 31 above shows the results with volume fractions of 0.125 and 0.09. Note that all samples were measured with a force trigger of 0.5g. In general all samples have similar stress-strain trends whether or not these are used or new particles. The used particles in this case underwent small strain compression before. Therefore, we conclude that there is no drastic material property change for used particles if the sample was previously compressed under small strain. The irregularity of the curves is probably a result of error discussed in section 5.

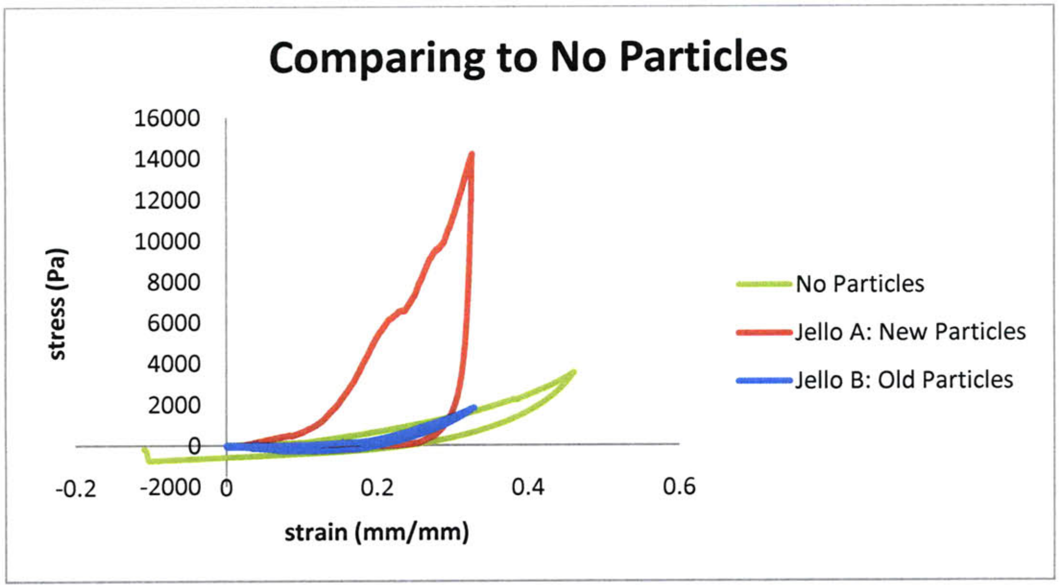


Figure 32: Samples with used particles compared to samples with no particles and new particles.

Figure 32 above, however, suggest that particles are not reusable. This is one of the earlier experiments that lead to exploration in particle reusability. Composite samples prepared with old particles seem to have a drastic decline in stiffness compared to composite samples made with new particles. The difference in these tests relative to the results from figure 30 and 31 above is that these used particles underwent large strain compression in their previous sample test. Thus, particles are reusable as long as they were previously embedded in samples that underwent low strain compression.

4.4 Ordered vs. Random

Throughout the study, the compartment of rigid particles and its effect on the material properties was also investigated. The concept of ordered packing and random packing is illustrated in figure 33 below.

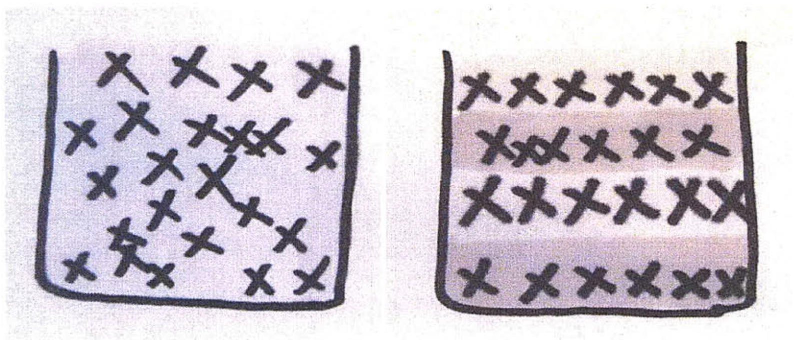


Figure 33: Illustration of random packing vs. layered packing.

Throughout the study all samples were prepared in random packing unless otherwise noted. Random packing is obtained by pouring all the particles into the mold all at once without interrupting the interactions between the particles. For ordered particle packing, the composite samples were prepared by layer. Each layer contains a 25 particles and a corresponding volume of gelatin solution depending on the desired volume fraction. Between the formation of each layer, the sample was cooled at 4°C allowing the gelatin to stiffen. The samples were then compressed with texture analyzer to collect force and displacement data. Figure 34 and 35 below compare the results for random packing and ordered packing.

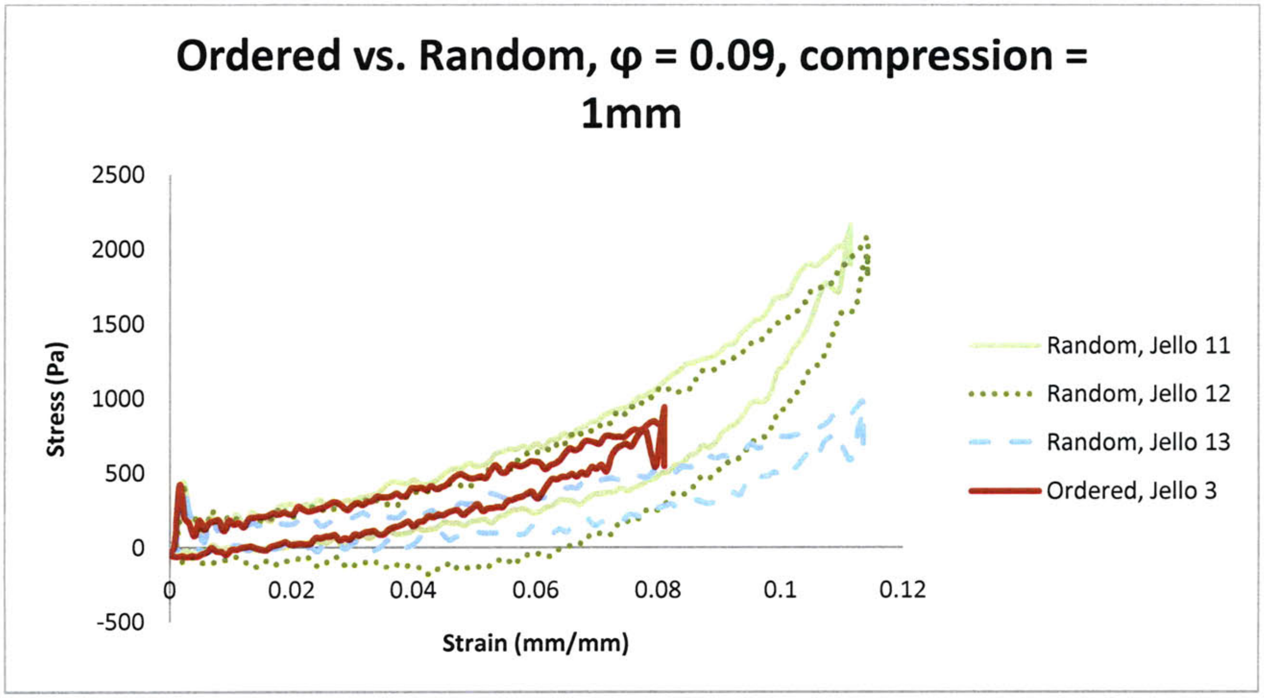


Figure 34: Ordered vs. random, comparison of samples with volume fraction of 0.09.

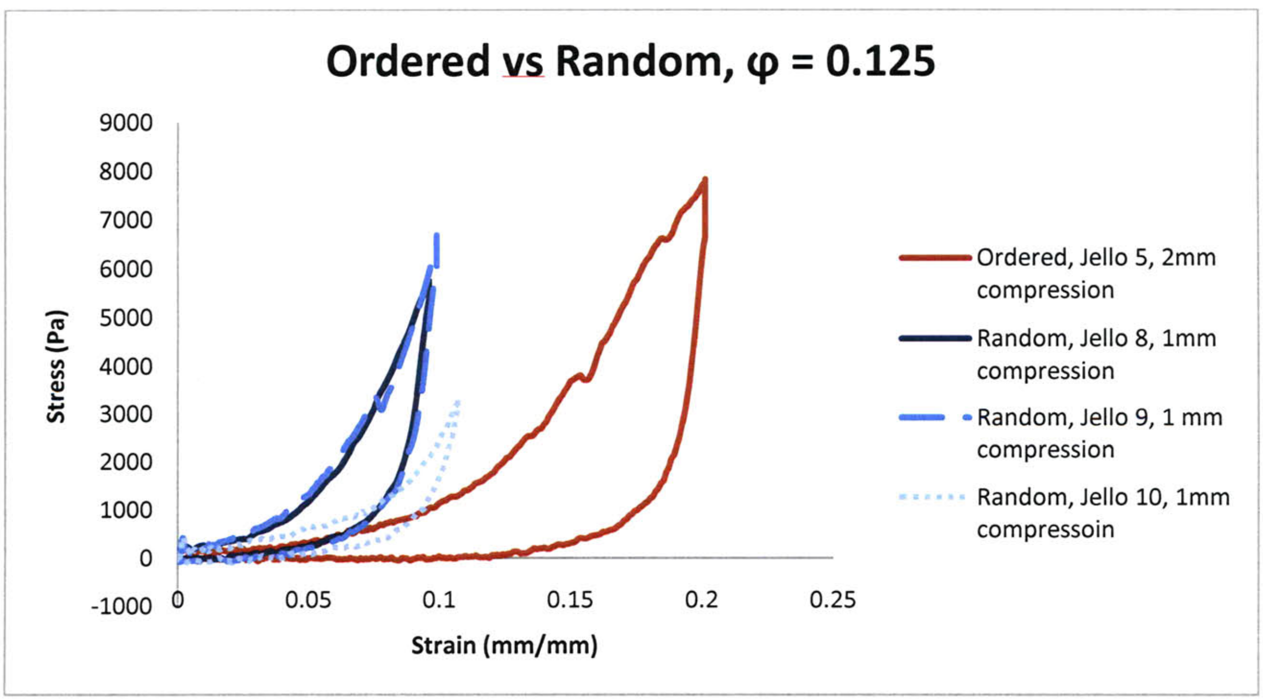


Figure 35: Ordered vs. random, comparison of samples with volume fraction of 0.125.

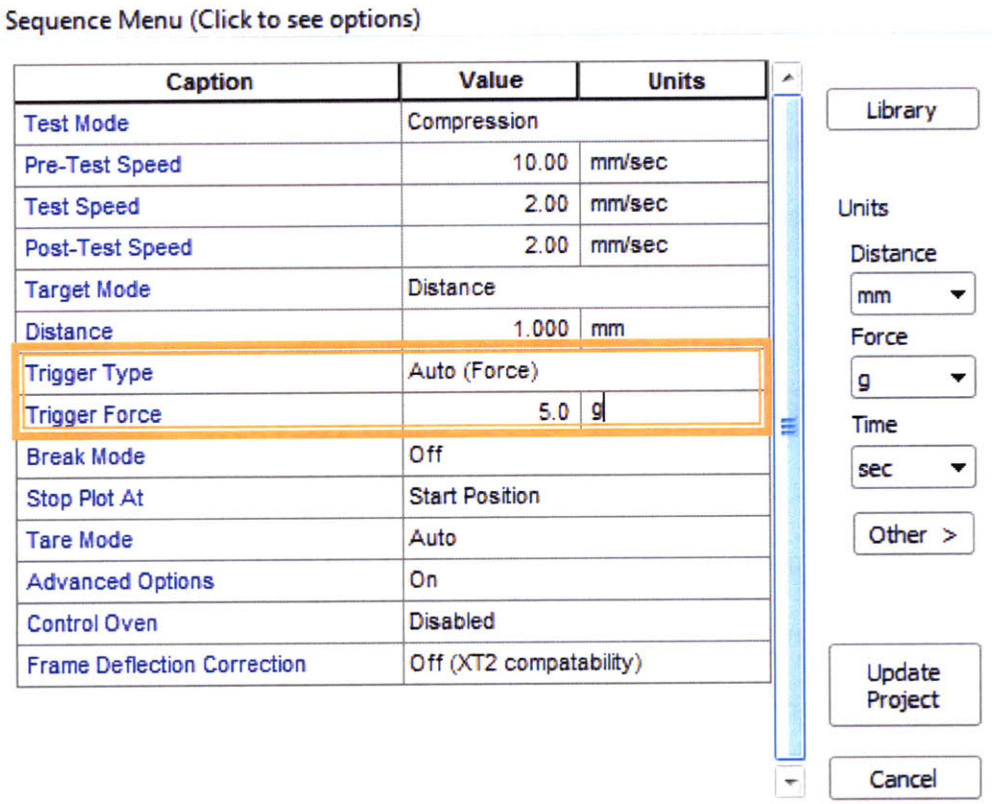
From the comparisons, there is no significant difference between random packing and ordered packing of particles in soft materials. The stress-strain behaviors for layered samples resemble that of the random samples. However, to be fully certain about this conclusion, further investigation would be needed due to the lack of sufficient data set. Only few samples were made and each sample behaves uniquely in its own way. In addition, there are many air bubbles in layered sample embedded within each layer due to the small volume added with pipette. This would definitely affect the volume fraction of rigid particles.

5. Discrepancy

There were many sources of discrepancy that were observed throughout the experiments. This is also the reason why many compression results are unique and difficult to draw definite conclusions based on the limited number of samples. A variety of possible error sources will be discussed in this session.

5.1 Experimental Parameters

Throughout the experiments, one of the most noticeable struggles was to decide on the type of trigger for data acquisition. "Button Trigger" was used for earlier experiments, whereas "Force Trigger" was used for later experiments. The desired type of trigger can be selected from the T.A. setting menu, as shown in figure 36 below.



Sequence Menu (Click to see options)

Caption	Value	Units
Test Mode	Compression	
Pre-Test Speed	10.00	mm/sec
Test Speed	2.00	mm/sec
Post-Test Speed	2.00	mm/sec
Target Mode	Distance	
Distance	1.000	mm
Trigger Type	Auto (Force)	
Trigger Force	5.0	g
Break Mode	Off	
Stop Plot At	Start Position	
Tare Mode	Auto	
Advanced Options	On	
Control Oven	Disabled	
Frame Deflection Correction	Off (XT2 compatability)	

Figure 36: Screenshot of T.A. Setting from Texture Exponent 32 software.

Button Trigger means that the computer will start collecting data as soon as "Run a test" is selected. Users typically adjust the probe position such that it is of certain distance above the sample. This way the texture analyzer will collect data points until the probe returns to its starting position. Even though Button trigger will allow identifying any shrinkage in samples

Force trigger; on the other hand, start collecting data when it detects a certain amount of force. The advantage of using force trigger is that users can accurately control the compression distance. However, if samples do not return to its original height after cyclic compression, there is no way in identifying shrinkage using force trigger. This would also affect all the cyclic strains, since the height is no longer the original height.

In addition, it is important to note that 2mm/min of loading speed and 10mm/min of unloading speed were used throughout the earlier experiments. This might also affect the accuracy of the compression data. Composite samples would return to its starting position a lot slower than the texture analyzer, resulting in overly steep unloading modulus.

5.2 Manufacturing Issues

Another major source of error roots from the imperfect sample manufacturing method. Throughout the many experiments, there were continuous exploration and improvement on making the composite samples. However, there is still not any consistent method to produce identical samples. Many problems exist in samples that affect the accuracy of the results.

5.2.1 Tilted Samples

Tilting is one of the most obvious discrepancies throughout the experiments. It is difficult to prepare perfectly horizontal samples. Figure 35 below shows some samples that exhibit such discrepancy.

Figure 37: Pictures of different tilted samples.

Figure 38: Residue left in mold. It is difficult to retrieve a perfectly shaped sample.

The tilting can be a result of numerous reasons. In the earlier sample preparation, a circular piece of wax paper was cut out to lie on the base of the mold. However, some gelatin solutions tend to leak underneath the piece of wax paper, resulting tilted sample when the sample stiffens and wax paper base is removed.

Another way to contribute to the tilting is the way sample is stored. It is possible to place sample on uneven racks or container. Lastly, the way samples are removed from the mold may affect the overall sample heights. It may happen in such a way that many residues from one side of the sample are left in the 3D printed mold, as shown in figure 38 above. It is difficult to get the whole sample out of the current mold design.

5.2.2 Residual Materials in Mold

It has been a repeating struggle to retrieve perfectly shaped samples. Either the samples came out slanted, or some parts are left sticking to the mold. As shown in the figure 39 below, it is possible for some composite materials to stick to the bottom of the mold, leaving a hollowed sample.

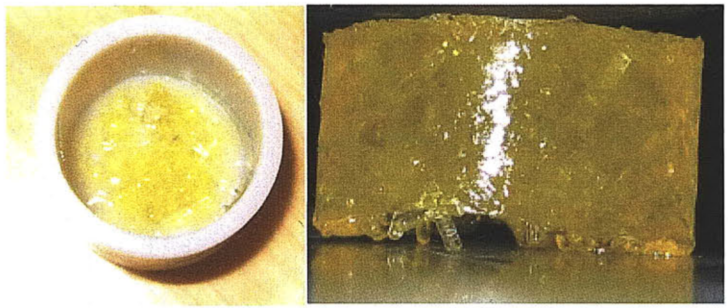


Figure 39: Picture of a hollowed sample and the residue left in the mold.

This phenomenon causes inaccuracy in material property calculation. The calculations throughout data processing are based on perfect cylinders. It also affects the accuracy of height measurement, even though heights are measured and averaged over 3 times.

5.2.3 Particles sticking out

Furthermore, because of the unique shape of the rigid particles used in these experiments, sometimes the composite specimens would have particle legs sticking out of the sample, as shown in figure 40 below.

Figure 40: Picture of particles sticking out

This occurrence is another source of error in the experiments. Often with large strains, the legs just get pushed after compression, which suggests that particles rearranged themselves. It was also difficult to retrieve data with small strain compression if the compression lengths were set to 1mm. the legs stick out for 1mm. This is also not a representation of compression area, as the probe is only compressing against the particles instead of the whole 20mm diameter wide composite surface. Lastly, it was difficult to decide whether the height measurement should include the legs that stick out or not.

5.2.4 Air Bubbles

Air bubble formation within the composite sample affects its volume fraction calculation. This is not as common in samples that contain random particles packing, as the air bubbles usually form on the top surface and it is easily detectable and preventable. However, with layered particles packing, an air bubble within the layers was a big concern.

Since the small volumes were pipette inside the sample mold, it was very easy to have a large number of small bubbles. In addition, since the volumes of each injection are so small, it is usually mixed with air from high pressure delivery. Such air bubble volume affects the overall volume of the composite specimen; thus altering the results of volume fraction comparisons.

5.3 Height Measurement

Besides the manufacturing defects mentioned in the above section, even with a perfectly prepared sample, it is difficult to obtain a standard measurement of the height. With the same amount of gelatin solutions and same number of particles, the composite samples may still vary in height. It is possibly due to the unpredictable interlocking arrangement of the particles, or simply the edge effect of the gelatin solution being poured into the mold. Figure 41 below shows a typical sample with volume fraction of 0.09. Gelatin solution tends to adhere to the inner wall of the mold, thus making the outer part of the composite sample slightly taller than the inner part, as shown in figure 41 below.

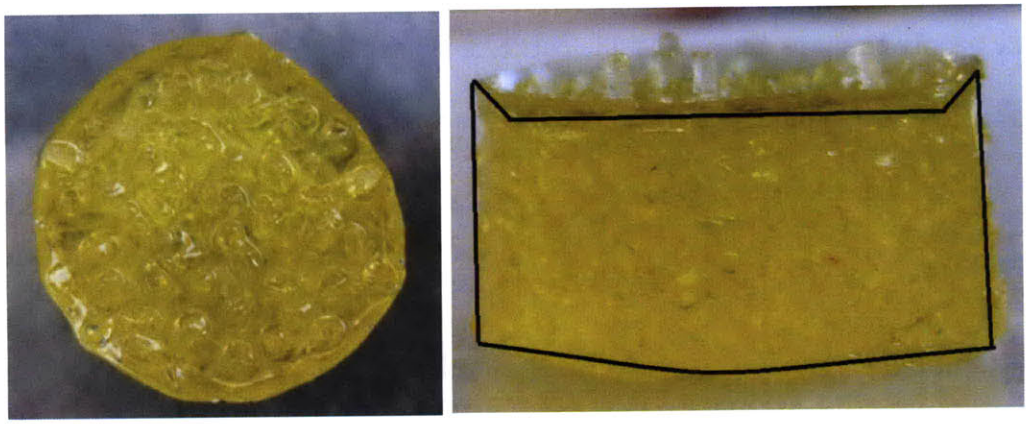


Figure 41: Picture of a typical composite sample. Note the surface roughness (left) and edge effect (right).

In addition, it is clear from the picture how rough the surface is. The roughness contributes to the uneven height measurement of the sample, which may lead to error in calculating strain. The height measurements used in all the data analyzes are typically an average value of 3 measurements. However, sometimes these 3 measurements are too different that it was hard to draw conclusion. The samples itself are very 'spongy', therefore it is possible that error could occur during the height measurement process.

6. Conclusion & Future Recommendation

In summary, volume fraction of rigid particles does increase the effective Young's Modulus of material. However, there were not enough results to draw a correlation on whether it is a linear relationship or exponential. For future exploration, more samples of different volume fractions can be made to investigate such correlations.

This study successfully improved the manufacturing method of the composite particles. The wax paper lining works well at times to pull out a whole sample. Nadia suggested trying a bottomless mold and hot glue it to a surface, and later after samples are set hot glue can easily be peeled off. This could be something to explore in the future to better the process in making consistent samples.

System compliance should also be taken into account in the future. It is important to record such data before every compression test. With the many compression data, it would also be helpful to write a Matlab script to aid in data processing in the future.

Whether or not different strain rates affect the material property is inconclusive. There are samples that suggest both sides of the arguments. Future work would also be needed in this area to further investigate.

Stress softening and stress hardening are both really interesting subjects that may be explored in the near future. Those were surprising results that were not expected, and do not appear on small strain and small volume fraction materials.

7. Acknowledgements

I would like to thank Professor Anette Hosoi for being my thesis advisor, supporting me along my thesis project and putting in the extra time to edit my thesis. Without her advices and input on the experimental results this project would not have gone this far.

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8. Reference

Koehl, M. A. R., *Mechanical Design of Spicule-reinforced connective tissue: Stiffness. J. exp. Biol.* (1982), 98, 239-267