Injection Well Drilling:
Effects of Crosslinking on Viscosity

Chemistry

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Abstract

Advances in hydraulic fracturing have lead to a boom in oil production, as oil is easier to obtain. Hydraulic fracturing works by pumping a mixture of water and chemicals into the earth to break apart the rock in underground wells to release the oil. An important property for the fracturing fluid to have is a high viscosity value. This may be obtained by the crosslinking between guar gum and boric acid that is controlled by pH. The focus of my experiment is to determine what conditions create the optimum gel crosslinked solution. The extended essay investigates the research question: “To what extent do different concentrations of the active ingredients in the polymer gel of guar gum affect crosslinking as measured in an adapted falling ball viscometer?”

The experiment tested nine trials of solutions with varied concentrations of guar gum and boric acid in increasing amounts of 1.0M sodium hydroxide. Each gel solution was tested in a falling ball viscometer I adapted to determine the time for three sizes of steel ball bearings to fall through 10cm of gel solution. A longer fall time is associated with more efficient crosslinking between the guar gum and boric acid. By analyzing the fall times of each concentration the most efficiently crosslinked gel solution was found.

Different concentrations of guar gum, boric acid, and the pH controller sodium hydroxide will determine how viscous the gel solution becomes. The investigation found that the highest concentration or percentage of guar gum tested (0.8%), the lowest concentration or percentage of boric acid tested (0.2%), and between two and three milliliters of 1.0M sodium hydroxide, combined to create the most efficiently crosslinked polymer.

Words - 275

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1.0 Introduction

Use of hydraulic fracturing, injection well drilling, or fracking, has caused much of the success of United States domestic oil and natural gas production. This method of extracting oil includes propelling high-pressure water and chemicals into underground wells. Chemical engineers adjust concentrations of these chemicals to manipulate the properties of the fracturing fluid to be best suited for the well. The fluid's viscosity or resistance to flow is manipulated by controlling the crosslinking of the polymer guar gum. The crosslinking process is controlled by pH because the borate ion acts as the crosslinking agent, and is produced in the equilibrium between boric acid and the borate ion. The three components in creating the crosslinking gel are: guar gum, boric acid and sodium hydroxide – a pH controller. Different concentration of each component will result in a different thickness of gel solution. The variability of the viscosity of the fluid is also important so the pH can be lowed to un-crosslink the fluid to remove it from the well. Knowing how each component affects the crosslinking of the polymer is required to manage the fluid for a fracturing well.

That said my extended essay will investigate: "To what extent do different concentrations of the active ingredients in the polymer gel of guar gum affect crosslinking as measured in an adapted falling ball viscometer?" Here I will test different concentrations of the chemicals: guar gum, boric acid, and sodium hydroxide, to experimentally determine each of their effects in thickening the polymer gel.

1.1 Background Information

Experimenting with this crosslinking polymer has led to my extensive knowledge of its characteristics. Through experimentation I have studied the effects of the polymer including: the nature of guar gum, boric acid-sodium hydroxide buffer, and initiation of crosslinking.

This fluid is unique because of its easily manipulatable viscosity. Viscosity is a unit of measure to describe the flow resistance of fluids and is calculated by the relation of sheer stress and sheer rate on the fluid. Different rheological models will give a viscosity value based upon the calculated slope of shear stress versus shear rate data. Newtonian fluids have a linear relationship between sheer stress and sheer rate; this makes their viscosity easier to find. Fracturing fluids (including the guar gum polymer) behave according to the power law fluid model. For non-Newtonian fluids, apparent viscosity (ưa) is determined to characterize the fluid, as they will not have a constant viscosity value under different conditions. A rheometer can find a fluid's apparent viscosity value, but testing may still be troublesome. "The testing problem is compounded in that many fracturing fluids (particularly crosslinked gels) are not truly fluids. Trying to characterize these materials with a “viscosity” can be very difficult" (Montgomery, 2013). The information from Montgomery, 2013 comes from a secondary source as a published textbook titled *Effective and Sustainable Hydraulic Fracturing*. This source provided invaluable information to the investigation and may be trusted as a published professional research guide.

A rheometer was unavailable to find a viscosity value for the polymer; therefore separate experiments were devised to evaluate the crosslinking efficiency of the polymer. For power law fluids the apparent viscosity will decrease with increasing shear rate (Montgomery, 2013). Through qualitative observations this process was observed.

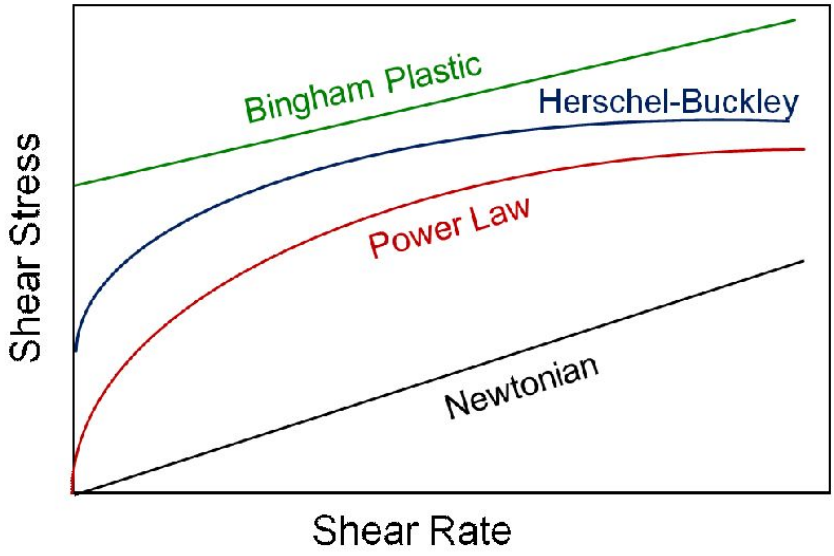


Figure 1: Calculations of Viscosity - (Montgomery, 2013)

1.2 Guar Gum

Guar gum is one derivative from the Guar plant. “The guar gum molecule is a linear, or highly anisodimensional carbohydrate polymer with a molecular weight on the order of 220,000” (Meer 1962). The chemical structure of guar is unique in that it will undergo complexing reactions through the cis-hydroxyl functionality (Montgomery, 2013). “This complexing reaction leads to crosslinking of the molecules resulting in a three dimensional network which manifests itself in gel formation” (Chudzikowski, 1971). Different concentrations of guar gum and crosslinking agents will affect the specific characteristics of the gel. “Guar products are considered to be nonirritating to eyes and skin, based on animal tests. It is nonhazardous, but proper care should be used to prevent inhalation of the product dust” (Beckwith, 2012).

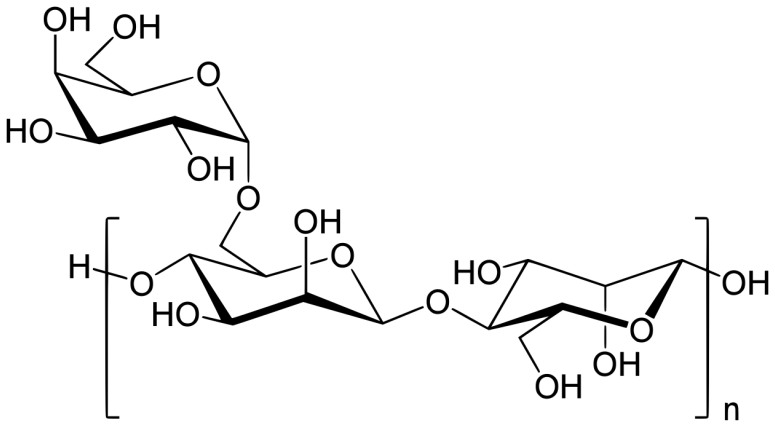


Figure 2: Molecular structure of guar gum

1.3 Borate Ion

The borate ion is a crosslinking agent, capable of facilitating crosslinking between the guar gum molecules. The borate ion can be produced through the equilibrium of boric acid and hydroxide ion, shown as the net ionic equation.

$$B(OH)_3\ (aq) + HO^-\ (aq) \Leftrightarrow B(OH)_4^-\ (aq)$$

The complete ionic equation equilibrium takes place both with water, and other bases.

$$B(OH)_3\ (aq) + 2H_2O\ (aq) \Leftrightarrow B(OH)_4^-\ (aq) + H_3O^+\ (aq)$$

$$B(OH)_3\ (aq) + NaOH\ (aq) \Leftrightarrow B(OH)_4^-\ (aq) + Na^+\ (aq)$$

As the hydroxide ion concentration is increased (the pH is raised), the equilibrium shifts to increase the concentration of the borate ion. Lowering the pH will shift the equilibrium to lower concentrations of the borate ion. When pH is ≥8 the concentration of borate ions is high enough to begin crosslinking; the crosslinking will revert when pH is ≤7 (Chudzikowski, 1971; Montgomery, 2013). This is the thinking behind the entire process: that by controlling the pH the crosslinking and resistance to flow of the polymer can be controlled.

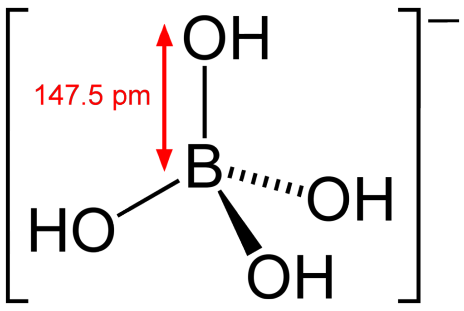


Figure 3: Tetrahedral structure of borate ion

Safety precautions for handling boric acid include an apron and protective eye wear. In case of skin contact, immediately flush with plenty of water and cover irritated skin with emollient (ScienceLab.com, 2013). See appendix A for full boric acid MSDS.

Safety precautions for handling sodium hydroxide pellets include an apron, protective eye wear and rubber gloves. In case of contact, immediately flush skin with plenty of water for at least 15 minutes and get medical attention immediately (ScienceLab.com, 2013).

1.4 Crosslinking

The borate ion has a tetrahedral form (figure 3) that will complex with the hydroxyl functionality on the guar gum polymer backbone to form a three-dimensional network (Montgomery, 2013). Borate is an efficient crosslinking agent as “borate gels reheal under shear stress because the borate crosslinks break and reform continually, with very little degradation” (Wiskofske 1997). Crosslinking increases the molecular weight of the molecule, that will increase the base viscosity of the polymer between x2 - x20 times (Montgomery, 2013). The cis-hydroxyl groups of two guar gum molecules will be connected by the hydrated borate ion (figure 4). For each borate ion that is fully crosslinked with the guar gum, four water molecules are also created. Each molecule of guar gum may create several links to other molecules. This is crosslinking taking place.

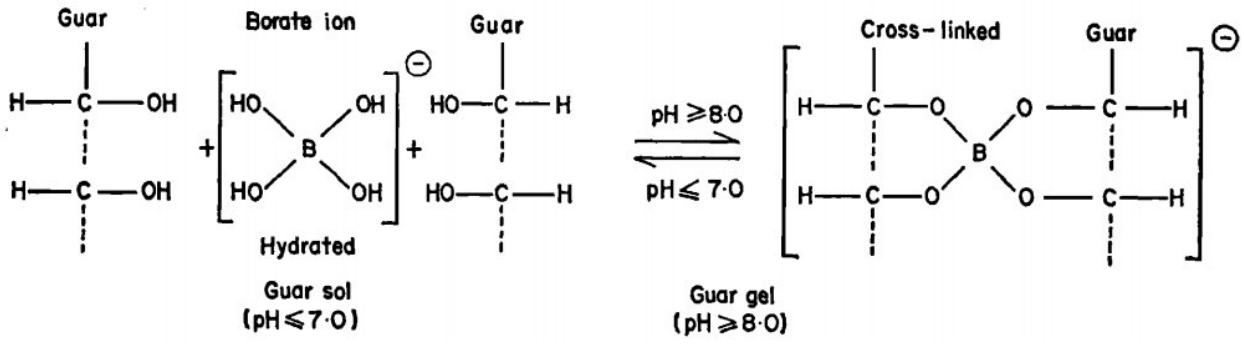


Figure 4: Crosslinking of borate ion with guar gum

2.0 Investigation / Method

A rheometer is used to find an apparent viscosity of a power law fluid for industry testing, but I was unable to access one, so I devised my own experiments to test for variations in thickness of the gel. This research provided a primary source from which I evaluated data. This is an adaption of a vertical falling ball viscometer – which normally gives the viscosity value for Newtonian fluids – to measure the time taken (in seconds) for a steel ball to flow through the polymer gel solution. The vertical falling ball viscometer is reasonable to use within the lab setting. When all other conditions are kept constant, the more time it takes for the steel ball to flow through the viscometer, the more effective crosslinking should be in the polymer. This data will allow evaluation of which concentrations result in the most effective crosslinking.

The experiments are designed to manipulate one variable at a time, testing to find the affect each mechanism has on producing the polymer of guar gum. The species that affect the thickening of the polymer are: guar gum, boric acid, and sodium hydroxide. Guar gum and boric acid are initially mixed in solution then sodium hydroxide is incrementally added to induce crosslinking. Over 9 trials with different concentrations of guar gum and boric acid, trends in the data suggest how each species determine the thickness of the gel.

The effect of either guar gum or boric acid is found by looking at the trials that keep one concentration constant and vary the concentration of the other. Sodium hydroxide is incrementally added to each solution, then the solution is tested, so the thickness of each trial is seen for each concentration of sodium hydroxide.

As each trial of the polymer is prepared, the experiments were supplemented with qualitative observations. Observations included: how the polymer reacted to sheer stress, the Weissenberg effect, and the ability for it to be lifted up on a stirring rod.

2.1 Solutions

To find the difference, different concentrations of guar gum and boric acid had on the crosslinking, the procedure tested combinations .2%, .5%, and .8% concentrations of guar gum and boric acid with each other. This resulted in nine different final solutions containing different concentrations. 300ml of each solution were made and tested. 1M sodium hydroxide was added in 1ml amounts, up to 5ml, to each of the solutions.

250ml boric acid solutions (see appendix A for solution specifics) were made for each desired concentration from crystallized boric acid in volumetric flasks. These solutions were concentrated enough that 27ml of the boric acid solution, added to 273ml of guar gum solution, would yield the desired concentration of boric acid in the 300ml solution (.2%, .5%, and .8%).

The guar gum was kept in powder form until ready to make the gel solution for the test. Each solution of guar gum was prepared by massing the amount of guar gum needed to make the desired concentration (.2%, .5%, and .8%) in 300ml, and added that amount to 273ml of distilled water. When the 27ml of the desired concentration of boric acid solution was added, the 300ml solution is ready for testing.

Solutions were made in 400ml beakers, using 100ml (± 0.5ml) graduated cylinders, 10ml (± 0.05ml) graduated cylinders, and an electronic balance (± 0.01g). A 500ml (± 0.1ml) volumetric flask was used to make the initial boric acid solutions.

The procedure prepared a 250ml, 1M Sodium hydroxide solution using a 250ml (± 0.1ml) volumetric flask. Using a 1ml (± 0.01ml) volumetric syringe, the sodium hydroxide solution was added in 1ml increments to each guar gum and boric acid solution.

After the first milliliter of sodium hydroxide was added, the solution was stirred vigorously for five minutes, until the gel solution reached a homogenous consistency (does not have a marbled appearance). After each successive milliliter was added, it was stirred vigorously for one minute or until homogenous again.

2.2 Testing Apparatus

The procedure utilizes an adapted traditional falling ball viscometer to find the time taken for a steel ball to flow through the gel solution (Krauser, 2013). The procedure used a double-open-ended glass cylinder tube with a diameter of 3.5cm, marked along the side from 0cm to 35cm going up the tube. The bottom of the tube was plugged with a rubber stopper and was held up on a ring stand with a test tube clasp. When the 300ml of test gel solution was added to the tube, the volume filled to about the 30cm line.

Three sizes of balls were tested by the procedure, a small, medium and large ball (see appendix B for specific measurements). The ball was positioned immediately under the top of the test solution/gel (held by a large magnet on the outside of the tube), and then allowed to fall through the solution. This was to assure the ball was falling at a constant rate when it was measured. The procedure then measured the time taken for the steel balls to fall 10cm, between the 10cm and 20cm lines; these lines were marked clearly to see exactly when the balls were passing through.

To record the time taken for a steel ball to fall thorough the tube, the procedure video recorded fall time or a watch was used. When the ball fall time was less than 600.0sec (10min), the procedure used the iPhone video app and iMovie software to find the exact fall time for each ball in seconds to one decimal place (± 0.1sec). When the ball fall time was greater than 600.0sec (10min), the procedure used a watch to calculate start and stop time and the total fall time of the ball.

3.0 Results

3.1 Data tables

All three sizes of ball bearings were recorded in one trial, however the data is more easily displayed by collecting the results for each size ball individually. The raw data tables as they were recorded can be found in Appendix C.

The volume of sodium hydroxide that was added to the particular solution increases to the right across each table. Each trial of differing concentrations of guar gum (GG) and boric acid (BA) are seen moving down each table. The measurements are the time (in seconds, measured to one decimal point) that each ball took to fall through 10cm length of the solution. Some times are missing and denoted by a “n/a”. These times took longer than could be accurately measured within the capabilities of this project.

Small Ball Trials						
Volume NaOH added (ML-1M)	*0*	*1*	*2*	*3*	*4*	*5*
0.2GG-0.2BA	0.1	0.2	2.0	4.7	14.7	24.0
0.2GG-0.5BA	0.1	0.4	3.1	8.1	4.9	5.4
0.2GG-0.8BA	0.1	0.2	1.6	4.5	8.9	8.9
0.5GG-0.2BA	0.1	10.4	552.0	247.0	214.0	1910.0
0.5GG-0.5BA	0.2	17.2	109.0	74.0	208.0	408.0
0.5GG-0.8BA	0.2	1.7	11.2	37.2	374.0	n/a
0.8GG-0.2BA	0.2	22.6	690.0	2640.0	n/a	n/a
0.8GG-0.5BA	0.2	9.5	171.0	600.0	1770.0	n/a
0.8GG-0.8BA	0.2	11.2	80.0	230.0	430.0	n/a

Medium Ball Trials						
Volume NaOH added (ML-1M)	*0*	*1*	*2*	*3*	*4*	*5*
0.2GG-0.2BA	0.1	0.2	0.5	1.1	1.0	0.8
0.2GG-0.5BA	0.2	0.3	1.8	0.8	0.5	0.7
0.2GG-0.8BA	0.2	0.2	0.5	0.9	0.6	1.1
0.5GG-0.2BA	0.2	6.1	43.8	49.7	36.1	43.1
0.5GG-0.5BA	0.3	9.2	33.7	18.7	30.8	33.9
0.5GG-0.8BA	0.2	1.3	6.9	18.7	143.0	455.0
0.8GG-0.2BA	0.3	26.8	264.0	1070.0	780.0	603.0
0.8GG-0.5BA	0.3	5.7	88.0	225.0	350.0	515.0
0.8GG-0.8BA	0.3	9.0	48.1	125.0	210.0	315.0

Large Ball Trials						
Volume NaOH added (ML-1M)	*0*	*1*	*2*	*3*	*4*	*5*
0.2GG-0.2BA	0.4	0.6	1.8	13.3	17.0	19.8
0.2GG-0.5BA	0.3	1.3	6.3	14.2	25.2	32.1
0.2GG-0.8BA	0.4	0.5	2.1	10.6	17.0	20.6
0.5GG-0.2BA	0.5	31.2	964.0	359.0	809.0	1080.0
0.5GG-0.5BA	0.4	52.5	222.0	161.0	302.0	606.0
0.5GG-0.8BA	0.4	7.6	39.7	88.0	660.0	n/a
0.8GG-0.2BA	0.5	175.0	1350.0	n/a	n/a	n/a
0.8GG-0.5BA	0.4	38.4	495.0	930.0	1800.0	n/a
0.8GG-0.8BA	0.5	54.2	194.0	310.0	n/a	n/a

Approximate pH values were obtained for each solution using pH litmus paper. These values may vary more due to uncertainly in measuring.

pH						
Volume NaOH added (ML-1M)	*0*	*1*	*2*	*3*	*4*	*5*
0.2GG-0.2BA	5	7	9	9	9	9
0.2GG-0.5BA	5	7	8	9	9	9
0.2GG-0.8BA	5	7	8	9	9	9
0.5GG-0.2BA	5	7	9	9	9	9
0.5GG-0.5BA	5	8	8	8	9	9
0.5GG-0.8BA	5	7	8	8	8	8
0.8GG-0.2BA	5	8	9	9	9	9
0.8GG-0.5BA	5	7	8	8	9	9
0.8GG-0.8BA	5	7	8	8	8	9

3.2 Qualitative Data

The full transcript of all qualitative observations can be found in appendix D.

One of the key observations relates to the usefulness of the different size ball bearings used. The idea for the falling ball viscometer is to evaluate the fall time of the ball through the liquid as if it were falling unhindered in a large volume. The diameter of large ball bearing, however, was so close to the diameter of the tube, that the fluid's flow was hindered by the inability to flow around the ball as if it were in a larger volume. The trials including the large ball bearing are interesting comparisons but the data gathered from these trials may not be effectively evaluated.

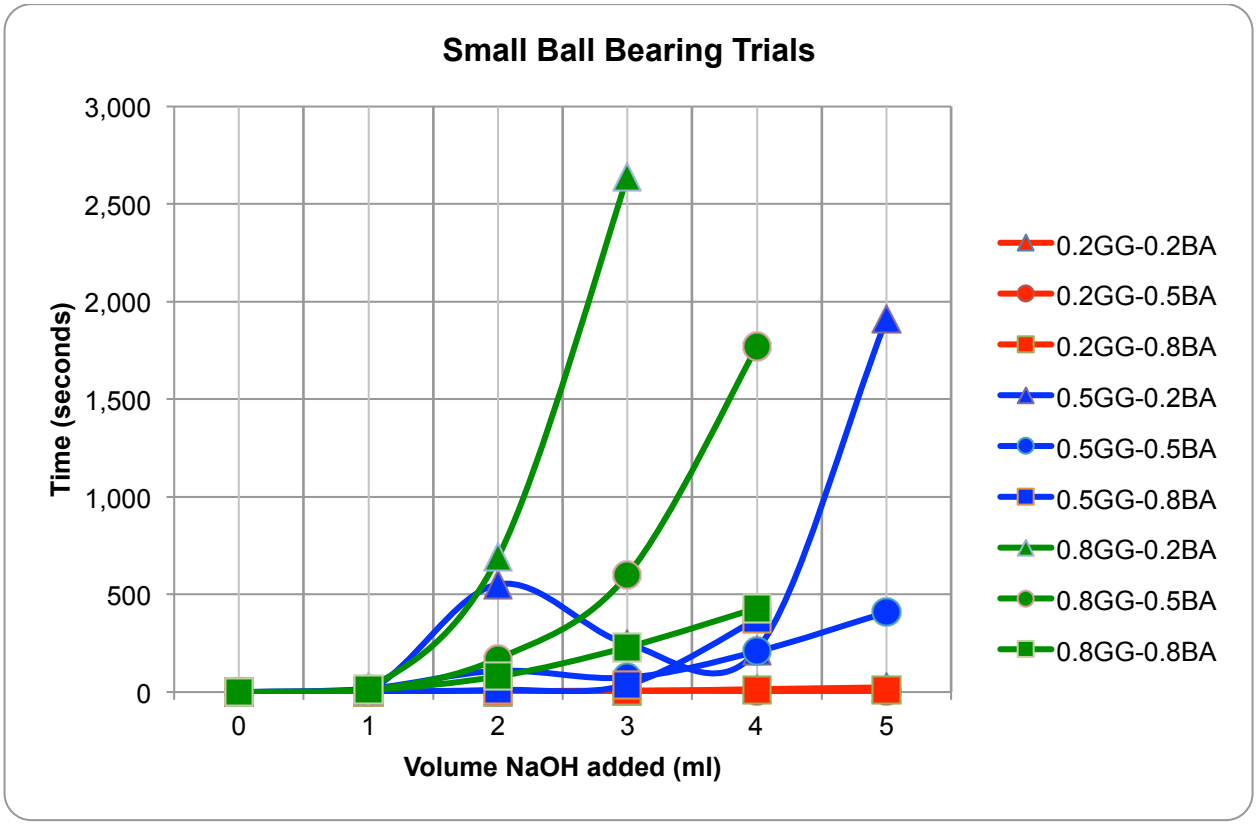
In all qualitative observations the characteristics of each solution was monitored. In addition to the main trials, a few tests were applied to each gel as different indicators of the presence of crosslinking. One of these tests was to see how well the solution would "stick together" or "clean itself up;" these were qualities of the gel forming. This

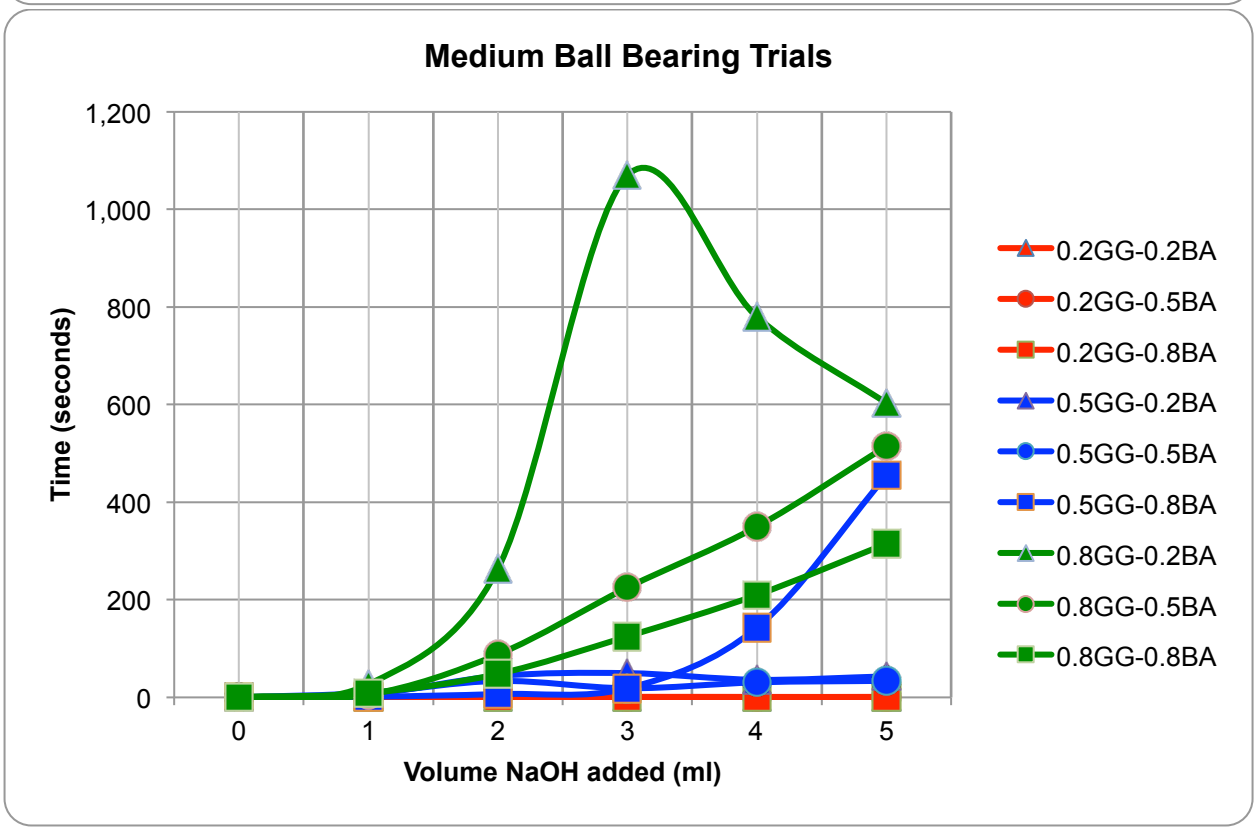
behavior was not noticed in the 0.2% guar gum trials, but was evident generally after the second milliliter of sodium hydroxide was added to the solution. Another test for crosslinking efficiency is to test for the Weissenberg effect. This was done by spinning the stirring rod in the beaker of solution and seeing if the gel solution would climb and spin up the rod. This was most often seen to take place after the third milliliter of sodium hydroxide was added, but often not seen after the fourth and fifth milliliter of sodium hydroxide.

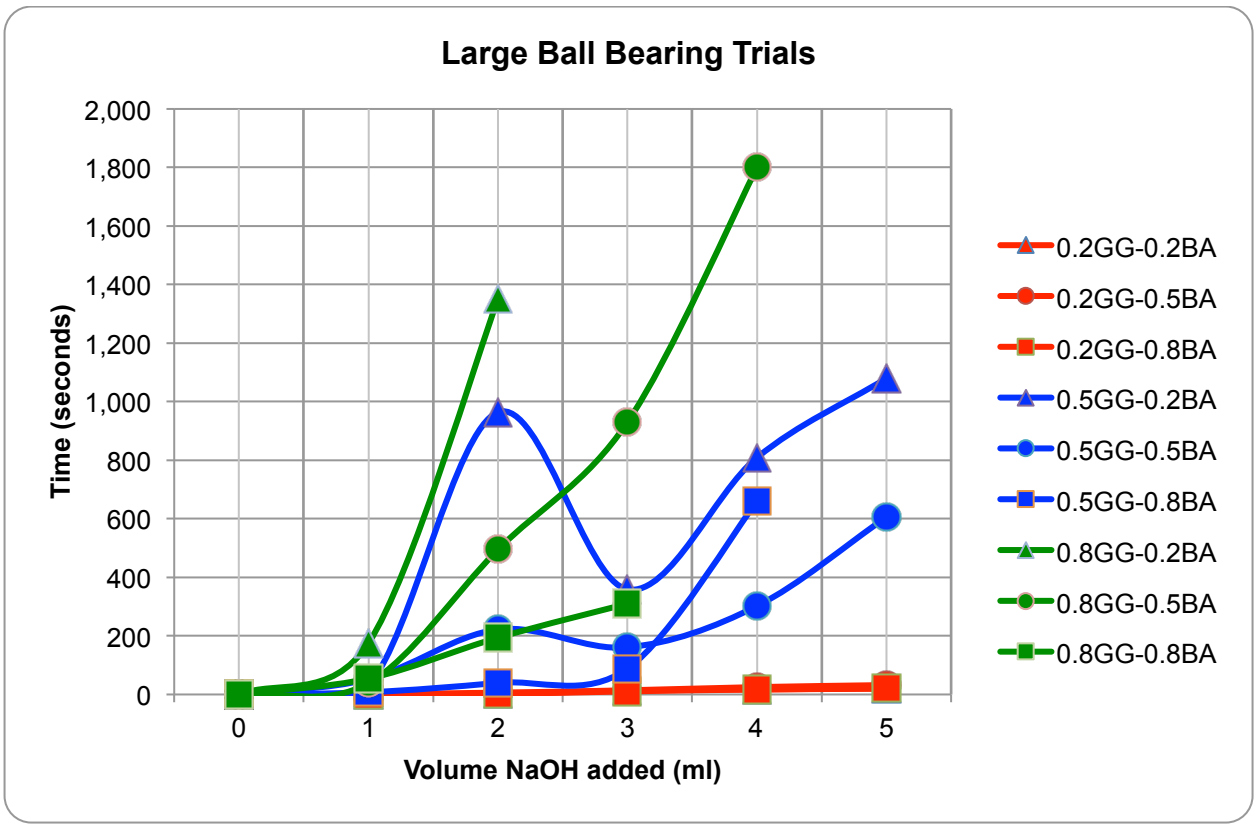
A good indicator of how thick the gel solution, and how effective the crosslinking, was how the solution acted when stirred and sheer stress was applied. Between when the second and third milliliter of sodium hydroxide was added – for 0.5% and 0.8% guar gum – the gel solution would stay together and move as one “blob,” unaffected by how much sheer pressure was applied. After the fourth or fifth milliliter of sodium hydroxide was added this changed so that when stirred the solution would “break” apart and stir as little clumps; this was also brought on by more applied sheer stress; the solution would then reform into one “blob” if left alone for a few minutes. This change in the solution also accompanied other changes, such as the test to see if the gel solution could be supported and held up along the stirring rod. Before the solution started to break, it was easy to support on the stirring rod. After the fourth and fifth milliliter was added to the solution, it would not be supported by the stirring rod and would break off.

This change can also be seen in some of the times for the main experiment. In some of the trials, there is a clear relative maximum at two or three milliliter of sodium hydroxide added, then a slight dip before rising again. This is most pronounced in the 0.8% guar gum and 0.2% boric acid trial for the medium ball bearing.

3.2 Graphed data






Large Ball Bearing Trials
Time (seconds)
2,000
1,800
1,600
1,400
1,200
1,000
800
600
400
200
0
0
1
2
3
4
5
Volume NaOH added (ml)
0.2GG-0.2BA
0.2GG-0.5BA
0.2GG-0.8BA
0.5GG-0.2BA
0.5GG-0.5BA
0.5GG-0.8BA
0.8GG-0.2BA
0.8GG-0.5BA
0.8GG-0.8BA

4.0 Analysis

The impact that higher concentrations of guar gum have on the thinness of the gel solution polymer is striking. Each ball took the longest time falling through the solutions with 0.8% guar gum, next longest in 0.5% guar gum solutions and the shortest time in the 0.2% guar gum solutions. This is seen by how the green lines on each graph has a larger slope and the values are greater than the other colored lines. The fall times for the 0.2% guar gum do begin to increase, but the slope is so low there is hardly an increase. The straight red line at the bottom of each graph represents this. Compared to the higher concentrations of guar gum, the liquid may have thickened but crosslinking did not take place.

This outcome can be explained by why the crosslinking of guar gum increases the viscosity of a liquid. Crosslinking works by increasing the overall molecular weight of the combined molecule (Montgomery, 2013). The guar gum provides the weight in this circumstance, so it is the main determining ingredient in how much crosslinking will take place. When there was not enough guar gum present to crosslink, it merely acted as a thickening agent.

The concentration of boric acid had a different effect on the crosslinking efficiency of the polymer. Evident in the 0.5% and 0.8% guar gum trials, as the boric acid concentration increased the time it took for the ball bearings to fall through the polymer decreased, not by a lot, but still a significant difference. The graphs provide this observation as within each color: the triangle doted line has the highest values, then the circle doted line and finally the square dotted line. This identifies that more effective crosslinking was produced by a lower concentration/ percentage of boric acid.

This small dip in crosslinking efficiency with increasing concentrations of boric acid may be explained by two chemistry principles. The first is the notion that boric acid is already in excess. More boric acid is not needed in order to fully crosslink with the available guar gum. Second is the notion that the crosslinking is more effective at a higher pH and that excess boric acid will only further lower the pH. As seen in the pH table: the pH becomes higher faster (likely high in general) in the trials of 0.2% boric acid. It also takes longer for the pH to increase in the trials that use 0.5% and 0.8% boric acid. This difference in the pH is enough to be noticeable. Therefore in an optimally crosslinked polymer, the boric acid concentration will be low.

Sodium hydroxide had the intended effect of inducing crosslinking by raising the pH. This is evident as in general there is a positive slope moving right across each graph. What was not expected is that in some of the data there is a noticeable peak or relative maximum in the data before decreasing and increasing again. This is unlikely to be error in the experiment as this change also accompanied a qualitative change in the observed polymer. This peak is most directly seen in the medium ball bearing graph trial of 0.8% guar gum and 0.2% boric acid. It is also seen to a lesser extent in the trials of 0.5% guar gum and 0.2% boric acid in all three ball tests.

This relative maximum was generally seen when the second or third milliliter of 1.0M sodium hydroxide was added, seen on each graph. The polymer at these peaks was observed to be collected into one “blob.” After the peak, as additional sodium hydroxide is added, the gel solution breaks apart more easily and resembles a chunky mixture. For an optimum gel solution, this procedure suggests the addition of between two and three milliliters of 1.0M of sodium hydroxide.

5.0 Conclusion

“To what extent do different concentrations of the active ingredients in the polymer gel of guar gum affect crosslinking as measured in an adapted falling ball viscometer?” This investigation tested different concentrations of guar gum, boric acid, and sodium hydroxide – the active ingredients in the crosslinking of the polymer of guar gum – to experimentally determine the effects each had in creating the most efficient crosslinking. It was found that the highest concentration or percentage of guar gum tested (0.8%), the lowest concentration or percentage of boric acid tested (0.2%), and between two and three milliliters of 1.0M sodium hydroxide, combined to create the most efficiently crosslinked polymer. It was found that guar gum is the main active ingredient in determining the efficiency of crosslinking.

6.0 Evaluation and Improvements

6.1 Random error

Random error present in the procedure came from the testing apparatus. The falling ball viscometer is an economical and easily available testing procedure, but has other limitations. Inconsistencies in how the balls were falling through the gel may have caused error. Air bubbles trapped in the polymer may have also caused this.

Improvement: The easiest solution would be to use a rheometer to measure the relative viscosity of the gel solution. Using the same procedure to reduce this error, confirm the solution is homogenous and as many air bubbles have been removed as possible.

6.2 Systematic error

The time recording system may be prone to systematic error as it was not made to handle this kind of data processing. The wrong time may have been recorded due to inconsistencies in this instrument.

Improvement: There is other software available to use better suited to time this kind of data. Using these programs would help reduce systematic error from these instruments.

6.3 Reliability of sources

The references used in this essay were from sources including: books, prior research, professional journals and chemical research databases. Each of these sources are peer reviewed and verified as reliable from other experts within their field. The research upheld principles of the chemistry of the reactions, including the pH range where crosslinking began as said in Montgomery (2013). These are well known academic publications written by qualified authorities.

6.4 Unresolved questions

What caused the relative maximum for crosslinking efficiency and qualitative change in the polymer between the second and third milliliter of sodium hydroxide?

An explanation for why this change occurred in the polymer, was not found by this investigation. Further research is needed to see if this was error in the procedure or what may have caused this. The qualitative change is verifiable, but can not be explained.

How would the crosslinking have changed with further addition of sodium hydroxide?

Each gel solution in this investigation was tested with up to 5ml of 1.0M sodium hydroxide. The highest approximate pH indicated using pH paper was a 9. Montgomery, 2013, says that the optimum crosslinking efficiency occurs at a pH of about 10.5 and at pH's higher than 10.5 the crosslinking will decrease. It would be interesting to see if this could be observed with this procedure or if any other variations occur like the relative maximum that was found in this procedure.

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8.0 Appendices

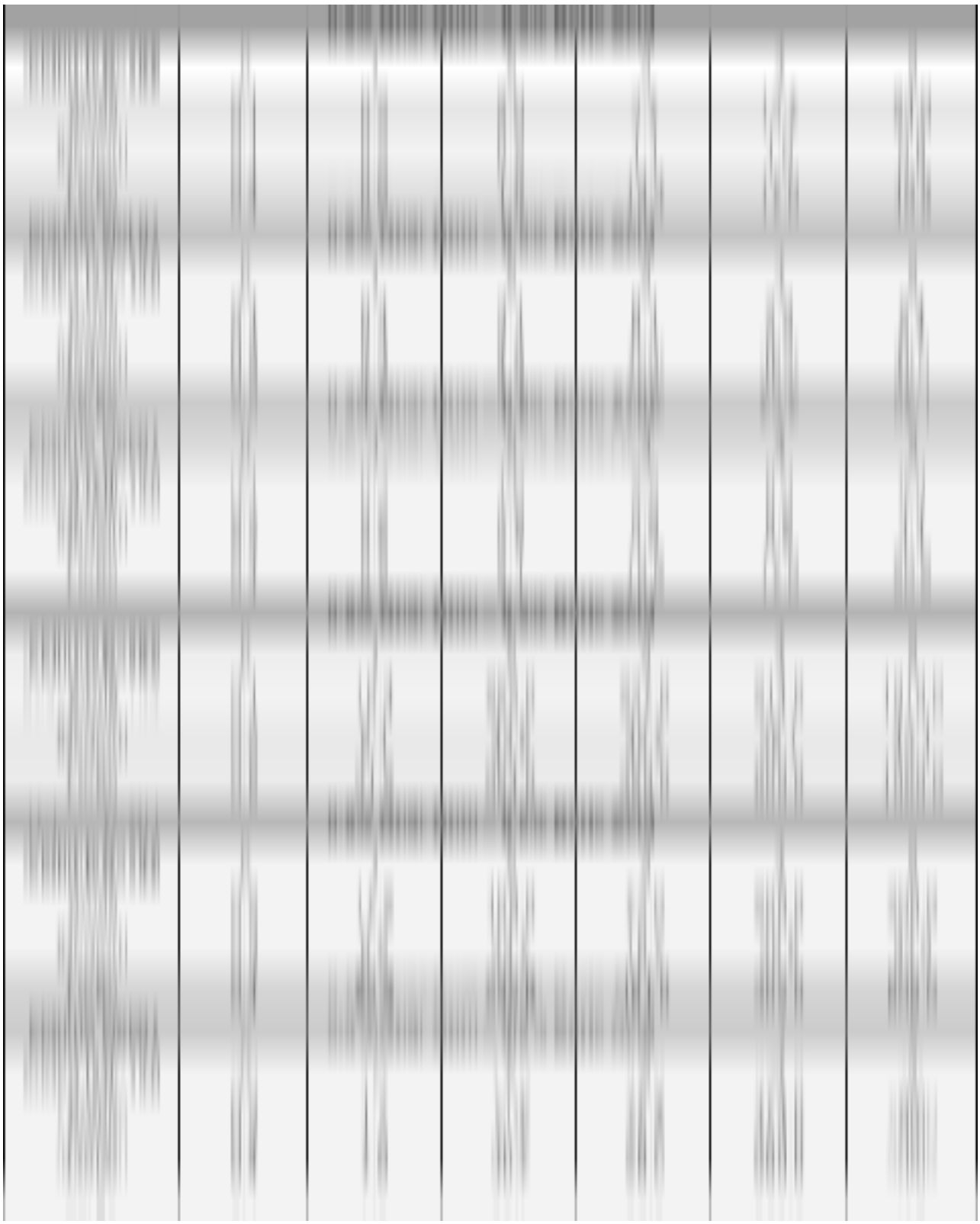
8.1 Appendix A – guar gum and boric acid solution specifics

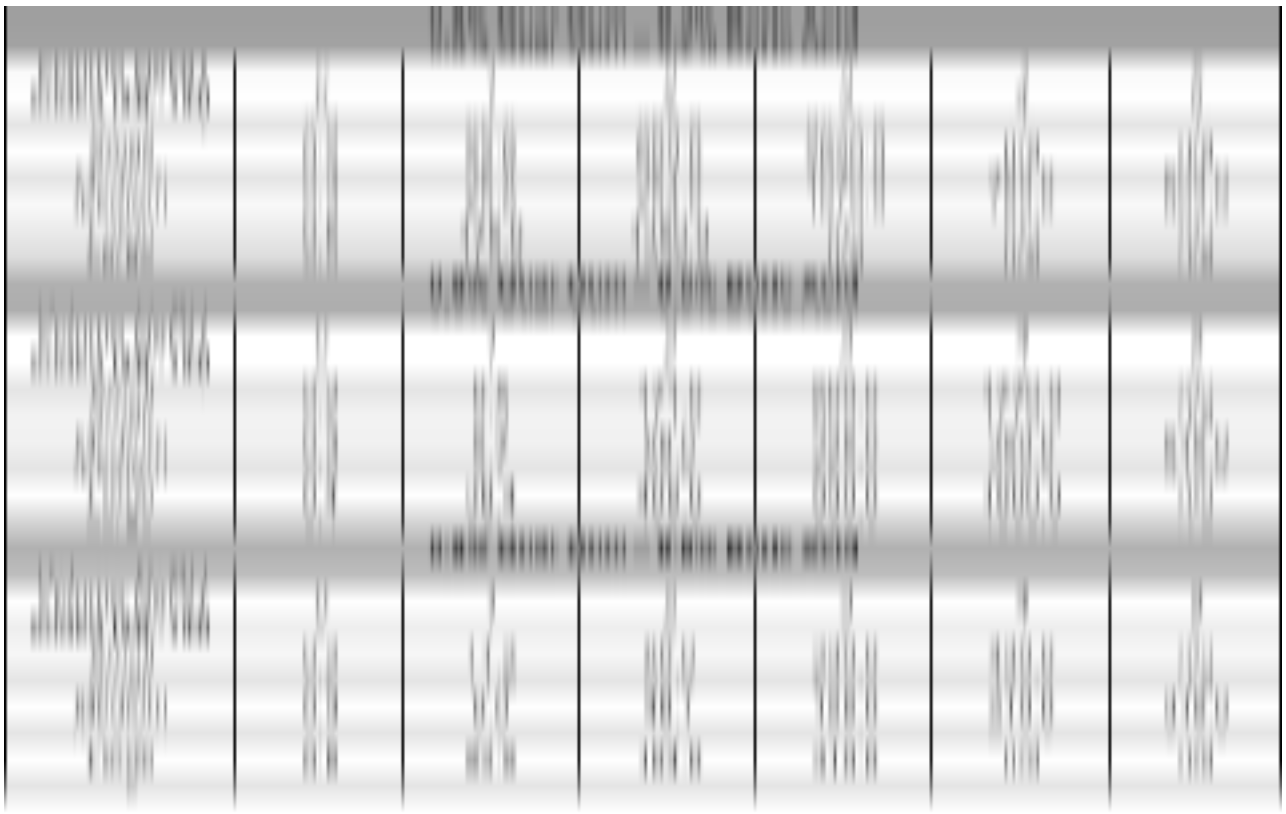
Powdered guar gum was added as a mass quantity to distilled water when creating the desired concentration. For 0.2% concentration 0.60g was added, for 0.5% concentration 1.50g was added and for 0.8% concentration 2.40g was added. Boric acid was premade in concentrated solution so that when added in a 27ml quantity, it would contain a 0.2%, 0.5% or a 0.8% concentration in the full 300ml of test solution. Each concentrated solution was produced in 250ml of distilled water using a volumetric flask. For 0.2% desired concentration 5.50g boric acid was used, for 0.5% desired concentration 13.75g boric acid was used, for 0.8% desired concentration 21.95g boric acid was used; in the 250ml solutions. To assist in the dissolution of the boric acid, each volumetric flask was heated in a hot water bath.

8.2 Appendix B – ball specific measurements

Three sizes of steel ball bearings were used in this experiment: a small medium and a large. The small ball bearing had a mass of 6.76g$\pm$0.01g and a diameter of 1.265cm$\pm$0.005cm. The medium ball bearing had a mass of 54.73g$\pm$0.01g and a diameter of 2.350cm$\pm$0.005cm. The large ball bearing had a mass of 109.88g$\pm$0.01g and a diameter of 3.020cm$\pm$0.005cm. Masses were measured using an electric balance and diameters were measured using a vernier calliper,

8.3 Appendix C – recorded data tables





8.3 Appendix D – qualitative data transcript notes

.2% guar gum .2% boric acid - 1M NaOH added
3ml became thicker as it sat and not when stirred
4-5ml started cleaning itself up
7ml small ball goes crazy and is either 10secs or over 80 – was able to pick some up on rod

.2% guar gum .5% boric acid - 1M NaOH added
1ml – stirred for about 5 minutes until it stayed consistent
2ml – pretty consistent, has the Weissenberg effect, cleans up better but not perfect
3ml – seems more fluid than before and not doing the Weissenberg effect, did fully clean itself up and made the noises as went down tube, stuck together more
4ml – still pretty fluid but cleaned up the best so far and went down the tube all together, stuck to itself the best, good cohesive properties
5ml – still pretty fluid, goes down tube easy

.2% guar gum .8% boric acid - 1M NaOH added
1ml – already thicker but not really sticking together much
2ml – starting to spin up, started to clean itself up but still fairly fluid
3ml – spins better than before, still pretty fluid, doesn't perfectly clean up
4ml – acts more like the gel but still pretty fluid, does spin, more cohesive and sticks together
5ml – bit more fluid again, spins a bit

.2% guar gum .8% boric acid - 1M NaOH added
0ml – this solution was made 24 hours before then tested but did not think it affected it here, pretty normal
1ml – really thickened, already sticking together, does break apart though, was showing different pH spots when testing pH
2ml – really broke apart as I was stirring together, came off in small globs, then formed up back together. Wow, hardest time getting it down, had to poke and shove it down. Went so slow.
3ml – new batch made next day, had similar time getting it in the tube
4ml – seemed like is was less jelly but not sure
5ml – when tested small ball it had no bubbles and in center of tube, also took forever when tested the large ball, went super slow at first so when first multiplied the time it took to travel 1cm it estimated to 1hr 28min, the actually time was a lot faster

.5% guar gum .5% boric acid - 1M NaOH added
0ml – just as liquid as usual
1ml – thickened really fast and stuck together nicely, almost cleaned up. Different stuff, will not break under shear stress, climbs the rod readily when swiftly stirred
2ml – fully staying together, did not break apart, spun around rod a lot. Did climb the rod when spun in place. Rod will not fall through gel when dropped. Took a long time to go down the tube, bubbled down

--------- first, our boric acid had crystallized some, made it again but next day had already crystallized. We heated up that same stuff, and tries to keep the stuff the same but the concentration may still be a bit off.
3ml – kind of breaks apart but not like some of the others. Went down tube pretty easy. Values are lower so not sure if I did that or if it is working properly
4ml – took a while getting down tube. Did break apart under sheer stress.
5ml – took a really long time getting down the tube, had to kind of break it to go
6ml – did take a long time

.5% guar gum .8% boric acid - 1M NaOH added
0ml – normal
1ml – flows easily, follows rod, and doesn't break. Pours easily, not sticking together so much, have to stir for about 6 minutes until equally thickens
2ml – will not break under sheer stress, flows together. Went down tube easily and flowed together, half liquidly but thick. Balls went down nicely
3ml – starts to break under a lot of sheer stress, stays together well, still flows pretty easily, rod does not sink much when dropped, bounces, will spin up onto the rod. Went down tube all together and filled it up but did not stop, went down by itself.
4ml – second batch of gel, seems about the same as before so far. Broke apart very easily when stirred, did not want to go down tube, broke it up then put it in bit by bit
5ml – broke very easily, more so just stirring, almost kept broken up form, didn't really settle back, but did form back.
- I think trials 4 and 5 on this were actually a .8% and .8% on accident

.8% guar gum .2% boric acid - 1M NaOH added
0ml – guar gum not fully dissolved, little clumps in it. Smell of the powder stronger.
1ml – got very thick, broke a stirring rod. Can lift up all gel onto stirring rod, does not break under sheer stress. Totally stuck together. Went down the tube well though, not to much trouble. May seem to be clumps in it still but is mostly same consistency. pH looks close to 8 but has a weird light blue color to that is not shown on the chart.
2ml – starts to break easy, not much effort and it does, reforms as normal but keeps rough shape
3ml – breaks very easy, almost like soap, is all in tiny pieces. Small ball took forever. Just going to test mid ball going forward. Bubbles may still be interfering.
4ml – broke when stirred, a large bubble may have made this one go faster
5ml – broke when stirred, was a large bubble on side as went down too. Started slow then sped up a lot

.8% guar gum .5% boric acid - 1M NaOH added
0ml – regular
1ml – thickens well, does spin up on rod. Sticks together some but does not completely clean up. Flows down tube easy
2ml – completely sticks together. Starts to spin up but not far. Starts to break when lots of sheer pressure applied. Very strong, can lift all up onto stirring rod. Went down tube together, had to push it down but not to bad.

3ml – Breaks easily when gently stirred. Does not spin up on rod. Can not pick up on rod. Large ball collects a lot of bubbles as nears the end, also moves faster then, not sure if connected.
4ml – really really broke up when stirred. Cannot pick up on rod. Large ball sped up.
5ml – breaks very easy, seems like stuff before

.8% guar gum .8% boric acid - 1M NaOH added
temp of boric acid after re heating between 60-65 degree Celsius
0ml - same liquid, guar gum good job of dissolving but maybe few clumps
1ml - took about 5 minutes to start to thicken, sired for about 10 minutes. Flows around stirring rod and up it, but gel does not break. Sticks together easily, but still flows out when brought up on rod. Gel appears smooth once fully thickened. Great Weissenberg effect, spun up on glass rod, went up about 12cm. Gel at room temp of 23.7 Celsius. Flowed down tube easily. Appearance is sticky like or gooey, not so much blob. Does not fully clean itself up.
2ml - is no longer sticky to the rod. Starts to break but not fully yet, follows spin of the rod. Harder to move through than before. exhibits some Weissenberg effect, but only goes up about 3cm. Can lift entire gel mass on stirring rod, holds up well. did not flow down tube. Readily bonds back together, cleans up very well. are not very bubbles in the tube.
3ml - breaks apart easily as stirring through the gel, like soup. Does not re-bond with it after a piece breaks off. This may contribute to the appearance of more bubbles in the tube. Am able to lift most of gel mass onto the top of the rod, can hold it up easily. Starts to spin around glass rod but does not climb up at all.
4ml - started with new solution next day, may be a bit different. Breaks very easily, all into tiny pieces as stirred. No Weissenberg effect at all, does not spin surrounding gel. Cannot lift onto rod, breaks off (could not lift in previous step either second mixture).
5ml - easy to stir right through the gel, broke up easy. No Weissenberg effect. Cannot lift up onto rod.