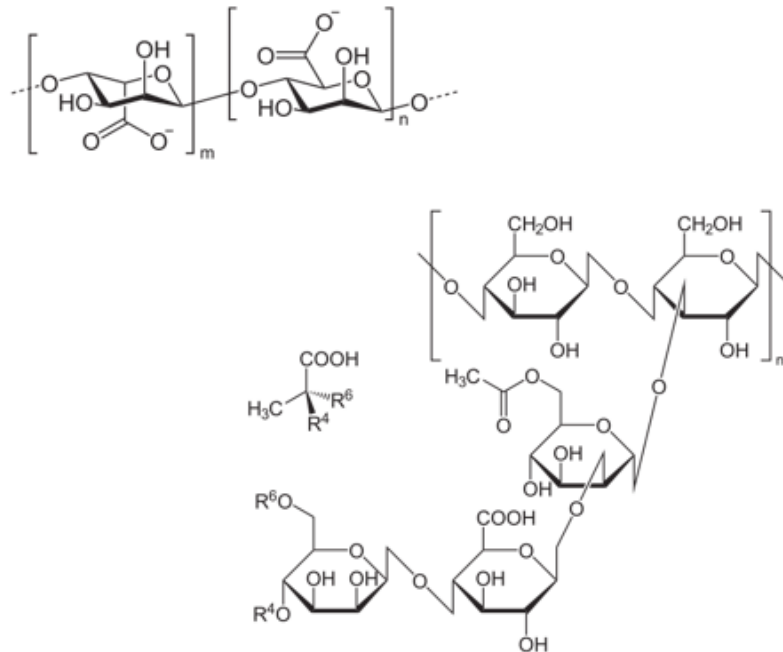




Strain Hardening Properties of Polymeric Materials for Artificial Blood Vessels

A Research Study Investigating the Strain Hardening Properties of Alginate and Xanthan Gum

Master's thesis in Learning and Leadership

HEKLA HJALMARSDOTTIR

KRISTINA NILSSON

MASTER'S THESIS 2020

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Abstract

Biomaterials and artificial body tissues are a part of life science and a field where there is always room for new inventions and improvement. Previous research shows that viscoelastic properties like strain hardening are important to consider in order to develop artificial body tissue. Therefore, this research study investigates the strain hardening properties of hydrogels based on the polysaccharides alginate and xanthan gum. The investigation was conducted by examining the polysaccharides rheologically in small and large amplitude oscillatory shear. The results indicate that both alginate and xanthan gum start showing strain hardening behavior at high strain (10%-100%). Therefore it can be suitable for future artificial soft body tissue engineering. Alginate was tested with ionoprinting as well to see if it is possible to insert local stiffness in polysaccharides, which the study concludes is possible. A lesson plan for a laboratory session in Swedish upper secondary school was developed to increase public engagement and knowledge of viscoelasticity and strain hardening properties.

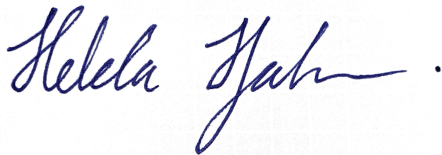
Keywords: Alginate, biomaterial, hydrogel, ionoprinting, polymer, rheology, strain hardening, viscoelasticity, xanthan gum

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Hekla Hjalmarsdottir



Kristina Nilsson

Chalmers University of Technology, Gothenburg, June 2020

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Glossary

biomaterial Materials that treats, replaces or changes organs in a biological body. For example in implants or other medical devices. 1

complex modulus A way to describe a material's resistance of elastic transformation when stress is applied. The complex modulus symbol is G^* and the unit is Pascal, [Pa]. 12

hydrogel A gel built up by polymers with hydrophilic chains. 4

ionoprinting A way of inserting stiffness in gels by using a power supply to force positively charged metal ions to migrate into a gel. This creates a local stiffness in the gel. 4

loss modulus The imaginary part of the complex modulus. The loss modulus symbol is G'' and the unit Pascal, [Pa]. 13

polyelectrolyte A polymer that is built up by units with an electrolyte group attached to it. 14

polysaccharide A polymer type made of monosaccharides. 15

resilience A material's ability to absorb energy during deformation. 4

rheology A scientific field that studies the flow and deformation of solids and liquids. 4

storage modulus The real part of the complex modulus. The storage modulus symbol is G' and the unit is Pascal, [Pa]. 13

strain The degree to which a material is deformed as a result of an applied force. Strain is in this report referred to as ε and the unit for strain is percent, [%]. 11

strain hardening The mechanical properties that makes a material stiffen with increased strain or strain rate. In this report strain hardening is defined as the phenomena that occurs when the stress of a material increases more rapidly than the strain rate. Another word for strain hardening is strain stiffening. In this thesis, however, only the former is used. 11

stress The tension that arises in the material as a result of a certain strain. Stress is in this report referred to as σ and the unit for stress is Pascal, [Pa]. 11

viscoelasticity A mechanical behavior that lies in between classic Hookean behavior and Newtonian liquid behavior. It is a way of describing materials that have both viscous and elastic behavior. 2

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1

Introduction

Biomaterials can be defined as materials that interact with biological systems in the form of implants or other medical devices, (Ratner, Hoffman, Schoen, & Lemons, 2013). The science of biomaterials has developed over the last 80 years and that the scientists in the 1940s and 1950s worked with the goal of creating materials that would not be rejected by human bodies. Today the techniques have advanced so that the main goal is not only to produce materials that are accepted by human bodies, but also supports the regrowth and recovery of human body tissues according to Ratner et al. (2013). The authors state that the field of biomaterials has saved lives and increased the living standard worldwide. This because biomaterials are used as prosthesis in many medical fields like orthopedics and cardiovascular science. As an example of the use of biomaterials in cardiovascular science, Silver and Doillon (1989) explain how it has been possible to replace aorta tissues in human bodies ever since 1950.

In this research study, polymers have been investigated to explore future possibilities to develop materials for artificial blood vessels. Polymers are molecules that are built up by a number of smaller units, monomers, (Koltzenburg, Maskos, & Nuyken, 2017). Koltzenburg et al. (2017) discuss the utility of polymers and list a number of different areas where polymers are used. The authors state that polymers occur naturally in the form of for example DNA and proteins. The authors further give examples of how polymers are used today and mean that polymers are invaluable when it comes to packaging in the food industry. Polymers are also used as additives in food, cosmetics and pharmaceutical products.

1.1 Background

Ratner et al. (2013) state that biomaterials play a crucial role in the field of tissue engineering. The authors further describe tissue engineering as a research field that started in the 1970s when scientists started to develop skin replacement. It was

however not until the early 1990s that the field was clearly defined as a medical field focusing on using engineering and life science to develop materials that maintain, reset or improve human body tissue. One part of tissue engineering that Ratner et al. (2013) explain is engineering of blood vessel tissue. The authors mean that artificial blood vessels could be a good way of treating for example atherosclerotic vascular disease. However, it has been difficult to mimic the mechanical properties, like viscoelasticity, of blood vessels and that has complicated the development of approved materials.

Viscoelasticity of a material can be described as something in between classical Hookean behavior that has been used to describe solids and the Newtonian viscous behaviors that has been used to describe liquids, (Barnes, Hutton, & Walters, 1989). As an example the authors bring up how silk threads not fully obey Hooks' law which means that they not are ideally elastic. Viscoelasticity became a way to describe this liquid-solid-behavior of those materials.

As mentioned by Ratner et al. (2013), viscoelastic properties of materials are also important when it comes to biocompatibility, which is the way materials interact with body tissue. Holzapfel (2000) describes mechanical properties of soft human body tissue and means that it, because of it's non-homogenous composition, behaves anisotropically. This means that the physical properties differs in different directions. Holzapfel (2000) states that soft body tissue can withstand large deformation under which the tissue stiffens in a non linear way and that the stress built up in the material depends on the velocity with which the material is deformed.

The viscoelastic properties of soft body tissue is a major issue when designing new artificial vessels. James and Sneyd (2009) describe how blood vessels are elastic and developed a model to describe the elasticity of blood vessels. However, the viscoelastic properties that Holzapfel (2000) describes, namely the fact that the tensile stress of body tissue is dependent on the strain rate, is not a considered in the model. Instead the model keeps the velocity with which the blood is pumped through the vessels constant. Bertaglia et al. (2019) and Nichelatti, Pettazzoni, and Pallotti (2017) mean that the cardiovascular behavior is strongly influenced by the viscoelastic properties of the blood vessels and that the understanding of these properties therefore is essential for understanding hemodynamics, that is the dynamics of blood flow in living bodies.

Since Bertaglia et al. (2019) focus on developing mathematical models describing the viscoelastic properties of blood vessels, these properties can be considered essential

also when investigating future biomaterials. Bertaglia et al. (2019) further state that:

Viscoelasticity of vessels play an essential role in the cardiovascular behavior (Salvi (2012); Nichols, O'Rourke, and Vlachlopoulus (2011); Holenstein, Niederer, and Anliker (1980), being one of the features that must be realistically included in the mathematical model when accurate numerical results are sought (Holenstein et al. (1980); Alastruey et al. (2011); Montecinos, Müller, and Toro (2014)). (p.1)

This emphasizes the importance of investigating the viscoelastic properties of polymers deeper in order to develop biomaterials with viscoelastic properties more similar to human body tissue than materials existing today.

More specifically Bertaglia et al. (2019) mean that viscoelastic properties of blood vessels can be divided into three different kinds of viscoelasticity: creep, relaxation and hysteresis. Barnes et al. (1989) describe creep as the deformation of a material under constant stress, and relaxation as the decrease in stress under constant strain. Bertaglia et al. (2019) describe hysteresis as the energy that is lost due to viscoelastic effects. The authors further state that there are several models that describe the linear viscoelastic behavior of blood vessels but that hysteresis is rarely considered.

Cui, Tan, Zhu, and Guo (2014) state that synthetic hydrogels have been developed to meet the mechanical requirements when designing artificial body tissue. The authors also mean that biological polymers, like protein, should be investigated as well as synthetic hydrogels. This because biological hydrogels differs from synthetic hydrogels by having special viscoelastic properties, for example strain hardening. Cui et al. (2014) state that:

This unique viscoelasticity of biopolymer-based hydrogels is fundamental to their biological function and the maintenance of normal physiology (p.56791)

Cui et al. (2014) mean that protein based hydrogels show strain hardening properties at large strains. For example, Janmey et al. (1994) mean that actin shows strain hardening properties at strains between 0-20% and Ma, Xu, Coulombe, and Wirtz (1999) mean that keratin show strain hardening properties at strains between 0 and 100%. Cui et al. (2014) further describe strain hardening as “a sharp increase in material stiffness at large strains” (p.1). The authors mean that the strain hardening properties of soft body tissue is important to consider since strain hardening properties help body tissue to resist deformation that could harm the tissue. The authors also mean that synthetic hydrogels existing today can be deformed repeat-

edly without losing energy and that they have high resilience. This is important properties since it facilitates repeated movement in soft body tissue.

Hydrogels can be described as crosslinked networks that can absorb and give off water, Palleau, Morales, Dickey, and Velez (2013). Baker, Wass, and Trask (2018) further mean that a problem with hydrogels is that their toughness is lower than that of soft body tissue. As a way to increase the toughness Baker et al. (2018) present ionoprinting. According to the authors, this method can be used as a way to insert stiffness in hydrogels by forcing the polymers in the hydrogels to locally crosslink themselves to positively charged metal ions that are applied to the surface of the hydrogel.

In this thesis, hydrogels based on calcium-alginate and xanthan gum/locust bean gum will be investigated rheologically to examine the strain hardening properties of the hydrogels. The term rheology is defined as the study of the deformation and flow of a matter, (Barnes et al., 1989). A rheometer is a laboratory equipment used to determine a material's rheological properties, which relates to force, deformation and time. The principle of a rheometer is applying a known force to a sample through shearing or changing the temperature and then measuring if the applied energy is stored or lost in the structure of the sample. Earlier studies show that calcium-pectin gels show strain hardening properties at approximately 10%-200% strain and that the strain hardening properties depends on the calcium concentration in the gel, (John, Ray, Aswal, Deshpande, & Varughese, 2019). Further, hydrogels based on pectin-alginate show strain hardening properties at approximately 40%-110% strain according to Michon, Chapuis, Langendorff, Boulenguer, and Cuvelier (2004). When it comes to xanthan gum/locust bean gum earlier studies show that hydrogels made with 60% xanthan gum and 40% locust bean gum show strain hardening properties at 50%-200% strain, (Michon et al., 2004).

Ionoprinting will also be conducted on calcium-alginate gels in this thesis. This to see whether it is possible to insert local stiffness in the gels and create materials with mechanical properties similar to those of human body tissue. Ionoprinting has in previous studies been successfully conducted on sodium polyacrylate hydrogels, (Palleau et al., 2013).

1.1.1 Economical Aspects to Consider for Polymeric Material Selection

Mazzucato (2018) argues that clear goals are important when it comes to scientific research and that there must be a larger goal with research than just curiosity. Mazzucato's (2018) study aims to illustrate how the European research could result in more innovation. Mazzucato (2018) means that the European countries need to work with targeted research, applied science, in order to increase innovation and manage future challenges like sustainability and growth based on innovations. Mazzucato (2018) means that in order to achieve this new problem solving based way of doing research, the research and science can be seen as a part of the product development process. Therefore it is important to make the right decisions early in the research/product development process.

When talking about applied science, fundamental science should also be mentioned since there are many scientists that mean that fundamental science is essential when it comes to making scientific progress. Fundamental science can be referred to as the basic science answering questions of how things work. Dainton (2013) means that fundamental science does not only answer questions that people find curious but also leads to innovation that increases public health and life quality. Mazzucato (2018) however, does not mean that applied research should be given priority over fundamental research. Instead, the author emphasizes the possibilities of increasing the collaboration between applied and fundamental science to develop new ways of performing research.

Faris, Oqla, and Mohd (2017a) mean that developing a product is based on three important tasks: defining the shape of the product, material, and production. The authors emphasize that if these tasks are carried out properly and early in the product development process, the product cost decreases and the product development process is shortened. Faris et al. (2017a) describe different ways of succeeding when it comes to product development and state that *"selecting an adequate material is considered as a key driver for attaining user satisfaction as well as market growth"* (p.49). Mogahzy (2009) means that *"material selection is an essential phase of the product design cycle."* (p.165) and argues that the material selection of a product is essential since the wrong selection of material not only leads to insufficient products but also increases production and product costs. Edwards (2014) states that *"Correctly selecting the materials, among other things, is critical to the success of any product"* (p.287). In this research study material selection have been considered aiming to perform targeted research.

Mogahzy (2009) presents two key factors that should be considered when choosing the right material: material processing and material properties. The first key factor affects the possibility to manufacture the product and the second key factor concludes the properties of the final product. The author further presents five criteria that the chosen material should meet. The first criteria is *good manufacturing possibilities*, the final product should be as easy and cheap as possible to produce. The second criteria is about *material properties*, by this the author means that the properties of the material should be related to fulfill the desired functionality of the final product. The third criteria is about *dependability*, meaning that the properties of the material should not change over time. The fourth criteria treats the *material's ability to resist changes* in environment or other external impacts. The fifth and last criteria is that the material should be *recyclable*.

Faris et al. (2017a) also mean that there are several different problems that must be considered when selecting the right material for creating successful products at low cost. The authors state that:

Such issues include physical, mechanical, electrical, magnetic properties, cost, availability, manufacturing abilities, durability, environmental impact, recyclability, and others. In addition, both metaphysical properties and user-interaction aspects including appearance, perceptions, and emotions have also to be considered during the material selection process.
(p.49)

Sapuan (2017) also discusses aspects to consider when choosing the right material. Apart from the aspects that Faris et al. (2017a) brings up, Sapuan (2017) also emphasizes maintenance, safety, transport and quality control.

In this research study one focus has been to perform targeted research by choosing the right material early in the research process. In accordance with the theories described in this section, the aspects presented in table 1.1 were considered when choosing the materials for this research study.

Table 1.1: *An overview of the aspects considered when selecting materials for the research study. The different properties are based on the theories that Mogahzy (2009), Faris et al. (2017a) and Sapuan (2017) emphasize. In the table the aspects are presented together with a motivation of why it is important to consider when choosing material in product development processes.*

Aspect	Theoretical motivation
Existing polymeric materials	Using existing materials, the product development process is shortened and the manufacturing possibilities that Sapuan (2017) emphasizes are increased since the material already is used commercially.
Biocompatibility	Mogahzy (2009) means that the material properties should be taken into consideration when selecting a material and since the purpose is to use the material in human bodies the biocompatibility is a pre-requisite.
FDA-approval	Since the new European regulation MDR, medical device regulation, has been postponed one year according to Commission European (2020) this research study has chosen FDA-approval instead. According to U.S. Food & Drug Administration (2020), FDA-approval means that the drug or the material has been investigated and that strategies have been made regarding the risks of the material or drug. The chemical's biocompatibility has also been investigated, if the material or drug is FDA-approved. By having FDA-approval as a requirement in the material selection process the product development does not have to include the process of getting the material approved, which saves time and money. The chances of good manufacturing possibilities increases since the material already is used in production. This is something that Mogahzy (2009) highlights. The FDA-approval also takes the material's safety into consideration which is emphasized as important by Sapuan (2017).
Availability/Supply	By ensuring that the material is easily accessible, the possibilities of reliable supply and good manufacturing that Mogahzy (2009) emphasizes is further increased.
Safety	The safety of the product, highlighted by Sapuan (2017) has been taken into consideration since FDA-approval was a requirement in the material selection process.

Aspect	Theoretical motivation
Commercial usage	The commercial usage has been investigated to ensure that the materials have good manufacturing possibilities, which is important according to Mogahzy (2009).
Molecular structure	The molecular structure of a material gives information about the materials' properties for example charge density. Faris, Oqla, and Mohd (2017b) mean that the wanted mechanical, electrical and physical properties should be taken into consideration when selecting materials.
Strain hardening properties	The mechanical property that is desired in this case is strain hardening, a type of viscoelastic property. A strain hardening material stiffens with increasing strain rate, which is how Holzapfel (2000) describe blood vessels.
Gelling abilities	The gelling abilities of a polymer is important when it comes to the material's stiffness and viscoelastic properties. Since Faris et al. (2017b) state that mechanical properties should be considered in the material selection process this aspects have been taken into consideration.

A literature review was performed on different polymeric materials, which resulted in that hydrogels based on alginate (with calcium) and xanthan gum (together with locust bean gum) best matched the aspects in table 1.1. These gels were therefore chosen for further investigation in this research study.

1.1.1.1 Knowledge Transfer for Targeted Research

Mazzucato (2018) means that targeted research is facilitated by cross-disciplinary work. By making different scientific fields work together with different industrial sectors, both private and public, Mazzucato (2018) means that public engagement and understanding of what science research is and what it does for society is created among the European citizens. Engaging citizens is a another key to create more targeted research according to Mazzucato (2018). The author also emphasizes the difficulties that arise with this since science often is considered to be difficult and hard to understand. This is something that Osborne (2007) describes and means that in order to educate citizens and make science more comprehensible for all people, teachers should focus on helping all students to understand the basic concepts of science and not focus on detailed information. In his book, *Science Education for Everyday Life*, Aikenhead (2005) also states that teaching at schools should focus

on preparing all students for what it is like to consume science as it would increase science knowledge in society. This is according to Mazzucato (2018) essential for achieving targeted knowledge in society as a whole.

In this research study, a lesson plan for a laboratory session for students in Swedish upper secondary schools is developed in order to increase the knowledge and engagement of polymeric materials and their mechanical properties among students.

1.2 Purpose

The study aims to investigate the strain hardening properties of the hydrogels based on the polysaccharides alginate and xanthan gum with locust bean gum. This to investigate the possible utilization of polymers for future economically sustainable innovations in the field of artificial artery tissue. The possibility to examine existing biocompatible and FDA-approved polymers to evaluate their strain hardening properties will be investigated by using small and large oscillatory deformation and ionoprinting. The research study also aims to increase public engagement and understanding of polymers by introducing strain hardening for students in Swedish upper secondary school.

1.3 Research Questions

The following research questions were chosen as a way to fulfill the purpose of the research study:

- What economical aspects of biocompatible materials could be taken into consideration for future commercialization?
- Do calcium-alginate gels and xanthan gum/locust bean gum gels have strain hardening properties that could make them suitable for soft body tissue engineering, such as artificial artery tissue?
- Which of the hydrogels is the most suitable, when it comes to strain hardening properties, for developing a material for artificial artery tissue?
- How can the viscoelastic property strain hardening be presented and explained to students in Swedish upper secondary schools?

2

Theory

This research study aims to find answers of the strain hardening properties of hydrogels based on alginate and xanthan gum/LBG. This chapter presents viscoelasticity and strain hardening in detail. Alginate, xanthan gum and locust bean gum and their properties are also described. The chapter ends with presenting different learning theories. These are the theories used to develop the laboratory session for students in Swedish upper secondary schools.

2.1 Viscoelasticity

Viscosity can be presented as internal friction. Barnes et al. (1989) state that it is “*a measure of resistance to flow*” (p.2). Viscoelasticity can be described as a property that lies between the classic Hookean elastic response and Newtonian viscous behavior, (Barnes et al., 1989). By Hookean elastic response the authors mean the linear proportional behavior of a spring applied by a force, which is the base of the classical elasticity of solids. Barnes et al. (1989) describe Newtonian viscous behavior of a liquid and mean that the internal resistance in the liquid is proportional to the velocity of the shear stress applied to the liquid. According to the authors, this description of solids and liquids was the accepted truth for the public until the 19th century. Thereafter, scientists began to question this way of describing viscosity, for example by proving that silk threads were not perfectly elastic according to Hooke’s law.

Properties like rigidity modules and viscosity can change depending on the stress applied to the material, (Barnes et al., 1989). This means that the behavior of the material stress is not linear with increasing strain or strain rate. As an example of non-linearity, the authors mention shear-thinning which means that the viscosity decreases when the shear rate increases. Barnes et al. (1989) then state that “*if we apply a very wide range of stress over a very wide spectrum of time, or frequency, using rheological apparatus, we are able to observe liquid-like properties in solids and*

solid-like properties in liquids.” (p.4). The authors mean that this makes it hard to state whether a material should be classified as a solid or a liquid. These materials can be referred to as semi-solids. It is a way to explain the non-linearity of materials that do not behave perfectly like they should according to either Hooke’s or Newtonian law but somewhere in between. Van Aken (2007) means that gels based on biopolymers are an example of semi-solid materials.

2.1.1 Strain Hardening

This study focuses mainly on one particular type of viscoelasticity, namely strain hardening, which is the phenomenon that occurs when increasing strain or strain rate leads to a nonlinear stiffening of a material. The stress, σ , of the material is given by equation 2.1 where σ represent stress, F force and A the area on which the force is applied.

$$\sigma = \frac{F}{A} \tag{2.1}$$

The stress of a material increases proportionally to the strain, ε , in the linear viscoelastic interval. At a certain point, the stress does not increase linearly but nonlinearly. This is where the nonlinear interval begins. Figure 2.1 shows the stress-strain diagram of a material applied to a strain, ε . The stress increases proportionally at first but after a certain point, it becomes nonlinear.

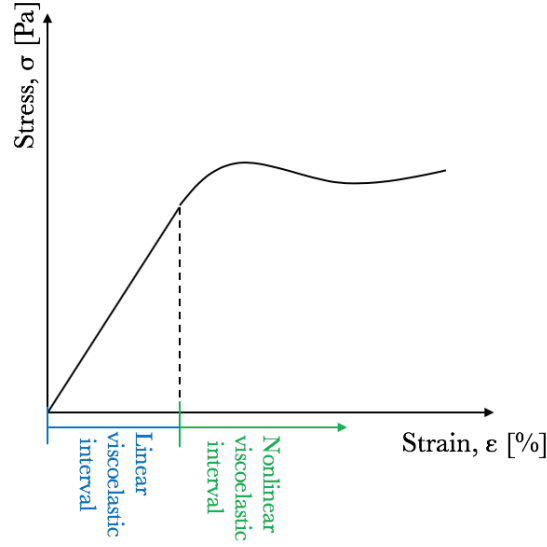


Figure 2.1: *An illustration of the linear and nonlinear viscoelastic interval. The diagram shows stress as a function of strain and represents the behavior of a material that is applied by a strain that increases with constant strain rate. Up to a certain strain level the stress increases linearly to the strain. This is the linear viscoelastic region, highlighted with the blue text. At a certain strain level the non-linear interval, highlighted in green text, begins and the stress changes nonlinearly when the strain is further increased. (Created by thesis authors in PowerPoint)*

Wang and Li (2015) and Michon et al. (2004) argue that if the nonlinearity is of the kind that the material stress increases more rapidly than the material strain, the material has strain hardening properties.

Strain hardening can further be described mathematically by calculating the complex modulus, G^* , of the material. G^* is calculated by equation 2.2 where G^* represents the complex modulus, σ the stress and ε the strain.

$$G^* = \frac{\sigma}{\varepsilon} \quad (2.2)$$

In figure 2.2 the complex modulus increases with increasing strain. This is what Wang and Li (2015) describe as strain hardening. In the nonlinear viscoelastic interval, G^* increases as the strain is increased. When strain is increased the material becomes harder to deform.

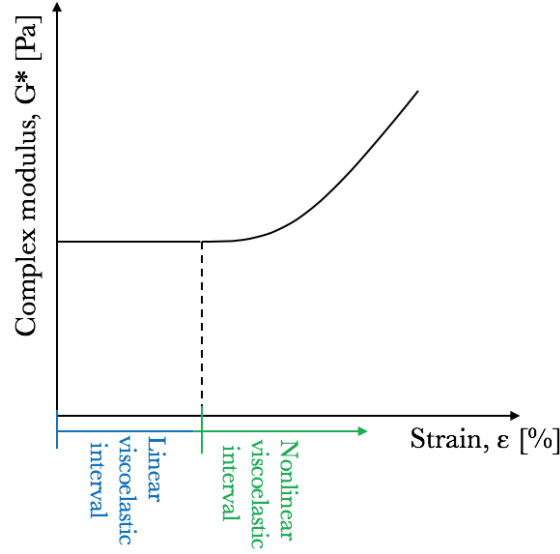


Figure 2.2: *An illustration of Wang and Li (2015)’s definition of strain hardening. The complex modulus is constant as long as the strain is in the linear viscoelastic interval. But in the nonlinear viscoelastic interval the complex modulus increases which means that it becomes harder to deform the material. The material’s stiffness is increased. (Created by thesis authors in PowerPoint)*

Barnes et al. (1989) explain how the complex modulus can be divided into the storage modulus (G') and the loss modulus (G''). The authors describe storage modulus as the real part of the complex modulus and the loss modulus as the imaginary part. According to Meyers and Chawla (2009) the viscoelastic properties of a material can be investigated by sinusoidal deformation. The loss and storage moduli are a mathematical description of the phase shift between the sinusoidal stress and strain curves that are generated from sinusoidal deformation. The authors mean that if the stress and strain curves are in phase the material is elastic and if they are 90° out of phase the material is viscous. The curves of viscoelastic materials are out of phase as well but between 0° and 90° . When the curves are out of phase they can be mathematically described in complex form as showed by equation 2.3.

$$G^* = G' + iG'' \quad (2.3)$$

Meyers and Chawla (2009) state that the storage modulus indicates how elastic the material is, that is how much energy the material can store. The loss modulus is described by the authors as the viscous part of the material and gives a picture of how much energy that is lost as heat. Sim, Ahn, and Lee (2003) and John et al. (2019) use the storage and loss moduli to define and identify strain hardening in their studies and mean that if G' and G'' increase with applied strain the material shows strain hardening properties.

Sim et al. (2003) present a model to analyze large amplitude oscillatory shear (LAOS) results, which was developed by results from Hyun, Kim, Ahn, and Lee (2002). This model entails plotting the reduced storage modulus (G'/G'_0) and the reduced loss modulus (G''/G''_0) as a function of strain, where G'_0 and G''_0 are the moduli in the linear viscoelastic region. Hyun et al. (2002) suggest that by analyzing the produced charts, identification of moduli behavior in LAOS can be conducted. The authors classified four different types of LAOS behavior in the nonlinear region; strain thinning, strain hardening, weak strain overshoot, and strong strain overshoot. Strain thinning behavior can be identified if both G' and G'' decrease as the strain increases. If however, the opposite is true, that is both G' and G'' increase as strain increases, it shows strain hardening behavior. For the weak overshoot type the G' decreases while G'' first increases and then decreases with the increase of strain. The final type, strong overshoot, both G' and G'' increase at first and then decrease as strain increases. John et al. (2019) concluded in their study that there are distinct indications that strain hardening and G'' overshoot are related. The authors identified that as the extent of strain hardening increased along with the intensity of the G'' overshoot, and therefore strain overshoot can be used to detect strain hardening behavior.

Saint-Martin (2017) describes in his PhD thesis how different polymers have different strain hardening properties depending on their chemical properties. In this study the strain hardening effects of two different polyelectrolytes have been investigated. Polyelectrolytes are polymers whose side chain has a charged group like a carboxyl group. The polyelectrolytes that have been investigated in this study are alginate and xanthan gum. The first one, alginate, is a negatively charged by a carboxyl group. The second one, xanthan gum, is negatively charged as well but the charge density is lower than that of alginate. Locust bean gum is presented as well as it is needed for xanthan gum to gel. The polymers and their strain hardening properties are presented in the next chapter.

2.2 Properties of Alginate, Xanthan Gum and Locust Bean Gum

A prestudy to the research study was conducted in the form of a literature review, in order to identify the most suitable polymers with desired strain hardening properties for the laboratory work. The method as well as the results of the literature review are presented in appendix A and the complete search history in appendix

B. The literature review resulted in the selection of two different polyelectrolytes for the laboratory work, alginate and xanthan gum. The properties of locust bean gum are presented as well. The commercial utilization and the properties of these polymers as well as an overview and comparison of the two different polymers are presented in this chapter.

2.2.1 Alginate

Alginate is a polyelectrolyte that exists naturally as it serves as a structural component in marine brown algae and capsular polysaccharides in soil bacteria, (Phillips & Williams, 2009). The authors further explain that the intercellular alginate gel matrix contributes to the plants mechanical strength and flexibility as the polymer's biological function is forming structure in brown algae.

Alginate is a common additive in the food industry as it contributes to desired attributes and textures of food products. The features that alginate can give rise to are in the form of stabilizing, thickening, emulsifying, and gelling agent in products including ice cream, pudding, salad dressing, and fruit juices according to Zhang, Daubert, and Foegeding (2007). Kibbe (2000) means that alginate is ordinarily considered as a safe, nontoxic and nonirritant material. However, excessive oral consumption might be harmful according to the author.

The molecular structure of alginate is illustrated in figure 2.3. Phillips and Williams (2009) explain that the structure of alginate can be described as “a family of linear binary copolymers of (1->4)-linked β -D-mannuronic acid and α -L-guluronic acid residues of widely varying composition and sequence” (p. 809). Every glucuronic acid residue has a carboxyl group attached which gives alginate a negative charge. β -D-mannuronic acid can be referred to as M-unit and α -L-guluronic acid as G-unit.

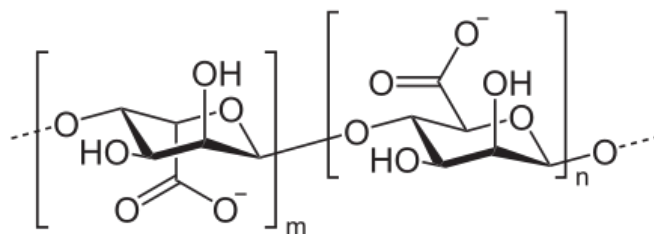


Figure 2.3: The molecular structure of alginate, which is built up by α -L-guluronic acid (in the bracket subscripted with m) and β -D-mannuronic acid (in the bracket subscripted with n). (Image retrieved from Commons (2014))

Hashemnejad and Kundu (2016) explain that alginate can build a network structure in the presence of multivalent cations and transition metals, for example Ca^{2+} , Ba^{2+} , Cu^{2+} and Mn^{2+} . The authors used CaHPO_4 as a calcium ion agent in their experiments on alginate hydrogel. Glucono delta-lactone (GDL) must be added as well to the alginate hydrogel solution as it obtains Ca^{2+} and together they create ionic crosslinking in alginate gels. Alginate's gelation process entails that Ca^{2+} binds to the G-units in the alginate chains. The authors describe that when G-units group together G-blocks are created and that when G-blocks connect with Ca^{2+} , junction zones are formed. These junction zones serve as crosslinks and give rise to a three-dimensional network.

Zhang et al. (2007) confirms that alginate does not gel on its own and an addition of cations is necessary for the gel composition, the most common being calcium ions. Their tests also showed that strain hardening increased with increasing the concentration of either Ca^{2+} or alginate while keeping the other concentration fixed. The authors investigated the nonlinear viscoelastic properties of alginate gels using torsion and compression test. During large deformation, the tested alginate gels displayed strain hardening behavior which was dependent on the composition of the gels. The authors explain that the strain hardening behavior in alginate originates from the deformation of the junction zones, which are deformed by stretching, compression and bending.

The strain hardening properties of alginate were investigated by Michon et al. (2004) and the authors mean that gels made of the combination of pectin and alginate show strain hardening behavior in the range of 50-110% strain, after that the gel ruptures.

2.2.2 Xanthan Gum and Locust Bean Gum

Xanthan gum is a polymer produced by micro-organisms, (Phillips & Williams, 2009). Just like alginate, xanthan gum is used in the food industry. The properties of xanthan gum makes it suitable in semi-manufactured food since it contributes to the desired volume and texture of for example dough and gluten free breads. Xanthan gum is also often used as a stabilizing and suspending agent in cosmetics, food and oral pharmaceuticals, (Kibbe, 2000).

Kibbe (2000) describes the safety guidelines put up for xanthan gum and means that the polymer is considered to be nontoxic if following the recommended daily intake of 10 mg/kg body weight.

The molecular structure of xanthan gum is presented in figure 2.4. According to Phillips and Williams (2009) it is built up like cellulose with β -D-glucose units linked together by (1,4)-glukoside links. On every other glucose-unit a trisaccharide is attached as well. This trisaccharide have a glucuronic acid residue in the middle. The glucuronic acid residue has a carboxyl group which gives xanthan gum a weak negative charge.

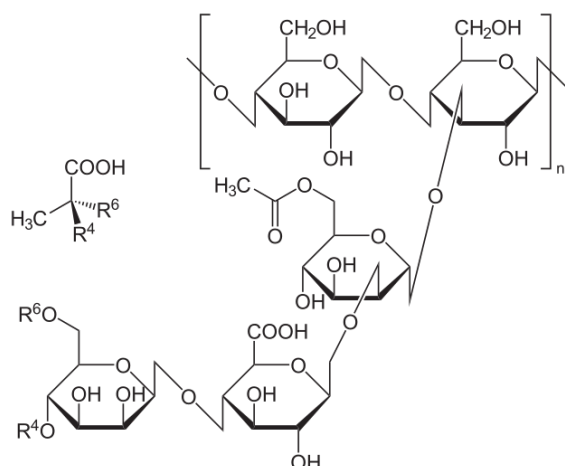


Figure 2.4: The molecular structure of xanthan gum. The unit consist of two β -D-glucose units and an attached trisaccharide is linked to the right β -D-glucose. The brackets with a subscripted n represents the repeating structure of this unit that builds up xanthan gum. (Image retrieved from Commons (2013))

Locust bean gum (LBG) can be used together with xanthan gum in order to increase viscosity of a polymer solution, (Casas & García-Ochoa, 1999). LBG is a polymer that is, just like xanthan gum, used as a thickening agent in the food and pharmaceutical industries. LBG is FDA-approved and safe to use as a food additive, (U.S Food & Drug Administration¹, 2019). The molecular structure of LBG is presented in figure 2.5.

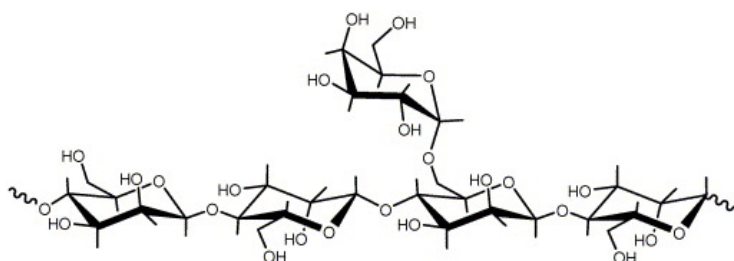


Figure 2.5: The molecular structure of LBG. The structure consists of a backbone that is built up by (1 $\rightarrow$ 4)-D-mannose units. A galactose unit is attached to the backbone as well. (Image used with permission from Coviello, Alhaique, Dorigo, Matricardi, and Grassi (2007))

Casas and García-Ochoa (1999) mean that if xanthan gum and LBG are mixed the viscosity of the mixture is higher than the sum of the viscosity of the separate polymer solutions. This is because of interactions between the backbone of xanthan gum and unsubstituted residues at the side chains of the LBG, according to the authors. Phillips and Williams (2009) mean that LBG together with xanthan gum create elastic reversible gels and Michon et al. (2004) prove in their study that the strain hardening properties of xanthan gum increases when the amount of LBG is increased.

2.2.3 Overview of the Polysaccharides Used in This Study

In the previous sections, alginate, xanthan gum and LBG were presented and described. However, the way the polymers match the material selection criteria chosen in section 1.1.1 has not been explained. In table 2.1 these aspects are presented again together with a description of how the chosen materials match these criteria.

Table 2.1: *The different criteria based on material selection theories, table 1.1, presented together with a description of how alginate gels and xanthan gum/LBG gels fulfill these criteria.*

Aspect	Alginate	Xanthan gum
Existing polymeric material	Yes, commercially available material	Yes, commercially available material
Biocompatibility	FDA-approved	FDA-approved
FDA-approval	Yes	Yes
Availability/ Supply	Available for purchase	Available for purchase
Safety	Non toxic	Non toxic
Commercial usage	Stabilizing and thickening agent	Stabilizing and suspending agent
Molecular structure	Copolymers of α -L-guluronic acid and β -D-mannuronic acid-units	Skeleton of β -D-glucose-units and a trisaccharide attached to every other glucose unit
Strain hardening properties	The strain hardening properties increase with increasing amount of Ca^{2+}	The strain hardening properties increase with increasing amount of LBG or konjac glucomannan
Gelling abilities	Gels with Ca^{2+}	Gels with LBG

Both polymers are used in the food industry to obtain a certain texture or viscosity in different products. The wide commercial use of the polymers means that they are available for purchase. As mentioned in section 1.1.1, Sapuan (2017) means that materials with good manufacturing possibilities should be chosen. Since alginate and xanthan gum are used commercially manufacturing strategies for these polymers exists, which indicate that the manufacturing possibilities for these materials are good.

As both polymers are FDA-approved, the product development process does not have to include getting the polymers FDA-approved. This saves time and cost and ensures good manufacturing possibilities for the polymers. According to Mogahzy (2009) important material properties should be considered when selecting material and since biocompatibility is essential when it comes to developing biomaterials this property is considered essential.

As mentioned in section 1.1.1, Faris et al. (2017b) emphasize that physical, electrical and mechanical properties also should be considered when selecting materials. In this research study polymers with different charge density were chosen for the laboratory work. According to Phillips and Williams (2009) alginate is built up of guluronic acid units and is negatively charged by a carboxyl group on every unit. The units that build up xanthan gum consist of five saccharides and since only one of them, a guluronic acid residue, has a negative charge the charge density of xanthan gum is lower than that of alginate. These differences in molecular structure made the two polymers suitable for this research study as the laboratory work then was based on polymers with different charge density. The strain hardening properties and gelling abilities of the polymers have also been considered since the purpose of the research study is to see whether alginate and xanthan gum have strain hardening properties.

2.3 Active Learning

In a traditional classroom, knowledge is conveyed by a teacher that stands in front of the students giving a lecture while the students take notes. This is a method that is still frequently used in schools and universities but that has been questioned since a traditional lecture does not enable the students to implement their new knowledge practically. Felder and Brent (2016) mean that this becomes a problem when the students go home to do their homework. As an alternative to this traditional way of teaching Felder and Brent (2016) present active learning as a way of teaching that

has been proved successful when it comes to student's long-term memory and deep learning. Active learning can be described as the opposite of traditional learning and aims to include the students in the lesson by asking them questions, giving them tasks to solve together, making them try to link the theory to real-life situations or predict an experimental outcome. This is according to Felder and Brent (2016) a way of teaching that activates the students which leads to that the students learn more and become more capable of applying the theories in practical work.

2.3.1 The 5E Model

One kind of activity based learning, the 5E-model, is presented by Tanner (2010). The author states that there are different aspects to consider when planning a lesson in order to create an atmosphere where learning is stimulated. First Tanner (2010) means that students need to be interested in what the lesson is about. Then the students must actively participate in the lesson by for example discussing the concepts brought up and compare new aspects with old ones in order to create a better understanding of the lesson content. The importance of processing new knowledge by revising earlier understandings is stated by Merrill (2002) who means that this is something that many teaching models have in common. Merrill (2002) means that in order for students to fully understand new concepts they must have the chance to see how the new knowledge changes their earlier beliefs.

To evaluate their understanding of the concepts Tanner (2010) further points to the importance of giving the students the chance to implement the knowledge practically during the lesson in order to see if they fully have understood the concepts. Based on the aspects, engaging and practically including students, Tanner (2010) developed the 5E model in order to facilitate lesson planning and student learning. The model consists of five key values: *engage*, *explore*, *explain*, *elaborate* and *evaluate*. By considering the first E, *engage*, the teacher makes an effort to make the lesson interesting enough to make the students pay attention. The purpose of the second E, *explore*, is to help students finding out what they already know and what new ideas they can get by using their existing knowledge to solve a task given by the teacher. When this is done the third E, *explain*, gives the teacher the opportunity to introduce the new concepts to the students that directly can adjust or develop their earlier knowledge with new information. After this the teacher can challenge the students by giving them more tasks where the students have to use the new knowledge to solve a problem. This is the elaboration phase where the teacher uses the fourth E, *elaborate*, to make the students use their newly generated knowledge

by solving a practical task. The last E, *evaluate*, aims to help the students reflect over the lesson to create an understanding of what they learned and what they have to study further.

By using these five concepts when planning a lesson Tanner (2010) means that the teacher creates a learning atmosphere where students are active and paying attention. This facilitates deeper learning and understanding of the concepts the teacher wants to mediate.

2.3.2 Cooperative Learning

Eilks and Hofstein (2013) describe methods for effective chemistry teaching and state that active learning stimulates learning in chemistry classrooms and enables deeper understanding among the students. Eilks and Hofstein (2013) specially highlight cooperative learning as a useful method when teaching chemistry. Cooperative learning is a type of active learning where the students instead of just listening to the teacher, talk to each other and exchange ideas. Instead of the teacher being in the center of attention Eilks and Hofstein (2013) mean that in a cooperative classroom the students are the ones talking during the lessons and the teacher can listen to their discussions and get a picture of what concepts they seem to have understood and what they need to study further.

2.3.3 Peer Discussion

The benefits of students discussing in the classroom were investigated by Smith et al. (2009). In their research study Smith et al. (2009) let students answer a question first without discussing the question together in groups. The number of correct answers were noted and the students were allowed to discuss in groups before answering again. The number of correct answers increased even in the groups where no students had answered correctly the first time. Smith et al. (2009) conclude that teachers' explanations are often not enough to get the class to understand the concepts. Even though the teacher's explanation might be considered as shorter or more correct than a fellow classmate's explanations, discussing with fellow classmates creates a deeper understanding of the concepts according to the authors.

3

Methods

In this chapter, the methods for the experimental part of the research project are presented. The laboratory work was conducted on the two chosen polymers, alginate and xanthan gum in combination with LBG, which were prepared and tested in the form of gels. Both gels were examined for their strain hardening properties using LAOS. Ionoprinting was also conducted in the case of calcium-alginate gels. The aspects that were taking into consideration when creating the lesson plan for a laboratory session in Swedish upper secondary schools are presented as well. First, the material for all of the laboratory work is presented.

3.1 Material

The polymers that were used for the gel preparation were alginate (Manugel DMB) from FMC BioPolymer, Xanthan gum, (Xantural 180) from CPKelco and LBG (GRINDSTED LBG 246) from Dupont. The salts that were used were calcium carbonate (Mikhart) from Provencale, GDL from Sigma Aldrich and sodium chloride from Acros Organics.

For the ionoprinting, petri dishes were used from Falcon. Aluminum foil was obtained from the lab. The copper agents in form of copper plate and copper rod were obtained from the workshop in the chemistry building at Chalmers University of Technology. The copper coins were privately owned.

The materials that were used for the laboratory session were an all around glue from Panduro, contact lens solution (ReNu) bought from a pharmacy, baking soda from Eldorado and food coloring from Dr. Oetker.

3.2 Gel Preparation

3.2.1 Calcium-Alginate Gel

The preparation of calcium-alginate gel was conducted in two separate steps, firstly by preparing an alginate solution and secondly by adding CaCO_3 and GDL to the solution to induce the gelation. The molar ratio between GDL and CaCO_3 was kept constant at 2. The alginate solution was prepared by mixing alginate, Milli-Q H_2O and 0.1 M NaCl in a beaker at 85°C for one hour. The concentration of the alginate dispersion was 2 wt%. For preparation of the gel, a mixture of CaCO_3 and GDL dispersed in Milli-Q H_2O was added to the alginate dispersion yielding a final alginate concentration of 1.5 wt%. A complete calculation for the preparation of calcium-alginate gel is presented in appendix C. The calcium-alginate gel was prepared for four different R-values, thus concentrations of Ca^{2+} , which are presented in table 3.1. The R value describes the ratio between added Ca^{2+} to the gel and the concentration of G-unit in the alginate. Thus, higher R value leads to higher concentration of Ca^{2+} . The first three R-values were used in the rheometer tests and all four during the ionoprinting tests.

Table 3.1: *The Ca^{2+} -concentration in relation to the R-value for the calcium-alginate gels. The R-value is the ratio between added Ca^{2+} and the concentration of G-unit in the alginate.*

R	Ca^{2+} [g/l]
1	1.09
0.75	0.82
0.5	0.54
0.25	0.27

3.2.2 Xanthan Gum/LBG Gel

The xanthan gum/LBG gel was prepared by heating Milli-Q H_2O to 90°C and then adding equal amounts of xanthan gum and LBG to the water after it reached the desired temperature. Equal amounts of xanthan gum and LBG were added for the entire research study, giving a 1:1 ratio between the polymers. The total polymer concentration was however varied and the tested concentrations were 1 wt%, 0.75 wt%, and 0.5 wt%. See appendix D for a sample calculation for the 1 wt% xanthan gum/LBG gel.

3.3 Shear Rheology

The experiments in this research study were conducted on a Discovery Hybrid Rheometer-3, presented in figure 3.1, and the geometry used for the tests on calcium-alginate gel was a ribbed plate and for xanthan gum/LBG gel was a flat plate, both with a diameter of 40 mm and the gap used was 0.5 mm. The experiments conducted on the rheometer were dynamic oscillatory shear tests and more specifically LAOS, which involves a nonlinear material response, which are methods described in section 2.1.1. The rheometer methods entailed two amplitudes sweeps (AS) up to 700% after the gel had set with 15 minute rest in between the amplitude sweeps. The amplitude sweeps were conducted at constant frequency and temperature, 1 Hz and 25°C. The calcium-alginate and xanthan gum/LBG hydrogels were allowed to set on the rheometer before the amplitude sweeps begun. The xanthan gum/LBG gel required extra time sweeps with a temperature ramp to gel properly on the rheometer as it gels very quickly in room temperature. The analysis methods of the rheometer test results were plotting the reduced moduli as a function of strain as a way of examining strain overshoots, described in section 2.1.1.



Figure 3.1: *The Rheometer used for LAOS testing, Discovery Hybrid Rheometer-3. The computer controls the rheometer by defining what method should be used. The sample is loaded on the metal plate that can be seen in the center of the rheometer. During a test, the front part of the rheometer is lowered down towards the plate and a pressure and torque is applied to the sample. The changes in stress, complex, storage and loss moduli are measured by the computer, which can then be analyzed in order to examine the properties of the sample. (Photograph taken by thesis authors)*

3.4 Ionoprinting on Calcium-Alginate Gels

The ionoprinting was conducted as a way to induce stiffness in hydrogels as described in section 1.1. Calcium-alginate gels were prepared in the same manner as described in section 3.2.1 and letting the gels set in gel molds prior to the ionoprinting was conducted. The gels were prepared in a variation of different sizes and shapes. In table 3.2 a specification of the gels dimensions is presented with their respective sizes.

Table 3.2: *A specification of the gels' different sizes and shapes prepared for the ionoprinting. The lengths and widths for the rectangular gels as well as the diameters for the circular gels were measured and the heights for all of the gels were calculated.*

Gel	Length (cm)	Width (cm)	Calculated height (cm)
	Diameter (cm)		
20 ml rectangular	11	2	0.91
10 ml rectangular	11	2	0.45
30 ml circular	10		0.38
15 ml circular	10		0.19
5 ml circular	3.5		0.13

After a gel had set it was released from the mold and placed on an aluminum foil sheet and a copper agent was put on top of the gel. An electric field was then created by applying electrodes to the aluminum as well as the copper. The cathode was applied to the aluminum foil sheet and the anode to the copper agent. The voltage used in the experiments was 30 V and was applied for approximately 10-20 seconds. Figure 3.2 shows a schematic overview of the laboratory setup for the ionoprinting.

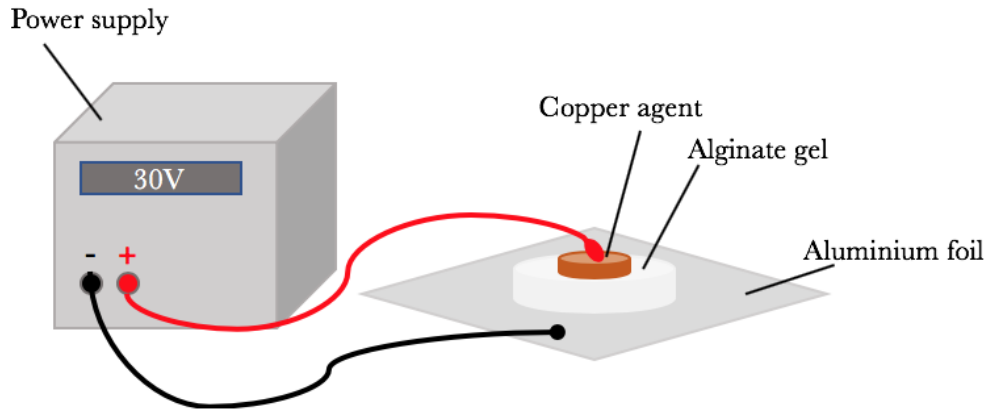


Figure 3.2: A schematic sketch of the ionoprinting laboratory setup. The image shows how an calcium-alginate gel was laid on an aluminum foil and how the copper agent then was placed on top of the gel. A power supply was used to create a voltage between the copper agent and the aluminum foil. The aluminum foil was connected to the cathode end of the power supply and the anode was applied to the copper agent. (Created by thesis authors in PowerPoint)

The ionoprinting test was conducted on gels with the four different values of R ; $R = 1$, $R = 0.75$, $R = 0.5$, and $R = 0.25$. The different gels were printed with different copper agents, which are presented in table 3.3. After the ionoprinting, the copper dispersion in the gels was documented with photographs and measurements, of the blue areas in the gels, for predetermined time intervals for two weeks after the tests.

Table 3.3: The ionoprinting testing scheme, which defines what copper agent is used for what gel.

R	20 ml rectangular	10 ml rectangular	30 ml circular	15 ml circular	5 ml circular
1	The edge of a copper plate	The edge of a copper plate	Swedish 2 kr	Euro 5 cent	The end of a copper rod
0.75	The edge of a copper plate	The edge of a copper plate	Swedish 2 kr	Euro 5 cent	The end of a copper rod
0.5	The edge of a copper plate	The edge of a copper plate	Swedish 2 kr	Euro 5 cent	The end of a copper rod
0.25	The edge of a copper plate	The edge of a copper plate	Swedish 2 kr	Euro 5 cent	The end of a copper rod

3.5 Laboratory Session for Students in Swedish Upper Secondary Schools

The lesson plan for a laboratory session for students in Swedish upper secondary schools was developed with the aim to increase public knowledge on strain hardening and viscoelasticity. The development process included inventing a suitable laboratory exercise and fitting it into a course by taking the course curriculum into consideration. The lesson plan was formed in accordance with the theories of active learning mentioned in section 2.3, more specifically the 5E model.

4

Results

The results from the laboratory work, which include shear rheology tests and the ionoprinting tests, are presented in this chapter. The finished lesson plan for introducing viscoelasticity and strain hardening for students in Swedish upper secondary schools is presented as well.

4.1 Laboratory Work

4.1.1 Rheological Properties of Calcium-Alginate gel

The evolution of storage modulus (G') and loss modulus (G'') of calcium-alginate gel during the gelation process is presented in figure 4.1. The intersection of G' and G'' indicates the gel point, which is at approximately three minutes for the calcium-alginate gel at the prevailing conditions.

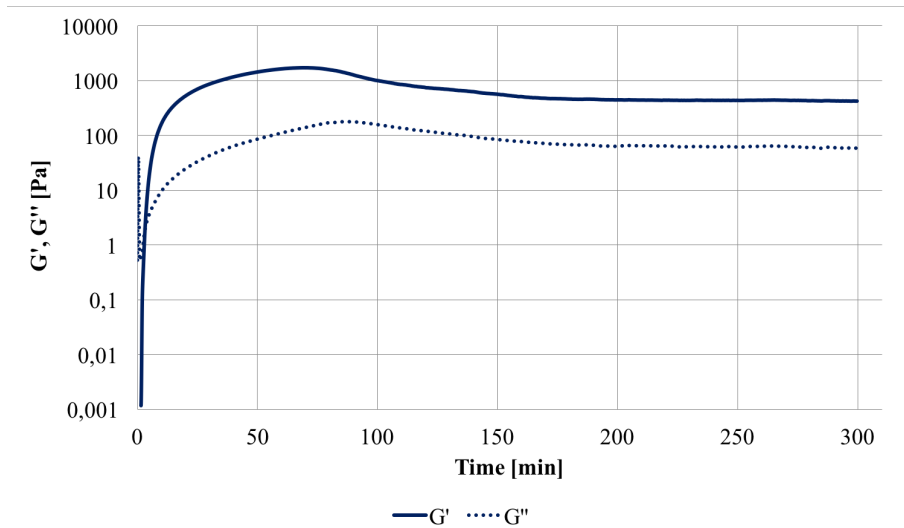


Figure 4.1: *The evolution of storage and loss modulus as a function of time for 1,5% alginate gel ($R=1$) at 25°C, 1% strain and 1 Hz.*

In figures 4.2 the reduced storage- and loss modulus, G'/G'_0 and G''/G''_0 , are presented as a function of strain of both amplitude sweeps for all of the Ca^{2+} -concentrations. G'_0 and G''_0 represent the modulus in the linear viscoelastic region, which is the region where the graphs are linear. The deviation of the linearity is described as strain overshoot and indicate strain hardening behavior. In these diagrams a variation of strain overshoots can be detected for the different Ca^{2+} -concentrations. The strain overshoot is the most prominent for the first amplitude sweep for the calcium-alginate gel with the highest Ca^{2+} -concentration, which starts at approximately 30% strain.

4. Results

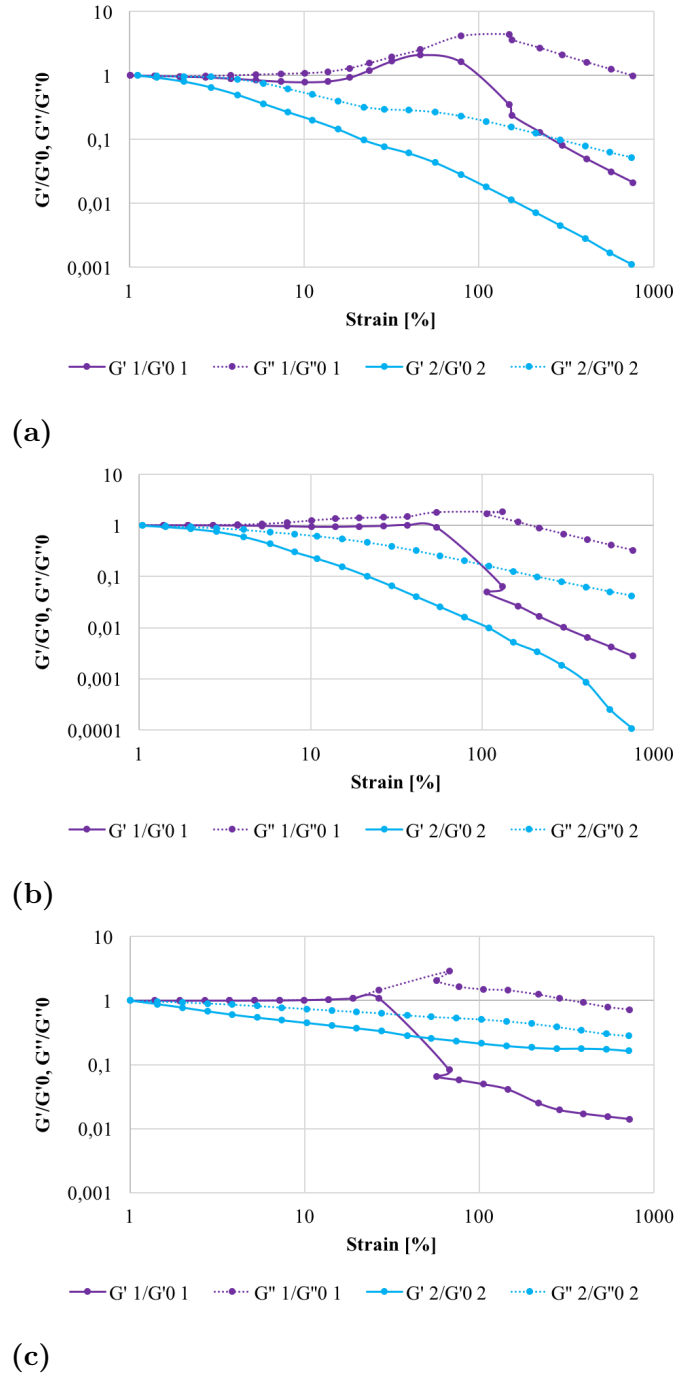


Figure 4.2: The reduced storage modulus (G'/G'_0) and loss modulus (G''/G''_0) as a function of strain for calcium-alginate gels with varying concentration of Ca^{2+} , (a) $R=1$, (b) $R=0.75$, (c) $R=0.5$, for two amplitude sweeps with 1-700% strain with a 15 minute rest in between at 25°C and 1 Hz. The first amplitude sweep is presented in purple and the second in blue. The strain overshoot is most prominent for the gel with the highest Ca^{2+} -concentration and gets more obscure as the concentration decreases.

4.1.2 Rheological Properties of Xanthan Gum and LBG gel

The gelling process of xanthan gum and LBG on the rheometer is presented in figure 4.3. Unlike the calcium-alginate gels, the temperature for xanthan gum and LBG gels is not kept constant. The temperature starts at 70°C and is then gradually decreased to 25°C. The storage modulus (G') intersects the loss modulus (G'') at approximately six minutes and 55°C, which is where the gel point is defined at the prevailing conditions.

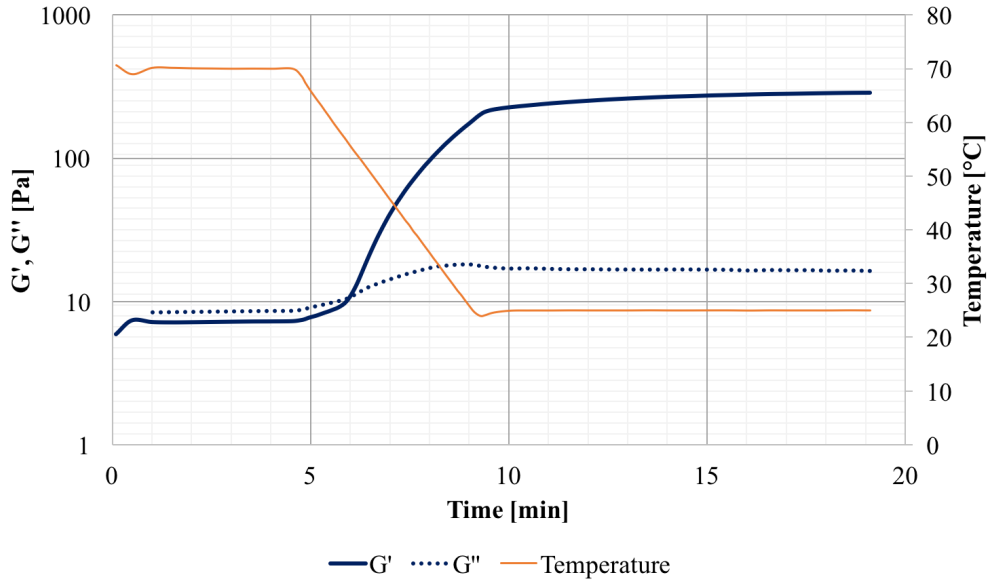


Figure 4.3: The evolution of storage and loss modulus as a function of time before the first amplitude sweep for xanthan gum and LBG. The image shows results for 1% xanthan gum/LBG gel, which starts at 70°C and is decreased to 25°C, 1% strain and 1 Hz.

Figures 4.4 present the reduced storage modulus (G'/G'_0) and reduced loss modulus (G''/G''_0) as a function of strain for both amplitude sweeps. The reduced moduli show similar strain overshoot graphs for both of the amplitude sweeps for all of the polymer concentrations. The strain overshoot occurs at the lowest strain, at lower than 100% strain, for the highest polymer concentration and at the highest strain, at higher than 100% strain, for the lowest polymer concentration.

4. Results

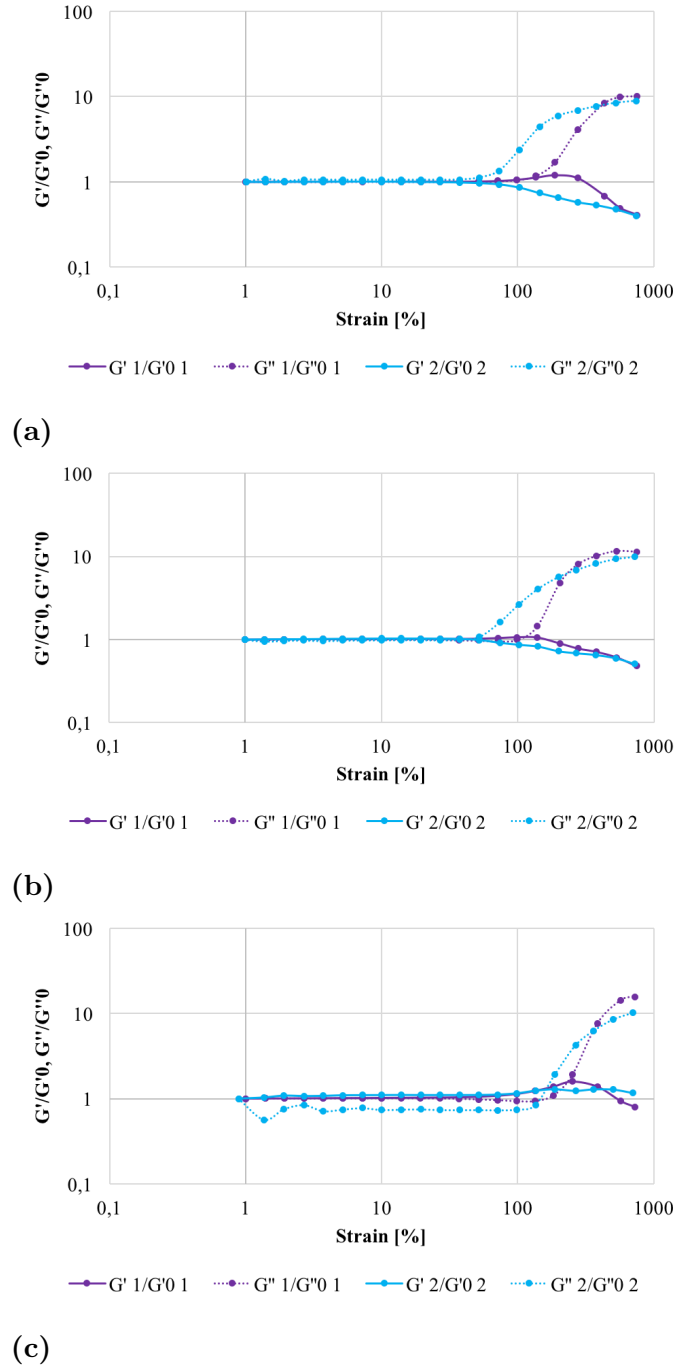


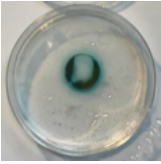
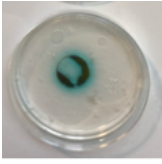
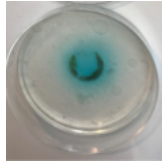
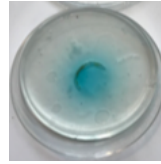
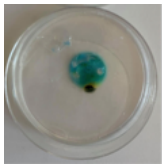
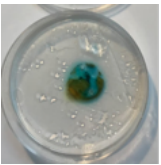
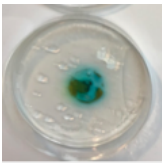
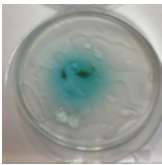
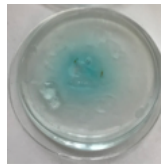
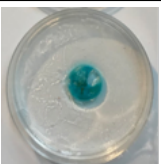
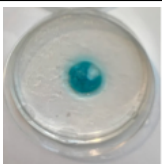
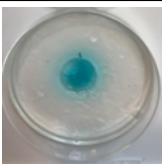
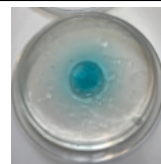
Figure 4.4: The reduced storage modulus (G'/G'_0) and loss modulus (G''/G''_0) as a function of strain for xanthan gum gels with varying polymer concentration, (a) 1%, (b) 0.75%, (c) 0.5%, for two amplitude sweeps with 1-700% strain with a 15 minute rest in between at 25°C and 1 Hz. The first amplitude sweep is presented in purple and the second in blue. The strain overshoot phenomenon starts at the lowest strain level for the highest polymer concentration and at the highest strain level for the lowest polymer concentration.

4.1.3 Ionoprinting on Calcium-Alginate Gels

All of the gels with the lowest Ca^{2+} -concentration, $R=0.25$, were too weak and broke either from releasing them from the gel mold or during the ionoprinting and are therefore not included in the results.

In table 4.1, the copper ion dispersion after ionoprinting is presented for the circular 30 ml calcium-alginate gel. As the copper ions are induced in the gel, they color the gel blue. The diffusion can be seen in the photographs as the blue color, the copper ions, spreads over the gel. The diffusion is illustrated with a selection of photographs taken at specific times after the tests as well as the measured radius of the copper ion dispersion at that time.

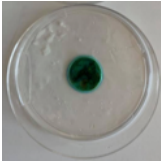
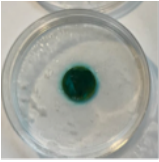
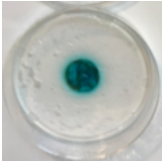
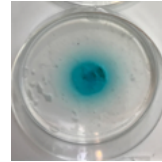
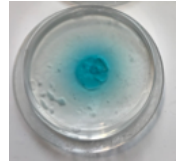
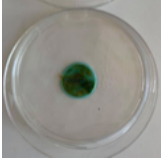
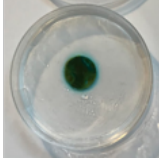
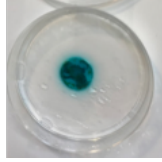
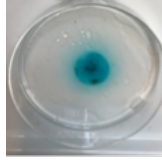
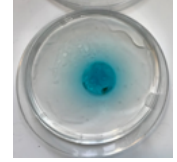
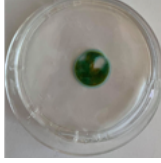
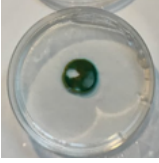
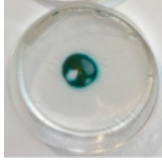
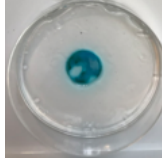
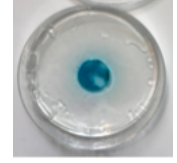
Table 4.1: *The copper ion dispersion development after ionoprinting with a 2 kr Swedish coin for the 30 ml calcium-alginate gel. The diffusion, which increases over time, is documented by photographs as well as the measured radius of the copper ions. (Photographs taken by thesis authors)*

R	0 hours	6 hours	24 hours	168 hours	336 hours
1	2.3 cm	2.6 cm	3.0 cm	4.0 cm	6.0 cm
					
0.75	2.3 cm	2.5 cm	3.0 cm	3.7 cm	5.0 cm
					
0.5	2.3 cm	2.1 cm	2.7 cm	3.2 cm	4.5 cm
					

4. Results

The results for the 15 ml calcium-alginate gels are presented in the same way as for the 30 ml, which can be seen in table 4.2. These gels were molded in the same size of petri dish and the one thing that sets them apart is the amount of gel and thereby the height of the gels. However, they were printed with a different coin of slightly different size. The copper ions dispersion is the greatest at the highest Ca^{2+} -concentration for both the 30 ml and 15 ml circular gels and decreases with lower concentration of Ca^{2+} .

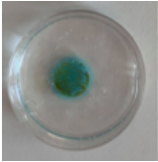
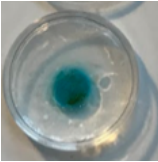
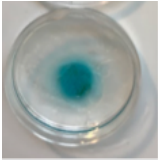
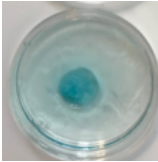
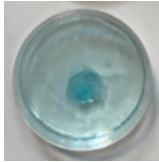
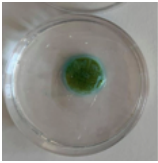
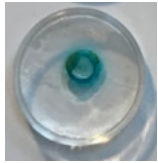
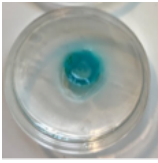
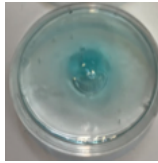
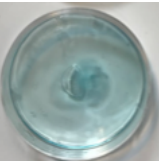
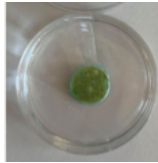
Table 4.2: *The copper ion dispersion development after ionoprinting with a 5 cent euro for the 15 ml calcium-alginate gel. The time dependent increase of diffusion is presented with photographs of the gels as well as the measured radius of the copper ions. (Photographs taken by thesis authors)*

R	0 hours	6 hours	24 hours	168 hours	336 hours
1	2.1 cm	2.4 cm	2.7 cm	3.7 cm	4.5 cm
					
0.75	2.1 cm	2.4 cm	2.4 cm	3.5 cm	4.5 cm
					
0.5	2.1 cm	2.3 cm	2.4 cm	2.2 cm	3.0 cm
					

4. Results

The results from the final ionoprinting test of the circular gels are presented in the same manner as previous tests and can be seen in table 4.3, where the smallest gels were printed with the end of a copper rod. The calcium-alginate gel with the lowest Ca^{2+} -concentration, $R=0.5$, was so fragile that it ruptured after the test and could not be further documented. Visually, the copper ions did initially not disperse at all and remained local but after two weeks the copper ions had spread over the entire gels.

Table 4.3: *The copper ion dispersion development after ionoprinting with the end of a copper rod on 5 ml calcium-alginate gel. The copper ion dispersion increases over time, which is presented with photographs of the gel and the measured radius of the diffusion. (Photographs taken by thesis authors)*

R	0 hours	6 hours	24 hours	168 hours	336 hours
1	1.0 cm	1.3 cm	1.2 cm	3.2 cm	3.2 cm
					
0.75	1.0 cm	1.2 cm	1.0 cm	3.2 cm	3.2 cm
					
0.5	1.0 cm				
					

In figure 4.5, the 20 ml rectangular calcium-alginate gel with the highest Ca^{2+} -concentration, $R=1$, is presented as an example of how the ionoprinting was conducted at one end of the gel. The photographs are taken just after the ionoprinting was conducted and then two weeks later. As can be seen in the photographs, the ionoprinting is visible and Ca^{2+} ions were transferred to the gel and that they diffused over the two weeks. The gel shrunk during the two weeks, which can be seen in the figure as well.



Figure 4.5: *The 20 ml rectangular calcium-alginate gel, $R=1$, photographs taken just after the ionoprinting was completed and two weeks later. The copper ions have diffused over the gel during the time period as well as shrinkage of the gel. (Photographs taken by thesis authors)*

4.2 Laboratory Session for Students in Swedish Upper Secondary Schools

A lesson plan for a laboratory session was developed for students that attend the first chemistry course, Kemi 1, in Swedish upper secondary schools. The lesson plan's intention is to present viscoelasticity and strain hardening in an informative and educational way that fits into the course plan. The complete lesson plan is divided into two parts, information for the teacher and laboratory manual for the students. The teaching information includes what learning outcomes are expected, how the laboratory exercise fits into the course plan, a short section of theoretical background on the subject and a suggestion on how to execute the lesson in accordance with the 5E model presented in section 2.3.1. The experimental part of the lesson involves creating a clay that imitates the attributes of the commercial toy "Silly Putty", which will be used to describe strain hardening properties. How the clay responds to different kinds of stimuli is illustrated with photographs in figure 4.6. If the clay is pulled apart slowly and with low force, it can stretch out very long. If the clay on the other hand is pulled apart quickly and with much force, it snaps and creates a clean cut. After the experimental part of the lesson, the students will

discuss a couple of questions related to viscoelasticity and strain hardening, first in pairs and then with the entire class. The developed lesson plan is presented in full in appendix E. However, the lesson plan is in Swedish as it is designed to accommodate the first chemistry course in Swedish upper secondary schools.



(a) *Initial state of the clay*



(b) *If the clay is pulled apart with little force from the initial state, it will stretch out*



(c) *If the clay is pulled apart with much force from the initial state, it will snap with a straight cut*

Figure 4.6: *The photographs illustrate how the "Silly Putty" clay behaves when it is pulled apart. When the clay is pulled apart slowly, the weak bonds break but the strong chains can slide over each other and untangle. If the clay is however pulled apart quickly, the chains do not have time to untangle so they break. (Photographs taken by thesis authors)*

5

Discussion

This chapter begins with a method discussion motivating the method choice of this research study. Thereafter, the experimental results from the research study is discussed in relation to previous research. The economical and sustainability aspects are discussed as well as the laboratory session for students in Swedish upper secondary school. In order to give a picture of what steps could be taken next when it comes to the different parts of this research study the chapter ends with a discussion of further research.

5.1 Method discussion

A literature review was thoroughly conducted before starting the research study. Literature regarding strain hardening properties of different polymers were searched for in four databases: SciFinderⁿ, Knovel, Scopus and Web of Science. This can be seen as a full coverage of literature based on the search criteria used: strain hardening and different polymer names. A description of the literature review is presented in appendix A and the full search history can be found in appendix B. During the research it was noticed that strain hardening also is referred to as strain stiffening in the literature. This has resulted in that more useful literature has been found during the research study. It can therefore be argued that the literature study should have included strain stiffening as an alternative to strain hardening in order to find more useful literature in the prestudy.

The rheometer tests that were performed are well known and commonly used methods when it comes to rheology studies. The common use of the methods that have been used in this research study increases the validity of the methods. During the laboratory work more experiments than the ones presented in this report were performed. This was a way of investigating different ways of identifying strain hardening properties of calcium-alginate and xanthan gum/LBG gels. The experiments presented in the report have not been repeated which decreases the validity of the results. Previous studies have however been used as a base for the result analysis

in order to increase the validity and credibility of the drawn conclusions. There are different plates to choose from regarding the rheometer, the reason for why the calcium-alginate gels were tested with a ribbed plate was to avoid slip, which it had shown tendencies to in previous tests.

The method used for the ionoprinting was based on previous work. However, as ionoprinting on polysaccharides has not been previously conducted, further experiments need to be performed to develop the method in order to generate desired results.

The method for developing the lesson plan for a laboratory session for students in upper secondary schools was based on well-known learning theories as well as it was anchored and adjusted to fit the course curriculum. However, the method could have included a testing process, that is testing the lesson plan with students and adjusting the lesson plan as needed.

5.2 Laboratory Work

5.2.1 Strain Hardening Properties of Calcium-Alginate Gel

The diagrams that present the reduced storage modulus and the reduced loss modulus as a function of strain, figure 4.2, show the most prominent strain overshoot for the highest Ca^{2+} -concentration. Strain overshoot is, as previously mentioned in section 2.1.1, an indication of strain hardening according to Hyun et al. (2002). As the strain overshoot was the most outstanding for the highest Ca^{2+} -concentration in calcium-alginate gels, it can be concluded that increased concentration of Ca^{2+} can contribute to increased strain hardening properties. This is in agreement with the results published by Zhang et al. (2007), who presented the same conclusion that strain hardening increases by increased amount of Ca^{2+} .

It can also be detected from the charts that the graphs for the second amplitude sweep do not behave in the same manner as in the first amplitude sweeps. There is no sign of any strain overshoot and the graphs do not even increase linearly in the linear viscoelastic region. The most probable cause for that deviation is rupture in the gels during the the first amplitude sweep as the strain increased. This can be confirmed by Michon et al. (2004), as they argue that a gel made of pectin and alginate shows strain hardening properties at shear strain between 50-110% before it breaks. It seems that the gel with the lowest Ca^{2+} concentration ($R=0.5$) ruptured

at the lowest strain. The reason for that might be that the gel was too weak to handle the increased strain.

5.2.2 Strain Hardening Properties of Xanthan Gum/LBG gel

The reduced moduli was presented as a function of strain for the xanthan gum and LBG gels in figure 4.4. As can be seen in the charts, G' decrease and G'' increase at first and then decrease as the strain is increased. Hyun et al. (2002) identifies this as a weak strain overshoot as described in section 2.1.1. Strain overshoot is a indicator of strain hardening, which is the most prominent for the highest polymer concentration as the deviation begins at the lowest strain. This indicates that strain hardening properties are dependent on polymer concentration. As previously mentioned, Michon et al. (2004) concluded in their study that strain hardening properties increased in a xanthan gum/LBG gel with increased concentration of LBG. Therefore, how strain hardening properties are affected by keeping the ratio between xanthan gum and LBG constant (1:1) and varying the total polymer concentration was investigated instead.

Unlike the calcium-alginate gels, the graphs for both amplitude sweeps accompanies one another quite similarly for the xanthan gum/LBG gels, which indicates that the gel recovered almost fully from the first amplitude sweep. The fact that the xanthan gum/LBG gels recovers quickly indicates that there is interaction between the polymers as described in section 2.2.2. That conforms with Phillips and Williams (2009) statement that the combination of xanthan gum and LBG create an elastic reversible gel when mixed.

5.2.3 Comparison of Calcium-Alginate and Xanthan Gum/LBG Gels

The results show that the calcium-alginate gel breaks at a certain point and the xanthan gum/LBG gel is more elastic and does not break. John et al. (2019) explain this by suggesting that the bundles in the alginate gel break, leading to a strain thinning behavior. This is the most distinct difference between the behavior of the two gels, calcium-alginate and xanthan gum/LBG, under the shear rheometer test. As the calcium-alginate gels ruptured at under 100% strain in the first amplitude sweep. The xanthan gum and LBG gels on the other hand did not and recovered

almost perfectly during the rest period between amplitude sweeps and show strain hardening properties at higher strains. The calcium-alginate gel does not have the same elasticity properties and therefore ruptures as the strain is increased. This however is not necessarily a disadvantage for alginate as the strain hardening properties are displayed at similar strain levels as actin and other proteins that build up soft body tissue as mentioned in section 1.1.

5.2.4 Ionoprinting on Calcium-Alginate Gels

As mentioned in 4.1.3 gels with the lowest Ca^{2+} -concentration, $R=0.25$, did not gel properly and therefore broke when released from the molds or during the ionoprinting. The reason for that is as previously described by Zhang et al. (2007) alginate requires an addition of cations, such as Ca^{2+} , in order to gel.

The results from the ionoprinting show that it is possible to perform ionoprinting on polysaccharide gels. Earlier this has only been done on synthetic polymers and therefore this is a new contribution to the scientific field. The results from the experiments in this thesis show that it is possible to insert a new mechanical property, stiffness, in calcium-alginate gels. It is possible that this is a way of creating materials with a gradient of mechanical properties similar to those in human body tissue and therefore further investigation on this field is a possible way of developing new biomaterials.

A result that could not be measured within our planned method but was identified was the fact that the gel stiffened where the copper ions were printed. This was discovered by lightly pressing the gel with a finger in different spots and it was distinctly harder where it had been printed. As the copper ions diffused over the gel, the local stiffness from the ionoprinting was perceived to spread out with the dispersion.

When conducting the ionoprinting, it was experienced easier to print on the thinner gels. Which probably has to do with the fact that the thinner the gel is, the shorter the distance between the anode and cathode is. This leads to a greater electric field over the gel in comparison to a thicker gel. The diffusion of copper ion in the gels also seems to be greater for the thicker gel than the thinner as the blue area is bigger when comparing the results for the 30 ml circular gel, table 4.1, and the 15 ml circular gel, table 4.2.

The rectangular gels that were ionoprinted with the edge of a copper plate, show

that it is possible to induce copper ions in which ever shape that is desired. That gives rise to the possibility to create anisotropic properties in the gel in any wanted shape. It would for example be possible to ionoprint a letter or to create a star.

The gels were printed with different copper agents. Even though copper ions imprinting was successful for all of the agents, in hindsight it might have been better to use the same agent for all gels. Especially concerning the larger circular gels (15 ml and 30 ml) that were not printed with the same agent, as the comparison between the gel sizes was not considered enough in favor of comparison of Ca^{2+} -concentration, for each gel size and shape, when the method was determined. It is unfortunately impossible to determine the copper content of coins as that information is not available for the public and therefore hard to conclude if more or less copper ions were imprinted from each coin. However, Palleau et al. (2013) describes that by using electric fields and thereby controlling the duration of ionoprinting and the magnitude of voltage applied to the gel that it is possible to manage the amount of ions imprinted in the gel. Therefore, by using a copper agent with known copper content it should be possible to control the amount of ions injected to the gel.

It must also be noted that the measurements of the copper ion diffusion where executed manually with a simple ruler. That can be regarded as a source of error as it is more likely that a measurement was wrong than that the copper ions reduced and diffused alternately. However, to minimize the error source the same person executed all of the measurements with the same ruler.

5.3 Laboratory Session for Students in Swedish Upper Secondary Schools

Viscoelasticity and strain hardening are complex concepts and tricky to explain to upper secondary school students. The developed lesson plan is therefore designed with the aim to introduce the subject in an easy, educational and activating way. The laboratory exercise involves creating a clay that resembles the toy "Silly Putty" and by testing the properties of that clay, explaining the terms viscoelasticity and strain hardening.

As described in section 1.1.1.1, Mazzucato (2018) explains that there is an urgency in society to engage citizens in a broader spectrum of science in order to create more targeted research. Aikenhead (2005) argues that education should prepare all stu-

dents for science consumption as it would increase the general science knowledge of the entire society as a whole. By introducing scientific research to students earlier, it might influence them to continue on the scientific path. However, school courses in Swedish upper secondary schools must follow certain curriculum and course plans, which leads to a dilemma for the teachers. Science simply cannot be taught in every single course. There is however a possibility to include different aspects of science into the main science courses as chemistry, biology, physics and general science.

Viscoelasticity is not a part of the first chemistry course and therefore perhaps not an ideal match to teach in the course curriculum. However, by describing viscoelastic properties with the help of a "Silly Putty" clay, chemical bonds and their influence on for example occurrence and properties can be introduced. The content that Skolverket has decided should be covered in the first chemistry course that can be applied to this laboratory exercise as well as the learning objectives is presented in the lesson plan in appendix E.

The first chemistry course in Swedish upper secondary school is only mandatory for the science and technology programs. Therefore, even though viscoelasticity can be introduced in the chemistry course, the knowledge would not reach all upper secondary school students.

Introducing ionoprinting in a laboratory session was also considered as a possible option. It was decided as too time consuming for a single laboratory session as the gels had to be made, molded and set before ionoprinting could be executed. The primary reason for not choosing ionoprinting as a laboratory session was however that the laboratory part of ionoprinting was determined as too dangerous, which would require too much from the teacher. This was concluded after the ionoprinting was conducted for the research study as it was experienced as fairly easy to get electrocuted depending on the gel and voltage. It could have been an option to change the laboratory session to a demonstration. The active learning could then have been in form of discussion between students. Since the intention of this research study, however, was to develop a laboratory session regarding strain hardening properties the "Silly Putty" became an easier way to help students understand this concept.

The lesson plan has not been tested in an educational environment yet as all teaching in upper secondary schools in Sweden was conducted remotely for the spring of 2020. The laboratory part of creating a "Silly Putty" clay has however been tested by the thesis authors during the research study with great success. The clay recipe that was developed during the research study displayed the desired strain hardening behavior.

5.4 Economical Aspects of the Material Selection

Before the start of this research study, materials used in the laboratory work were carefully selected according to nine aspects based on the material selection theory in section 1.1.1. According to Edwards (2014), the material selection process is a complicated process that can be performed in many different ways with special material selecting methods. In this study no particular material selection method were used. Instead nine aspects were taken into consideration when selecting the materials that later were used in the laboratory work. This has been a way of working for targeted research that Mazzucato (2018) discusses. By taking aspects like availability, biocompatibility, commercial usage, safety and mechanical properties into consideration when selecting material for the laboratory work, the research study supported the long term goal: manufacturing of artificial blood vessels with mechanical properties similar to human body tissue. This has also been a way of taking the economical sustainability of the research study into consideration.

Sapuan (2017) means that aspects like maintenance, transport and quality control should be considered in the material selection process. It can be argued that these aspects were not considered when selecting material to this research study. However, by using existing materials that are used commercially, methods for these aspects already exists and the aspects were therefore not considered as important as assuring that the materials were biocompatible and safe to use. It can also be argued that aspects like user-interactions were not taken into account. Rodriguez, Peças, Carvalho, and Orrego (2020) however describe in their article how sustainability nowadays is an important criteria for customers. The authors mean that companies must strive for safe products with small environmental effects. It can therefore be argued that user-interactions actually were included when selecting material since the materials that were selected are renewable and considered to be safe to use. The environmental sustainability has also been taken into consideration due to this aspect. By choosing materials that are renewable the environmental impact of the research study can be considered to be minimized.

5.5 Further Research

In future laboratory tests, it would be advised to conduct each experiment more than once to avoid any potential standard faults. The results that were generated from the rheometer tests show that calcium-alginate rupture at a certain point and

xanthan gum/LBG does not. Therefore, it could be interesting to see what would happen if alginate, xanthan gum and LBG were mixed. The mixture could be exposed to the same tests as performed here and it is interesting to see if those result would show that the gel mixture have the strain hardening properties of alginate but also is elastic like the xanthan gum/LBG gel. Could this be a way of solving the problem with low ductility that Cui et al. (2014) describe when it comes to synthetic hydrogels?

In this research it was ensured that the alginate and xanthan gum were FDA-approved. The European MDR, medical device regulation, will be launched in May 2021 according to the Commission European (2020). Are alginate and xanthan gum MDR-approved as well? If they are, it means that they meet the European standards when it comes to medical devices which can be considered necessary since future products should not just be approved for usage in the United States.

For the ionoprinting experiments carried out in this thesis the voltage was held constant. The calcium concentration and the shapes as well as height of the gels were varied. The voltage showed to be too high for the thin gels which resulted in that the copper burned right through the gels. Therefore further research could include testing varying the voltage to compensate for the differences in height of the gels.

The results from the ionoprinting show that it is possible to insert local stiffness in calcium-alginate gels and that this could be a way of mimicking mechanical gradients of body tissue. If it is possible to create biomaterials with mechanical gradients, is it also possible to make the polysaccharides respond to a mechanical stimuli like blood pressure?

The copper ions gel with alginate and create local stiffness but as the copper ions diffused over time, the mechanical properties could change as well. How does the mechanical properties change with the diffusion and does that effect the usage of the material? As a way to facilitate these further studies, image analysis is suggested to analyze the diffusion of the copper ions. This could be a way of measuring the blue area of the gels, the area of the copper ions, with definition of were the line goes between area of copper ions and gel without copper ions.

Mazzucato (2018) argues that a way to create more targeted research is to conduct more cross-disciplinary work, which aligns with Aikenhead's (2005) statement that teaching should focus on preparing students for consuming science to increase the science knowledge in the society. This will not be achieved by just introducing dif-

ferent aspects of science and presenting research in science oriented programs as the Swedish upper secondary schools. Could the laboratory session lesson plan be introduced in Naturkunskap, a course in general science, as well in order to reach a broader audience?

The aspects or criteria that were chosen in the material selection process did not include recyclability. The materials are renewable but since recycling is a big part of today's society it would be interesting to investigate the recyclability of alginate and xanthan gum further in order to increase the environmental sustainability of the materials.

This research study has been based on a strive of achieving more targeted research as a way of increasing the possibility of developing commercially successful products. The effects of this problem-based way of performing research has not been measured in this study. Is targeted research a way of generating more innovation of the research performed? In order to see what effects targeted research have on science, it is suggested that future research strive for targeted research as well, and that the effects are measured.

6

Conclusion

The foundation of this thesis is based on a strive for targeted research. Even though the effects of this way of conducting research were not measured, it increased awareness of how materials can be selected in order to facilitate future commercialization. The aspects considered during the research study are: existing polymeric material, biocompatibility, FDA-approval, availability, safety, commercial usage, molecular structure, strain hardening properties, and gelling abilities. These aspects were chosen with the goal to increase the possibility that the research could eventually result in identifying polymeric materials with good manufacturing possibilities.

Calcium-alginate gel and xanthan gum/LBG gel both display strain hardening behavior to some extent in this research study. Just like protein-based hydrogels calcium-alginate gels and xanthan gum/LBG gels show strain hardening properties at large deformation and high strain. Their strain hardening properties could therefore make them suitable for artificial soft body tissue engineering. It can be concluded that calcium-alginate gel displays strain hardening behavior in very similar shear strain interval as protein based hydrogels that have been used to build up artificial soft body tissues and can therefore be considered more suitable than xanthan gum/LBG gels. It can also be concluded that it is possible to conduct ionoprinting on polysaccharides as alginate. This makes ionoprinting an interesting future research field since this could be a way of developing biomaterials with viscoelastic properties more similar to those of human body tissue than materials existing today.

Viscoelasticity and strain hardening can be presented and discussed with a laboratory exercise that entails creating a clay similar to "Silly Putty". The lesson plan is designed in accordance with the 5E model to achieve the positive effects of active learning. Even though the lesson plan has not been tested in a teaching environment, it can be determined as relevant as it is thoroughly planned, anchored in the course curriculum, and based on active learning.

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A

Literature Review of the Polymers

The literature review was initiated with identifying renewable polymers that are FDA-approved, excluding proteins and synthesized polymers. The literature review was conducted for the polymers that were identified likely to have the desired strain hardening properties. The search was conducted in a manner that strain hardening was combined with each polymer name and their synonyms, such as "strain hardening" AND (polymer name OR synonym 1 OR synonym 2 OR ... OR synonym n). Articles and conference proceedings were retrieved from four databases, SciFinder[®], Knovel, Scopus and Web of Science. The literature retrieved from the databases was limited to include only English results. A complete scheme over the search is shown in appendix B, where the exact search phrases are presented along with dates and how many hits were in each database. As can be seen, two of the polymers, galactomannans and ethylcellulose, did not get any hits at all.

Table A.1 illustrates the total number of search hits for each polymer before and after duplicates were removed as well as how many articles were found interesting after two screenings of the abstracts. The first screening was divided between the team members for efficiency and resulted in 86 articles. A second control of the abstracts that made it through the first reading was then conducted by both team members, resulting in 46 articles for the eleven remaining polymers. The 46 articles were however in fact only 31, as there was an overlap of articles between the polymers. The articles were divided between the team members and thoroughly analyzed.

Table A.1: *An illustration of how the number of articles in the literature review were successively reduced.*

Polymer	Number of articles...			
	per polymer	minus duplicates	after 1 st screening	after 2 nd screening
Alginate	19	10	3	3
Bacterial cellulose	5	3	3	1
Carboxymethylcellulose calcium	23	18	3	2
Carrageenan	28	11	5	4
Cellulose acetate phtalate	258	229	11	6
Cellulose, powdered	19	18	7	4
Hydroxyethylcellulose	121	65	16	9
Hydroxypropylcellulose	120	66	20	10
Methylcellulose	156	154	7	3
Pectin	19	8	5	2
Xanthan gum	16	10	6	2
Total	784	592	86	46

In figure A.1, an illustration of the literature review is presented in the form of a flow chart. The flow chart illustrates how the articles as well as polymers were narrowed down through the process of the literature review. The original thirteen polymers were as mentioned earlier initially narrowed down to eleven and then finally down to three. The three remaining polymers: alginate, carrageenan, and xanthan gum were chosen for the laboratory work. However, as can be seen in the flow chart carrageenan is overlined because it was decided to rather do more tests and focus on the two polymers than to do fewer tests on more polymers.

A. Literature Review of the Polymers

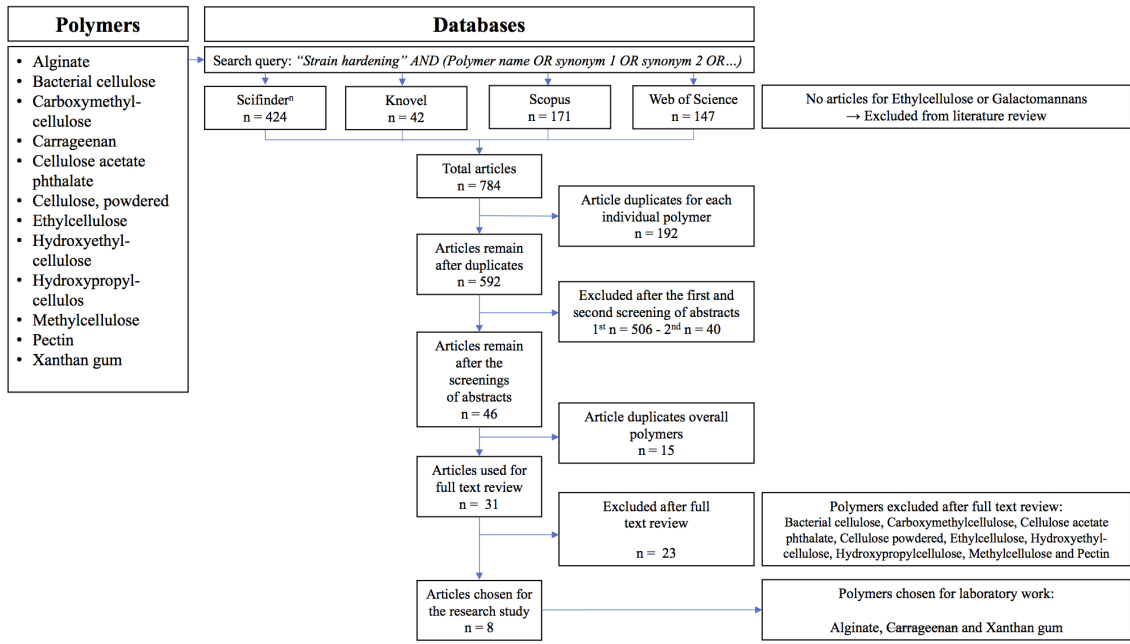


Figure A.1: A flow chart over the literature review that illustrates schematically how the number of articles were successively reduced.

B

Database Search

Table B.1: *An overview of the search results distributed over the databases from the literature review.*

Polymer	Date	Search phrase	Scifinder ⁿ	Knovel	Scopus	Web of Science
Alginate	Feb 10 th 2020	"strain hardening " AND alginate*	7	2	5	5
Bacterial cellulose	Feb 11 th 2020	"strain hardening" AND "bacterial cellulose"	2	0	1	2
Carboxymethyl-cellulose calcium	Feb 10 th 2020	"strain hardening" AND ("Carboxymethylcellulose Calcium" OR avicel OR "cellulose gel" OR crystalline-cellulose OR E460 OR emcocel OR fibrocel OR tabulose OR vivacel)	18	0	3	2

B. Database Search

Polymer	Date	Search phrase	Scifinder ⁿ	Knovel	Scopus	Web of Science
Carra-geenan	Feb 10 th 2020	"strain hardening " AND (carrageenan OR "chondrus extract" OR E407 OR gelcarin OR "irish moss extract" OR "SeaSpen PF" OR viscarin)	8	1	9	10
Cellulose acetate phthalate	Feb 11 th 2020	"strain hardening" AND ("Cellulose acetate phthalate" OR "acetyl phthalyl cellulose" OR aquateric OR CAP OR "cellulose acetate hydrogen" OR "1,2-benzenedicarboxylate" OR "cellulose acetate hydrogen phthalate" OR "cellulose acetate monophthalate")	132	0	75	51

B. Database Search

Polymer	Date	Search phrase	Scifinder ⁿ	Knovel	Scopus	Web of Science
Cellulose, powdered	Feb 10 th 2020	"strain hardening" AND ("cellulose powdered" OR cepo OR E460 OR elcema OR sanacel OR solka-floc)	19	0	0	0
Ethyl-cellulose	Feb 10 th 2020	"strain hardening" AND (ethylcellulose OR aquacoat OR E462 OR ethocel OR surelease)	0	0	0	0
Gallactomannans	Feb 10 th 2020	"strain hardening" AND (gallactomannans OR E412 OR "Guar flour" OR "jaguar gum")	0	0	0	0
Hydroxyethyl-cellulose	Feb 11 th 2020	"strain hardening" AND ("hydroxyethyl cellulose" OR alcoramnosan OR cellosize OR cellulose OR "hydroxyethyl ether" OR HEC OR idro-ramnosan OR liporamnosan OR natrosol)	36	17	35	33

B. Database Search

Polymer	Date	Search phrase	Scifinder ⁿ	Knovel	Scopus	Web of Science
Hydroxypropyl-cellulose	Feb 12 th 2020	"strain hardening" AND ("hydroxypropyl cellulose" OR cellulose OR "hydroxypropyl ether" OR E463 OR hyplose OR klucel OR methocel OR "nisso HPC" OR "oxypropylted cellulose")	39	15	34	32
Methyl-cellulose	Feb 10 th 2020	"strain hardening" AND (methylcellulose OR benecel OR celacol OR culminal MC OR E461 OR methocel OR metolose)	153	0	0	3
Pectin	Feb 11 th 2020	"strain hardening" AND pectin*	5	2	6	6
Xanthan gum	Feb 11 th 2020	"strain hardening" AND ("xanthan gum" OR "corn sugar gum" OR E415 OR Keltrol OR Merezan OR "polysaccharide B-1459" OR Rhodigel OR "xantham gum")	5	5	3	3

C

Calculations for Alginate Gel

Chemicals

- Alginate
 - 70% G-unit
 - 30% M-unit
- CaCO_3
 - $M_{\text{CaCO}_3} = 100.09 \text{ g/mol}$
- GDL
 - $M_{\text{GDL}} = 178.14 \text{ g/mol}$
- NaCl
 - $M_{\text{NaCl}} = 58.44 \text{ g/mol}$

Alginate solution

The alginate solution is 2%, thus 20 g alginate in 1 l of Milli-Q H_2O .

$$Alg_{sol} = alginate + \text{H}_2\text{O} + 0,1MNaCl \quad (\text{C.1})$$

$$\begin{aligned} m_{NaCl} &= M_{NaCl} \times V_{alg,sol} \times 0,1MNaCl \\ &= 58.44g/mol \times 1l \times 0,1mol/l \\ &= 5.844g \end{aligned} \quad (\text{C.2})$$

Calculation of [G-unit]

$$\text{wt}\%_{G-unit} = 70 \% = 0.7$$

$$M_{G-unit} = 193.132 \text{ g/mol}$$

$$\begin{aligned} [G - unit] &= \frac{c_{alg,in} \times \text{wt}\%_{G-unit}}{M_{G-unit}} \\ &= \frac{20g/l \times 0.7}{193.132g/mol} \\ &= 0.0725mol/l \end{aligned} \quad (\text{C.3})$$

The molar relation between $[Ca^{2+}]$ and $[G-unit]$

$$R = 2 \times \frac{[Ca^{2+}]}{[G-unit]} \quad (C.4)$$

$[Ca^{2+}]$ and $[GDL]$ for $R = 1$

$$\begin{aligned} [Ca^{2+}] &= \frac{R}{2} \times [G-unit] \\ &= \frac{1}{2} \times 0.0725 \text{ mol/l} \\ &= 0.0362 \text{ mol/l} \end{aligned} \quad (C.5)$$

$$\begin{aligned} [GDL] &= 2 \times [Ca^{2+}] \\ &= 2 \times 0.0362 \text{ mol/l} \\ &= 0.0725 \text{ mol/l} \end{aligned} \quad (C.6)$$

Masses of $[Ca^{2+}]$ and $[GDL]$ for $R = 1$

$$\begin{aligned} m_{Ca^{2+}} &= [Ca^{2+}] \times M_{CaCO_3} \times V_{alg,sol} \\ &= 0.0362 \text{ mol/l} \times 100.09 \text{ g/mol} \times 6 \times 10^{-3} \text{ l} \\ &= 0.02174 \text{ g} \end{aligned} \quad (C.7)$$

$$\begin{aligned} m_{GDL} &= [GDL] \times M_{GDL} \times V_{alg,sol} \\ &= 0.0725 \text{ mol/l} \times 178.14 \text{ g/mol} \times 6 \times 10^{-3} \text{ l} \\ &= 0.07749 \text{ g} \end{aligned} \quad (C.8)$$

Final volume of alginate gel

$$\begin{aligned} V_{fin} &= \frac{c_{alg,in} \times V_{alg,sol}}{c_{alg,fin}} \\ &= \frac{20 \text{ g/l} \times 6 \times 10^{-3}}{15 \text{ g/l}} \\ &= 0.008 \text{ l} = 8 \text{ ml} \end{aligned} \quad (C.9)$$

V_{H_2O} needed to dissolve $CaCO_3 + GDL$

$$\begin{aligned} V_{H_2O} &= V_{fin} - V_{alg,sol} \\ &= 8 \text{ ml} - 6 \text{ ml} \\ &= 2 \text{ ml} \end{aligned} \quad (C.10)$$

D

Calculations for Xanthan Gum and Locust Bean Gum Gel

Chemicals

- Xanthan gum (Xantural 180)
- LBG (GRINDSTED LBG 246)

Xanthan gum and LBG solution

The solution of xanthan gum and LBG is 1%, thus 10 g of polymers total in 1 l of Milli-Q H₂O. The xanthan gum/LBG solution is mixed in the ratio 1:1 of the polymers, that is 5 g of xanthan gum and 5 g of LBG, a total of 10 g.

$$XG/LBG_{sol} = XG + LBG + H_2O \quad (D.1)$$

E

Laboratory Lesson Plan

STUDSLERA I KLASSRUMMET OCH I SAMHÄLLET

Laboration för Kemi 1 på gymnasiet Lektionsplanering



Laboration framtagen av
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STUDSLERA I KLASSRUMMET OCH I SAMHÄLLET

Lärarhandledning

Syfte

Eleverna skall använda sina kunskaper om kemisk bindning för att förstå vad som händer när studsleran dras ut med olika hastighet. De skall även träna på att kemiskt resonera kring hur studslerans mekaniska egenskaper kan vara användbara i samhället och försöka göra egna verklighetskopplingar.

Laborationen är framtagen enligt teorier kring aktivt lärande och lektionsmomentet är planerat i enlighet med 5E modellen.

Lärandemål

Efter lektionen skall eleverna...

... ha resonerat kring kemisk bindning och bildat sig en uppfattning om att olika påverkan på ett material kan leda till olika respons i materialet.

... kunna redogöra för hur strain hardening kan vara användbart i samhället och ge exempel på material som beter sig på liknande sätt som studsleran gör.

Centralt innehåll (citerat från Skolverket, 2020)

- Kemisk bindning och dess inverkan på till exempel förekomst, egenskaper och användningsområden för organiska och oorganiska ämnen.
- Det experimentella arbetets betydelse för att testa, omvärdera och revidera hypoteser, teorier och modeller.
- Planering och genomförande av experiment samt formulering och prövning av hypoteser i samband med dessa.
- Utvärdering av resultat och slutsatser genom analys av metodval, arbetsprocess och felkällor.

Förmågor (citerat från Skolverket, 2020)

- Kunskaper om kemins begrepp, modeller, teorier och arbetsmetoder samt förståelse av hur dessa utvecklas.
- Förmåga att planera, genomföra, tolka och redovisa experiment och observationer samt förmåga att hantera kemikalier och utrustning.
- Kunskaper om kemins betydelse för individ och samhälle.
- Förmåga att använda kunskaper i kemi för att kommunicera samt för att granska och använda information.

Teori

Den engelska kommersiella versionen av studslera, Silly Putty, är ett material som består av flytande silikon, en typ av icke-Newtonska vätska, som ger studsleran ovanliga egenskaper. Dessa egenskaper kan beskrivas med hjälp av viskoelasticitet då studsleran beter sig som en viskös vätska under en längre period men som en elastisk solid under en kort tidsperiod, Wikipedia (2020). Det finns flera tester man kan göra på studslera för att testa dess viskoelastiska egenskaper, man kan, som namnet förtäljer, studsas den. Man kan också dra ut den långsamt så att den blir oändligt lång och tunn. Drar man istället väldigt hårt så går den mitt itu och lämnar ett rakt snitt. Ju mer man knådar leran, ju hårdare blir den och om man låter den ligga orörd under en längre period så flyter den ut till en pöl.

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Lektionsplaneringen är framtagen utefter teorier om aktivt lärande, vilket bygger på att eleverna ska engageras i sitt egna lärande. Laboration är ett lämpligt sätt att introducera aktivt lärande för att laboration som koncept kräver aktivitet från eleverna. Det är viktigt att tillåta eleverna att tänka själva och inte bara följa recept, därför är laborationens genomförande inte givet. Eleverna ska fundera själva först för att sedan presentera ett förslag på genomförande för dig, läraren, innan de får påbörja laborationen. Eftersom att materialet är givet så bör detta inte vara en större utmaning för eleverna men det tvingar dem att fundera över vad det är som de ska göra. Eleverna ska också diskutera om laborations olika koncept både sinsemellan med laborationspartner men även i helklass.

Den teori som specifikt tillämpades vid skapandet av lektionsplaneringen är framförallt 5E-modellen som beskrivs av Tanner (2010). Då 5E-modellen används vid lektionsplanering menar Tanner (2010) på att läraren skapar en atmosfär som bjuder in till lärande genom att aktivera elever och därmed rikta deras uppmärksamhet åt rätt håll. 5E-modellen består av fem olika delar: *Engage*, *Explore*, *Explain*, *Elaborate* och *Evaluate*, som beskrivs kort här nedan och förklaras sedan hur de ska uppnås i lektionsgenomförandet.

- Engage: Fånga elevernas intresse för att få deras uppmärksamhet.
- Explore: Hjälp eleverna att använda kunskap de redan har för att lösa en uppgift.
- Explain: Introducera nya koncept som eleven kan bygga på med sin tidigare kunskap
- Elaborate: Få eleverna att tillämpa sina nyförvärvade kunskaper.
- Evaluate: Tillåt eleverna att reflektera över lektionen och sitt lärande

Planeringens uppbyggnad överensstämmer även med Eilks och Hofsteins (2013) teorier kring kooperativt lärande som är en användbar metod av aktivt lärande i kemiundervisning. Kooperativt lärande bygger på att eleverna ska samtala med varandra och utbyta tankar och idéer. Vikten av elevdiskussion belyser även Smith et al. (2009) som menar på att lärarens förklaringar inte alltid är tillräckliga för att eleverna ska förstå materialet till fullo. Denna lektionsplanering bidrar till elevdiskussioner genom att tillåta eleverna att diskutera materialet sinsemellan innan frågeställningarna tas upp i helklass.

Filmer för mer inspiration

<https://www.youtube.com/watch?v=hoaCCWu9Av4>

<https://www.youtube.com/watch?v=BxP-G4LuQMo>

<https://www.youtube.com/watch?v=r0l6qnF-XHc>

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Lektionsgenomförande

Introduktion	Engage: Visa eleverna leran - studsa med den, dra ut den med olika hastigheter utan att förklara varför den beter sig som den gör. Det blir ett sätt att väcka elevernas intresse. Skriv upp några saker eleverna skall tänka på under labben. • Stöter vi på saker vi pratat om tidigare lektioner? Skriv upp! • Stöter vi på något som kan liknas vid saker i samhället? Ni vet ju att vi jobbar för att förstå vår verklighet! Hur kan dagens labb kopplas till verkligheten?
Laborativ del	Explore: Eleverna får tillverka leran och testa att dra ut den långsamt, snabbt och sedan göra en boll och studsa den och diskutera frågorna som finns i laborationshandledningen. Elaborate: Eleverna får svara på frågor och diskutera tillsammans • Vad händer? Kan eleverna förklara detta? Du som lärare har chans att gå runt och lyssna för att fånga upp elevernas tankar.
Diskussion	Explain: Diskussion i helklass: vad händer? Vad har eleverna kommit fram till? • Låt eleverna fundera på hur dessa egenskaper kan användas i samhället. • Verklighetskoppla på olika sätt. Presentera olika föremål, ex ryggskydd, och låt eleverna diskutera hur ett material med dessa egenskaper kan vara användbart för just den produkten. • Hur kan teorin i labben kopplas till teorin vi gått igenom? Peka på hur tidigare lektioner länkas ihop med labben för att det skall bli tydligt för eleverna att laborationen handlar om saker som de faktiskt redan kan.
Utvärdering	Evaluate: Ge eleverna en exit ticket där de får svara på tre korta frågor. • Varför kan leran dras ut långsamt men inte snabbt? • Vad känner du att du behöver träna mer på? • Nämn en mekanisk egenskap som leran har.

Riskanalys

När linsvätskan (som innehåller borsyra) och bikarbonat blandas sker en kemisk reaktion där borsyran blir till borax som har rapporterats som hälsofarlig. Det är ytterst små mängder av borax men det rekommenderas ändå att handskar används för att undvika onödiga hudirritationer som även limmet kan ge upphov till.

STUDSLERA I KLASSRUMMET OCH I SAMHÄLLET

Laborationshandledning

Syfte

Nu skall du använda dina kunskaper om kemisk bindning för att förstå vad som händer när studslera dras ut med olika hastighet.

Tänk på att använda begrepp du lärt dig på tidigare lektioner när du diskuterar under laborationen.

Material

- Lim - 1 dl
- Linsvätska - 2 tsk
- Bikarbonat - 0,5 tsk
- Karamellfärg - några droppar
- Bägare
- Spatel

OBS!
Glöm inte
skyddsglasögon,
labbrock och
handskar

Genomförande

Nu skall du tillverka studslera! Hur vill du göra?

- Skriv ett förslag på hur du skall genomföra laborationen
- Skriv också ner vilka tester du vill göra med leran när den är färdig

När du skrivit ner ditt förslag på hur du skall tillverka och testa leran visar du ditt förslag för din lärare och får det godkänt innan du får fortsätta

Diskutera med din laborationspartner

- Vad händer när du genomför dina tester?
 - Förklara både på makro- och molekylnivå
- Vilka mekaniska egenskaper har leran?
- Kan dessa mekaniska egenskaper vara användbara i verkligheten? I vilket sammanhang?