

Mechanical Characterization of the Effects of Paraffinic Oil on the Thermoplastic Elastomer, Styrene-Ethylene-Butylene-Styrene

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Abstract

Room temperature eutectic liquid metals, specifically the gallium-alloy gallium–indium–tin (also known as galinstan), are deformable conductive materials. Dispersed in a polymer composite, liquid metals have the potential to create materials for stretchable electronics with thermally and/or electrically useful behavior. The benefit of liquid metal dispersed into a polymer matrix over solid conductive fillers is that liquid metals, particularly as dielectric material for stretchable capacitors, have a reduced impact on bulk material stiffness. There are currently studies of the mechanical properties of galinstan dispersions in thermoset polymers in literature. For example, published results in polydimethylsiloxane (PDMS) have shown that galinstan can maintain its electrical properties with minimal alteration to the mechanical properties of the polymer matrix. Unfortunately, thermosets have limitations such as that the process of working with and the manufacturing of thermoset polymers is intensive and lengthy. Thermoplastic elastomers, however, are easier to process than thermoset polymers as they can be re-molded into a variety of desirable configurations at a temperature above their melting point and re-solidified upon cooling. This allows for materials that are more adaptable and cost-effective. In the work presented here, the mechanical properties of the thermoplastic elastomer styrene-ethylene-butylene-styrene (SEBS) were observed at varying concentrations of the SEBS solid swollen in paraffinic oil which resulted in varying bulk polymer moduli and elongation at break. It was found that the rigidity, elongation, and melting behavior of the SEBS elastomer could be tuned through the choice of paraffinic oil. The understanding of the mechanical behavior of the host polymer matrices is vital in determining their use for soft and stretchable electronics, for example in the creation of liquid metal dispersions, and will enable further analysis into the relationship and impact of dispersing galinstan into thermoplastic polymers.

Keywords: thermoplastic polymers, styrene-ethylene-butylene-styrene (SEBS), tensile behavior

1. Introduction

Deformable dielectric materials can be used in applications such as sensors, stretchable electronics (e.g. capacitors, inductors), and soft robotics¹. The usage of gallium-based liquid metal alloys, for example the gallium-indium-tin alloy galinstan, as deformable conductors has gained attention because they are simple to fabricate with, stretchable, and reconfigurable². While bulk galinstan is conductive, a dispersion of galinstan in a polymer is a non-conductive composite that is useful as a dielectric material. These resulting composites can withstand large deformations while maintaining their electrically useful behavior. However, when adding fillers, such as galinstan, into a host polymer matrix the overall rigidity is typically increased, which subsequently results in the decrease of the polymer's desirable soft and stretchable properties³. Therefore, it is crucial to understand the mechanical behavior of the host polymer matrix in order to maximize its desirable properties and enable further analysis into the impact of adding fillers like galinstan. This understanding of how the manipulation of the polymer matrix impacts the mechanical behavior of the

elastomer composite is critical when utilizing them for applications such as galinstan dispersions where the limiting factor for mechanical behavior is the polymer matrix.

Two major classifications of common polymers are thermosets and thermoplastics. Thermosets are polymers that once set into the solid-state through the process of curing cannot be reverted back to a liquid, moldable state. In contrast, thermoplastic elastomers, such as styrene-ethylene-butylene-styrene (SEBS), are solid at low temperatures and liquid at high temperatures which allows alterations for remolding and reuse. SEBS and other polymer gels have been studied for their ballistic capabilities under compressive stress^{4,5} and for pharmaceutical⁶, robotic⁷, and biomedical applications⁸. Not only do the two classifications of polymers have different mechanical capabilities, their chemistries are vastly different as well. The SEBS elastomer, a triblock copolymer of styrene, ethylene, and butylene, is made by swelling, or dissolving, the midblock of the SEBS solid polymer powder in oil, the swelling agent, but not the end blocks, which results in an elastomer. The oil, commonly mineral oil, used in the swelling of SEBS in this work is made up of paraffinic hydrocarbons that have varying chain lengths. Therefore, the viscosity of the paraffinic oil can vary significantly depending on the lengths of the alkane chains and potentially alter the SEBS elastomer causing changes that can be evaluated as a function of the average alkane chain length and resulting viscosity.⁹ The effects of varying the type of solvent used has been observed in systems such as SEBS and thermoreversible physical gels^{10,11,12}. The impact of altering the oil viscosity on the mechanical behavior of SEBS has been investigated previously by Kim et al.^{13,14}. However, the hydrocarbon oils evaluated in their work contained varying aromatic, naphthenic, and paraffinic concentrations. This is problematic as there is potential that those hydrocarbon oils are not only dissolving the midblock of SEBS but the end blocks as well. Through the use of a strictly paraffinic swelling agent, it was found in this study that the mechanical behavior of SEBS can be tuned resulting in a range of elastomer rigidity, elongation, and melting behavior. In addition, the percentage of oil to SEBS solid polymer greatly alters those mechanical behaviors. A higher percentage of oil than SEBS solid polymer leads to a softer elastomer while a higher percentage of SEBS solid polymer leads to a more rigid elastomer. By varying the percentages of oil and SEBS solid polymer, the material rigidity can be manipulated, and it has been previously observed to have an impact on the morphology in the phase behavior¹¹. However, the mechanical impact that the percentage of specifically paraffinic oil to SEBS solid polymer has on the rigidity and elongation has not been thoroughly investigated, even though it has the potential to allow the material's rigidity to be drastically altered to fit a variety of mechanical specifications. Therefore, when utilizing the SEBS thermoplastic characteristic, it is important to consider the viscosity of the paraffinic oil and the oil to SEBS solid polymer ratio.

The rigidity and elongation properties of the SEBS elastomer determine its potential usefulness. The rigidity of SEBS affects its resistance to deformation and changes its fluidic properties at varying temperatures while its elongation at break is indicative of its elasticity and stretchable capabilities. The rigidity of an elastomer, like SEBS, is measured in terms of its modulus and indicates the resistance to deformation that it can withstand when stress is applied. The lower the modulus of an elastomer the less resistance to deformation it is able to maintain thus causing it to be softer and/or more stretchable. The elongation of an elastomer at break indicates the materials ability to deform without mechanical failure and allows for evaluation of the materials elasticity. A longer elongation generally translates to a more stretchable and elastic material. This work focuses on how the manipulation of the swelling agent alters the resulting SEBS elastomer's rigidity and elongation properties. The mechanical behavior was characterized using tensile testing and rheology, and the melting behavior was evaluated using rheology. Altering both the rigidity and elongation properties of SEBS allows future work to be able to tune host polymer matrices to a specific rigidity and elongation before adding fillers. This will enable a deeper understanding of the SEBS elastomer that will aid in the development of the types of polymer matrices used in the field of stretchable electronics and soft robotics. Gaining a fundamental understanding through this work of SEBS and how the viscosity of the paraffinic oil used in the swelling of the SEBS polymer alters the desirable mechanical properties of the elastomer, as well as how the percentages of oil and SEBS solid polymer affect rigidity, will allow for the determination of the most desirable elastomer for use in applications such as galinstan dispersions for stretchable electronics and soft robotics.

2. Experimental

2.1. Materials

The styrene-ethylene-butylene-styrene (SEBS) solid polymer was donated from Kraton Polymers (Belpre, OH) as a solid powder. Light paraffin mineral oil was clear, of high purity grade purchased from VWR Chemicals, LLC (Solon, OH) and used as-received. Heavy paraffin mineral oil was purchased and used as-received from Spectrum Chemicals

(Gardena, CA). Hexadecane, 99% purity was from Fisher Scientific (Hampton, NH) and used as-received. Extra heavy paraffin mineral oil was made by mixing solid paraffin wax with the heavy paraffin mineral oil.

2.2. SEBS Elastomers Formulation

Four paraffinic oils of varying viscosities were used: hexadecane (8 cP), light paraffin mineral oil (29 cP), heavy paraffin mineral oil (116 cP), and extra heavy paraffin mineral oil (463 cP). SEBS powder and the oil were weighed out in the appropriate formulation ratios to make 10 and 20 vol% SEBS elastomers. As the SEBS elastomers used are oil-based, samples will be named for the amount of oil used. For example, a SEBS sample of 90 vol% oil and 10 vol% polymer will be named SEBS 90:10. The SEBS/oil formulations were put into an oven at roughly 120 °C for 2-5 hours and mixed at 30 minute intervals until all of the SEBS solid was dissolved obtaining a solution that was visually homogenous and absent of any visible undissolved powder. It was found that the higher the viscosity of the paraffinic oil and the higher the concentration of SEBS solid the more time was required to attain a completely dissolved homogenous solution. This resulting elastomer solution was then used for both rheological measurements and mechanical characterization.

2.3. Tensile Testing

Mechanical characterization was performed through tensile testing on a mechanical load frame to find the modulus and the elongation at break of the SEBS elastomer samples, thus determining the rigidity and percent elongation of the materials. The tensile samples were prepared by heating the previously prepared SEBS elastomers until they were roughly 120 °C. Each tensile sample was prepared and performed in triplicate. Then, the hot solution was poured into Teflon dog bone molds of the appropriate geometry for tensile testing that had been previously heated to the same temperature as the solution. The gauge length of the sample mold was 35 mm long x 5 mm wide x 5 mm thick. The samples were then allowed to cool to room temperature before being carefully removed from the molds directly before testing to avoid possible malformations. Tensile testing was performed using an Instron Series 5581 Mechanical Load Frame (Norwood, Massachusetts) with a crosshead speed of 5 mm/min and a 10N load cell. To ensure accuracy, the width and height of each sample was measured before testing, and the length of the tested gauge was measured once loaded on the load frame in order to ensure uniformity of testing between samples. The amount of pressure used to secure the samples in the grip was the same for each formulation of the sample to promote uniform failure in the gauge length and reduce the impact of slippage. Graphical representation of tensile testing data will abbreviate 'mineral oil' to 'MO'.

2.4. Rheology

Rheology of all four paraffinic oils was performed to determine their individual viscosities and temperature dependencies. Viscosity of the paraffinic oils was measured from 0.01% to 25% strain at 25 °C at 1 Hz and from 0.01 to 25 Hz at 25 °C at 1% strain. Additionally, viscosity was measured from 25 °C to 120 °C in order observe the behavior of the oils at higher temperatures that are used in the making of the SEBS elastomers. Since the oil composes 80 to 90 vol% of the SEBS elastomers, the behavior of the oils at higher temperatures is important to determine what part of the SEBS elastomers behavior at high temperatures is due to the oil and what is due to the SEBS. Rheological tests for each paraffinic oil were performed in triplicate.

Rheology of SEBS elastomers was used to determine the frequency, strain, and temperature dependence of the materials. All rheological measurements were performed using a Texas Instruments DHR-2 rheometer (New Castle, DE) that was equipped with a temperature-controlled oven. The SEBS elastomer samples were prepared by directly melting the sample onto a circular plate, 25 mm in diameter, at a gap height of 1 mm then cooling to room-temperature. Viscosity and modulus were measured from 26 °C to 120 °C at 0.1% strain at a heating step size of 5 °C per 120 seconds. Viscosity and modulus of the SEBS elastomer samples were measured from 0.01% to 25% strain, 1 Hz and 0.01 to 25 Hz, 0.1% strain at 26 °C and 120 °C to observe the mechanical difference between when the sample is expected to behave as a solid versus a liquid. The rheological tests for the SEBS elastomer with each paraffinic oil were performed in triplicate for both formulation ratios at 10 and 20 vol% SEBS solid to ensure accurate measurement.

3. Results and Discussion

3.1. Characterization of Modulus and Elongation

The rigidity of SEBS elastomers was characterized by measuring strain applied to the elastomer and the resulting stress, which was quantified in terms of a modulus. A comparison of the modulus values of SEBS elastomers taken at room-temperature as a function of oil concentration, specifically light mineral oil, is shown in Figure 1. The data demonstrates that the modulus of the elastomer greatly varies and is dependent upon the ratio of oil to SEBS solid.¹⁴ The lower the content of oil in SEBS elastomers the higher the modulus resulting in a more rigid elastomer. By varying the concentration of oil, the SEBS can be manipulated to result in a desired modulus. This allows the rigidity of the elastomer to be tuned with high specificity by adjusting the ratio of oil to SEBS solid. The ratios 90:10 and 80:20 of oil to SEBS solid were chosen for further evaluation in this work to determine the effects of manipulating the type of paraffinic oil in SEBS elastomers. This is because compared to the 70:30 and 60:40 ratios shown in Figure 1 they both had high sensitivity in tensile testing and are softer materials.

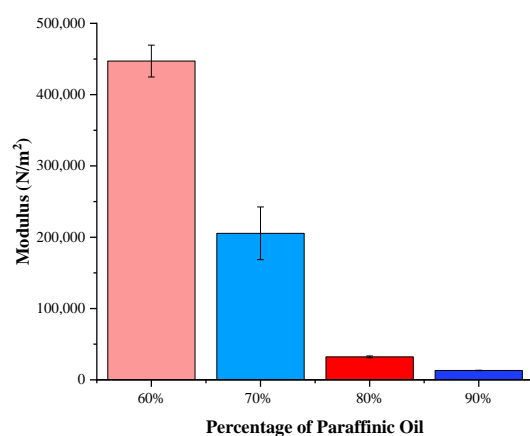


Figure 1. Effect of manipulating the paraffinic oil (light mineral oil) percentage in SEBS elastomers on the resultant modulus. Measurements performed at room-temperature.

The oils used to swell SEBS solid polymer were linear, paraffinic alkane chains. These paraffinic chains vary in chain length.^{9,11} The paraffinic oils were found to be largely temperature independent and have viscosities of 8 cP for hexadecane, 29 cP for light paraffin mineral oil, 116 cP for heavy paraffin mineral oil, and 463 cP for extra heavy paraffin mineral oil. As demonstrated by the rheology of the paraffinic oils used, the longer the average alkane chain length the higher the viscosity of the oil. The varying viscosities of these oils were found to have a significant impact upon the modulus. Figure 2a and 2b shows the modulus of SEBS elastomer samples with hexadecane, light mineral oil, heavy mineral oil, and extra heavy mineral oil used. Hexadecane, having the lowest viscosity (8 cP), resulted in the SEBS elastomer with the lowest bulk modulus compared to that of the other oils for both SEBS 90:10 and 80:20 elastomers. As the viscosity of the paraffinic oils is increased, the modulus of the resulting SEBS elastomers increases.

In SEBS 90:10 elastomers, the modulus for extra heavy mineral oil, as noted on Figure 2a with a blue asterisk, was not measured. The extra heavy mineral oil elastomer for SEBS 90:10 was highly brittle and thus couldn't be measured for modulus and elongation at break. However, the SEBS 80:20 elastomer for extra heavy mineral oil was able to be tested. Figure 2b shows that the modulus for SEBS 80:20 extra heavy mineral oil was the highest out of the four paraffinic oil SEBS 80:20 elastomers. Extra heavy mineral oil was measured as having the highest viscosity (463 cP) and thus, it results in elastomers with the highest modulus fits within literature trends.¹³ Therefore, it is predicted that if testing the SEBS 90:10 elastomer with extra heavy mineral oil was feasible it would have the highest modulus out of the four paraffinic oil SEBS 90:10 elastomers.

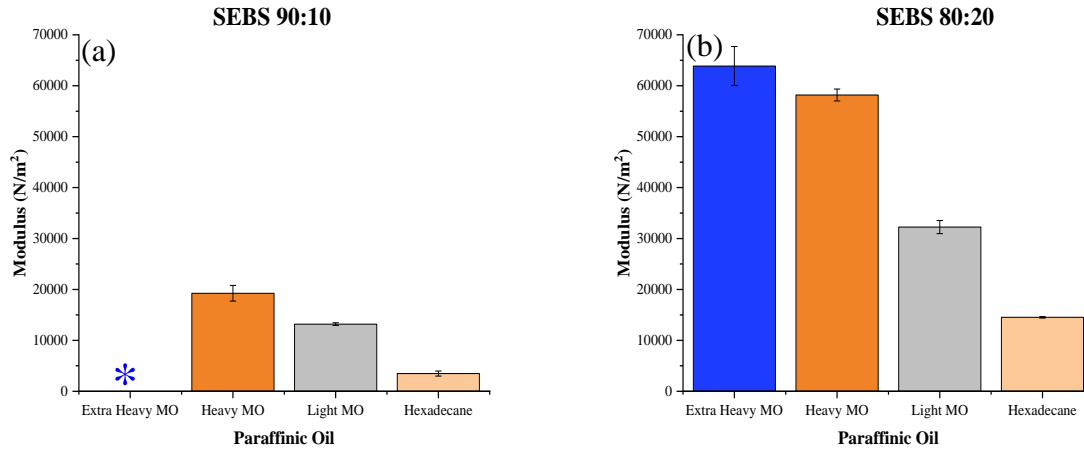


Figure 2. Modulus impact of the different paraffinic oils' hexadecane, light mineral oil, heavy mineral oil, and extra heavy mineral oil in both SEBS elastomer ratios of (a) 90:10 and (b) 80:20 oil to SEBS solid.

Measurements of the elongation required to break the SEBS elastomer samples of each of the four paraffinic oils reflects the elasticity and stretchability of the elastomers. The larger the elongation at break the more stretchable the resultant elastomer is. Figure 3a shows that as the viscosity of the paraffinic oil used in SEBS 90:10 elastomers decreases the elongation at break increases. The lowest viscosity paraffinic oil, hexadecane, has the longest elongation at break (282%) and thus the highest elasticity. A difference in breaking behavior was also noted. When the hexadecane SEBS elastomer for both SEBS 90:10 and 80:20 elastomers was elongated to the breaking point instead of slowly tearing, which is the typical behavior of the rest of the SEBS elastomers tested, the elastomer gauge length failed all at once in one "clean" break. The breaking of the hexadecane SEBS elastomer in this way suggests that this elastomer is better able to distribute deformation changes throughout the gauge length rather than having weak points of stress that cause a slow tear such as in the other swelling agent SEBS elastomers. It is thus hypothesized that if measured the extra heavy mineral oil SEBS 90:10 would have the smallest elongation at break. This agrees with the previous observation that SEBS 90:10 is highly brittle. This may be because SEBS 90:10 elastomers are more influenced by the oil than SEBS 80:20 elastomers due to having a higher concentration of oil. From the SEBS 80:20 elastomers elongation at break data (Figure 3b), there is no measured dependence upon the viscosity of the paraffinic oil. Further investigation is needed to determine whether there is an oil percentage threshold above which the oil viscosity no longer has significant impact upon the elongation at break.

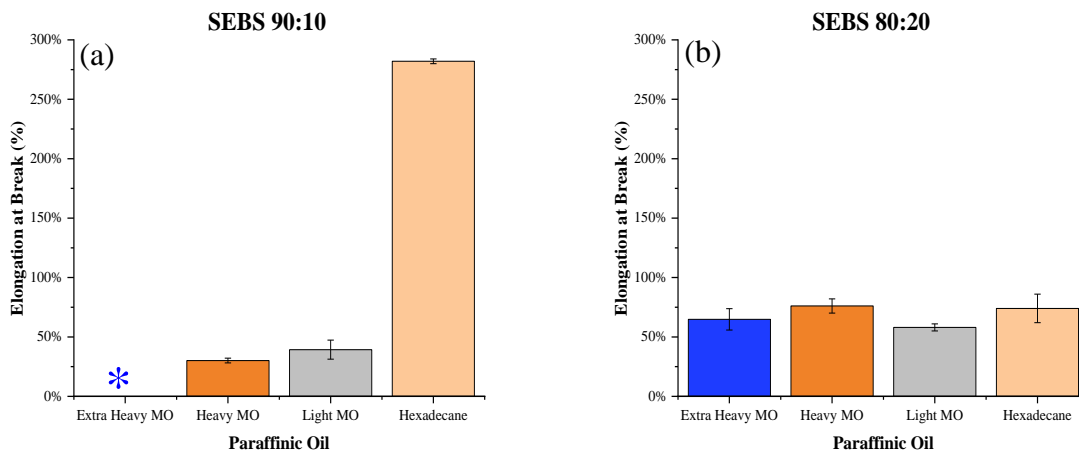


Figure 3. Elongation at break impact of the different paraffinic oils' hexadecane, light mineral oil, heavy mineral oil, and extra heavy mineral oil in both SEBS elastomer ratios of (a) 90:10 and (b) 80:20 oil to SEBS solid.

3.2. Rheology and Melting Behavior

The storage and loss modulus of the SEBS elastomers, shown in Figures 5 and 6, were measured as a function of temperature and paraffinic oil. The modulus from the rheological measurements for the SEBS elastomers at 26 °C generally concurred with the tensile testing data that as the viscosity of the oil in the SEBS elastomer increases the modulus also increases. As the temperature is increased, the SEBS elastomers melt and change from a solid to a liquid. The temperature at which this transition occurs is indicated by the intersection of the storage and loss modulus curves shown in Figures 5 and 6. Since the paraffinic oils were found to be temperature independent within the range measured, the observed melting behavior of the SEBS elastomer formulations were not due to the oils themselves. As the temperature is increased from 26 °C to 120 °C, the loss and storage modulus of the SEBS elastomers decreases. For example, the storage modulus for SEBS 90:10 with extra heavy mineral oil decreases from 42 kPa to 1 Pa as the temperature increases from 26 °C to 120 °C. This reflects the thermoplastic behavior of SEBS elastomers. Figure 5 displays the melting behavior of SEBS 90:10. The SEBS 90:10 elastomer with the highest viscosity of all the paraffinic oils used, extra heavy mineral oil, was found to begin to melt at 64 °C (Figure 4). Decreasing the viscosity of the oils decreased the temperature at which the SEBS 90:10 elastomers began to melt. This is seen as the heavy mineral oil elastomer began melting at 61 °C, the light mineral oil elastomer melts at 48 °C, and the hexadecane elastomer at 40.3 °C. These transitions are shown in Figure 4. Additionally, for both SEBS 90:10 and 80:20, as the viscosity of the oil decreases the slope of the melting behavior at low temperatures become steeper (Figures 5 and 6). This indicates that the SEBS elastomers require less heat to melt as well as it allows them to melt at lower temperatures as the oil viscosity decreases.

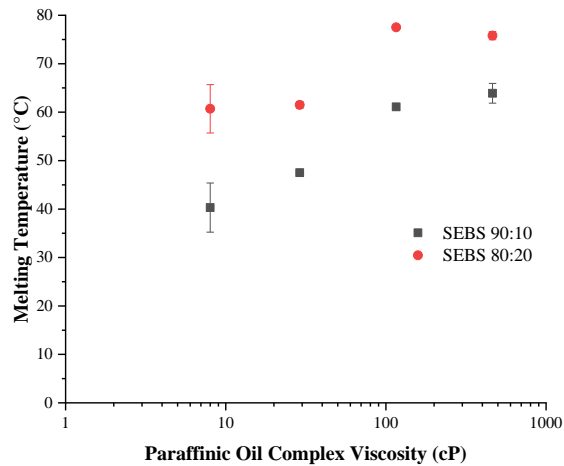


Figure 4. Melting temperatures of the oils with varying viscosities used in SEBS 90:10 and 80:20.

SEBS 90:10

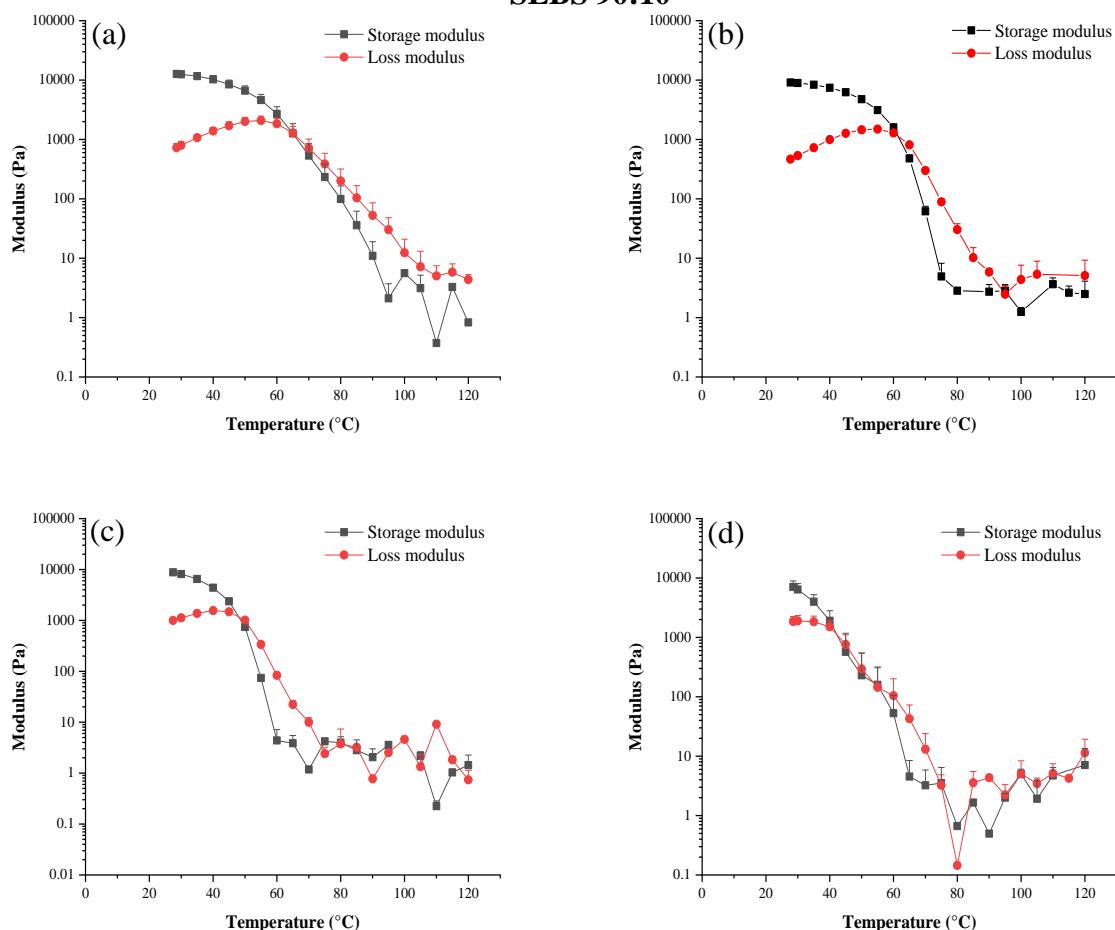


Figure 5. Melting behavior dependence of SEBS elastomers that have an oil to SEBS solid ratio of 90:10 on the various paraffinic oils used: (a) extra heavy mineral oil, (b) heavy mineral oil, (c) light mineral oil, and (d) hexadecane. For all parts, only the positive error bars are shown for clarity. The crossing of the storage and loss modulus indicate the temperature at which the SEBS elastomers change from a solid to a liquid. Data taken at 0.1% strain, 1 Hz. Modulus measurements at the higher temperatures reached the sensitivity limits of the rheometer.

Viscosity of the paraffinic oil had a significant impact on the melting behavior of the SEBS 90:10 elastomers. The melting behavior of the elastomers is also expected to be dependent upon the volume concentration of the oil used.^{13,14} As the volume of oil used decreased, from 90 to 80 vol%, the melting temperature of each respective elastomer increased (Figure 4). This could be because the SEBS solid has a high melting temperature at roughly 370 °C, and a glass transition temperature, T_g , of 82 °C.^{15,16} By adding more oil, which is liquid, the temperature at which the resulting SEBS elastomer melts decreases. Therefore, by adding less oil to the SEBS solid the bulk melting temperature increases. Figure 6 displays the melting behavior of SEBS 80:20. The SEBS 80:20 elastomers using heavy mineral oil, light mineral oil, and hexadecane all followed the same trend as the SEBS 90:10 elastomers in having decreasing melting temperatures of 78 °C, 62 °C, and 61 °C, respectively, as the viscosity of the paraffinic oil decreased (Figure 4). However, the extra heavy mineral oil used in the SEBS 80:20 elastomers was an outlier to the trend, having a lower melting temperature than heavy mineral oil (76°C, Figure 4). Future work will investigate the mechanism by which alkane lengths of the oil within SEBS elastomer ratios directly impact and their melting behavior.

SEBS 80:20

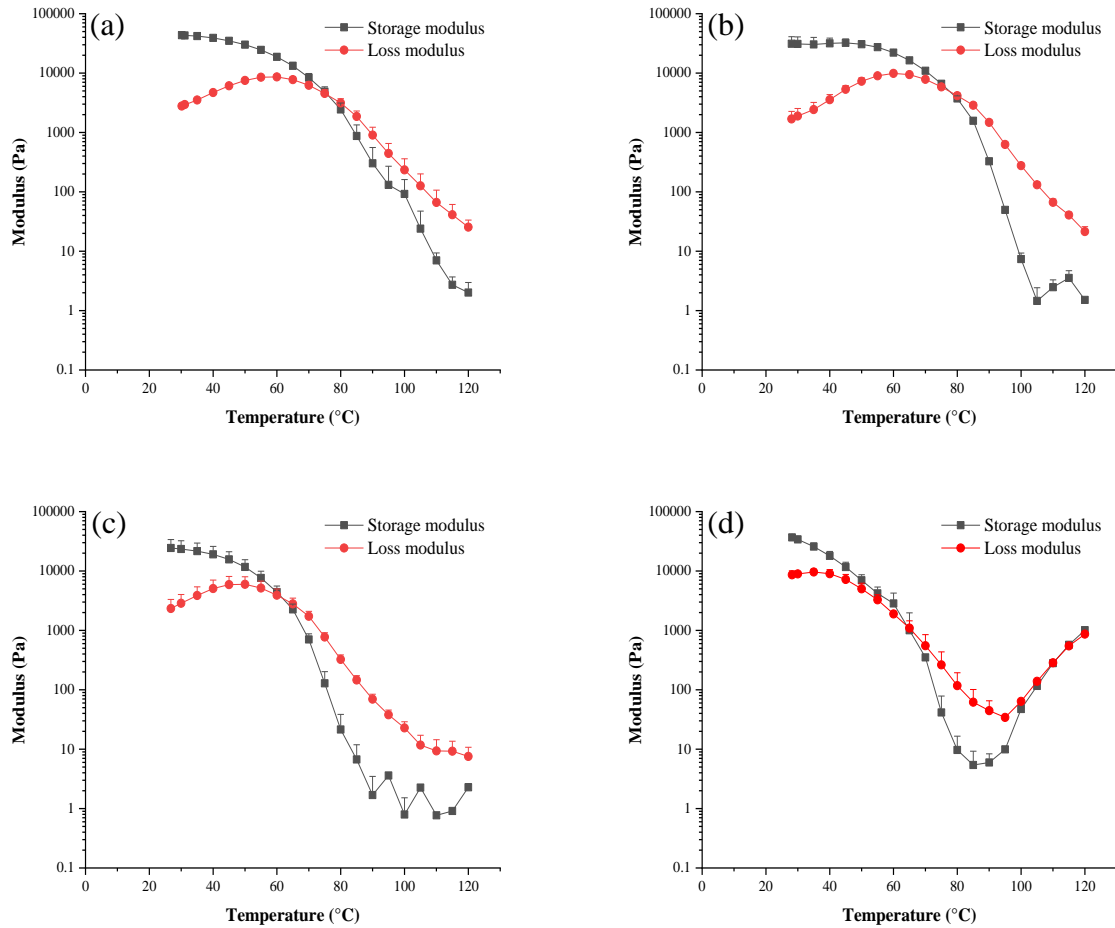


Figure 6. Melting behavior dependence of SEBS elastomers that have an oil to SEBS solid ratio of 80:20 on the various paraffinic oils used: (a) extra heavy mineral oil, (b) heavy mineral oil, (c) light mineral oil, and (d) hexadecane. For all parts, only the positive error bars are shown for clarity. The crossing of the storage and loss modulus indicate the temperature at which the SEBS elastomers change from a solid to a liquid. Data taken at 0.1% strain, 1 Hz.

4. Conclusions

Thermoplastic elastomers, such as styrene-ethylene-butylene-styrene (SEBS), can be remolded and reused due to their ability to be solid at low temperatures and liquid at high temperatures. This advantage allows them to be used for materials such as deformable dielectric materials that are useful for sensors, soft robotics, and stretchable electronics. Due to this, it is vital to have an understanding of the mechanical behavior of SEBS in order to fully utilize its desirable properties and enable further analysis into its potential applications. The oil used in the swelling of SEBS to result in the elastomer are paraffinic hydrocarbons, which have varying chain lengths. This leads to variable viscosity of the paraffinic oil that is dependent on the alkane chain length. These chain lengths lead to mechanical behavioral changes such as the resultant rigidity, elongation, and melting behavior of the SEBS elastomer. Additionally, by varying the percentage of oil to SEBS solid polymer the same mechanical properties can be tuned. The SEBS elastomer rigidity and elongation affects its resistance to deformation, thermoplastic properties, elasticity, and stretchable behavior.

In this work, the relationship between SEBS concentration, swelling agent, mechanical properties, and melting point was determined. Through the manipulation of the viscosity (8-463 cP) and concentration of the paraffinic oil (80 and 90 vol%), the mechanical behavior of SEBS elastomers can be tuned in terms of rigidity, elongation, and melting behavior. The tunability of SEBS is valuable because by tuning to a softer elastomer allows the SEBS elastomer to be

used for applications such as for soft robotics and stretchable electronics¹, where a softer material is preferred. On the contrary, by having the ability to tune the SEBS elastomer to a higher rigidity, it can be useful for applications such as the development of new ballistic mediums⁵, where a more rigid material is preferred. SEBS was designed in this work to have a modulus as low as 3.5 kPa and as high as 447 kPa with elongation at break from 30% - 282%. Furthermore, the mechanical behavior of SEBS elastomers is dependent upon the viscosity of the paraffinic oil used to swell the SEBS solid. As such, future work will focus on the effects that utilizing shorter paraffinic chain lengths has on creating a softer elastomer. In addition, future work will examine the effect that using isolated (i.e. single molecular weight) paraffinic chains smaller than the hexadecane used in this work has on the elasticity and softness of the resultant SEBS elastomer. It was found that as the viscosity of the paraffinic oil used in swelling is increased for SEBS 90:10, the modulus increases while the elongation at break decreases. Similarly, as the viscosity of the paraffinic oil used in swelling is increased for SEBS 80:20 the modulus increases, however, there is no correlation in the elongation at break decreases. Future work is needed to determine that if the oil percentage is above a specific threshold then the oil viscosity no longer causes significant impact upon the elongation at break and the mechanism that determines this threshold. By decreasing the concentration of the oil from 90 to 80 vol%, it was found that the modulus and the rigidity of the resultant SEBS elastomer increased by roughly 200-400%. Further investigation into lower concentrations of oil is needed to gain a complete understanding of how the concentration of SEBS solid affects the mechanical behavior of SEBS elastomers. With respect to thermal properties, as oil viscosity increases, the melting temperature of the SEBS elastomers increases. For example, in SEBS 90:10 the melting temperature using the lowest viscosity oil, hexadecane, was 40 °C while the melting temperature using the highest viscosity oil, extra heavy mineral oil, was 64 °C. Further investigation is needed to focus on the specific relationship that the melting behavior within SEBS elastomer ratios has with the alkane lengths of the oil. Overall, it was found that increasing oil concentration from 80 to 90 vol% decreases the melting temperature by up to 34%.

By altering both the formulation parameters of SEBS, future work will be able to tune the host polymer matrix to a specific rigidity and elongation before adding fillers, such as galinstan. This will allow for a fundamental understanding of SEBS elastomers and the effects that the viscosity and concentration of the mineral oil used in the swelling of the SEBS polymer has on the desirable mechanical properties for future use for soft robotic and stretchable electronic applications.

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