

Original article

Evaluation of viscoelastic properties, hardness, and glass transition temperature of soft denture liners and tissue conditioner

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Abstract

Soft denture liners and tissue conditioners are widely used for the denture patients to cushion masticatory force and condition abused tissues, respectively. This study assessed methods for the evaluation of the viscoelasticity and glass transition temperature (T_g) of the silicone permanent soft liner, acrylic permanent soft liner, and tissue conditioner. Three rheological parameters of storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$), T_g , and hardness were determined using dynamic mechanical analysis (DMA), differential scanning calorimetry (DSC), and the Shore A0 hardness test. Five specimens were measured for each material. The time-temperature superposition principle was applied to produce master curves of E' , E'' , and $\tan \delta$ for the tested materials at a reference temperature of 37 °C. The acrylic permanent soft liner and tissue conditioner exhibited viscoelastic behavior and sensitivity to frequency, especially at lower frequencies. The silicone permanent soft liner showed elastic behavior and was frequency-independent. T_g for the acrylic permanent soft liner was higher than that for the tissue conditioner, which in turn was higher than that for the silicone permanent soft liner for both DMA and DSC. In DMA, a higher frequency led to higher T_g values. A positive linear relationship was found between Shore A0 hardness and E' values, but not E'' and $\tan \delta$ values. Shore hardness reflects elasticity, but not viscosity. The results of the present study can be used to improve methods for evaluating the viscoelasticity and T_g of soft denture liners and tissue conditioners.

Introduction

The number of elderly people who wear dentures has increased as society has aged [1]. Patients with high ridge resorption have become increasingly common, and the stability and retention of the dentures are influenced by progressive resorption of the residual ridge [2]. Even if a well-fitting denture is worn, the adaptation between the intaglio surface of the denture and the denture-bearing area can decrease due to systemic diseases, such as diabetes, that promote the absorption of the jaw bone and long-term denture use [3]. In addition, the use of an ill-fitting denture can cause distortion and denture ulcers in the denture-bearing area. To resolve these problems, tissue conditioning and reline are necessary in a clinical situation. Tissue conditioners are applied to recover the distortion of the denture-bearing area to normal, take dynamic impression and reline the dentures temporarily [4-6], as the viscoelasticity of the tissue conditioner enables the functional stresses on the denture-bearing area to be uniformly distributed [6]. Reline is used to resurface the intaglio surface of a denture with denture liners. There are two types of denture liner, namely hard and soft [7]. Hard denture liners are used to improve the adaptation of ill-fitting dentures [7]. Soft liners are used to reduce the functional forces transmitted to the denture-bearing area, by means of their cushioning effect, for denture wearers who complain of masticatory pain due to severe alveolar resorption, thin and non-resilient oral mucosa, or both [7 - 9].

The clinical efficacy of soft denture liners and tissue conditioners depends on the viscoelastic properties of their materials [10]. Penetration tests [11,12], static tests such as creep tests and stress relaxation tests

[6,13], and dynamic tests such as the non-resonance forced vibration method [10] have been employed for viscoelastic analysis. Soft denture liners and tissue conditioners are subjected to dynamic forces such as mastication in the oral environment. The evaluation of the dynamic viscoelastic properties is thus considered to be the most clinically useful [10,14,15]. Such an evaluation can be conducted using dynamic mechanical analysis (DMA). DMA measures changes in the viscoelastic properties with the cyclic application of stress at various temperatures [16]. Three rheological parameters, namely the storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$), are determined in this method. Furthermore, the time-temperature superposition principle can be applied to extract the viscoelastic properties of polymeric materials over a wide time scale though there are some exceptions [16]. However, this principle has been rarely applied to dental materials.

In addition, the analysis of the glass transition temperature (T_g), at which polymeric materials generally exhibit a large variation in mechanical properties (from hard/brittle to soft/flexible), is important for the rigorous evaluation of materials. Physical properties such as viscoelastic properties rapidly change at T_g . Polymeric materials lie in the glassy region below T_g , in the transition region at around T_g , and in the rubbery region and then flow region above T_g [17]. In DMA, T_g can be calculated by detecting the temperature corresponding to the maximum $\tan \delta$ or the E'' peak position [18]. Differential scanning calorimetry (DSC) measures the changes in temperature and heat flow related to the thermal transition of materials. It has been used to evaluate phase transition phenomena such as crystallization, glass transition,

and melting [19]. The thermal properties, including T_g , of materials used for dentures have been previously evaluated using DMA [20 - 25], and DSC [22, 26, 27]. A study on autopolymerized hard direct denture relined resins found that the T_g values obtained using DMA were higher than those obtained using DSC [28]. Although the employed method influences the determined T_g values of dental materials, there is little information regarding this issue for soft denture liners and tissue conditioners.

The hardness requirements for soft denture liners and tissue conditioners are specified in ISO 10139 [29, 30]. This simple test uses a durometer to evaluate the hardness of materials. However, there is no information on the relationship between the values obtained from a Shore A0 hardness test and the viscoelastic properties, which are important for the clinical efficacy of soft denture liners and tissue conditioners.

In this study, we assess methods for the evaluation of the dynamic viscoelasticity of soft denture liners and tissue conditioners. The time-temperature superposition principle is applied, T_g is measured, and the relationship between Shore A0 hardness and viscoelastic parameters is derived. It is hypothesized that the time-temperature superposition principle can be applied for the evaluation of soft denture liners and tissue conditioners. We also speculate that there will be differences in the T_g values obtained using DMA and DSC and that there will be a correlation between Shore A0 hardness and E' values.

Materials and methods

The materials used in the present study are listed in Table 1. GC RELINE II (Soft) and BIO LINER were selected as the permanent soft denture liners. These liners are made of silicone and acrylic, respectively. SOFT-LINER was selected as the tissue conditioner. It is an acrylic temporary soft liner.

Dynamic mechanical analysis (DMA)

The dynamic viscoelastic properties of the test materials were evaluated using an automatic dynamic viscoelastometer (Rheovibron DDV-25FP, A&D Co. Ltd., Tokyo, Japan). This device is based on the principle of non-resonance force vibration [10]. Five specimens of each material were prepared in the form of rectangular blocks (30 mm × 6.0 mm × 2.0 mm) using a metal mold according to the manufacturer's instructions. The test was conducted 1 hour after the preparation of specimens at 0.1, 0.2, 1, 5, and 10 Hz over a temperature range of -150 °C to 200 °C. Measurements were performed 5 times for each material. The ends of the specimens were clamped over a span of 15 mm and a strain of 0.05% was applied [28]. The rheological parameters, namely the complex dynamic tensile modulus (E^*), tensile storage modulus (E'), tensile loss modulus (E''), and loss tangent ($\tan \delta$), are respectively defined as follows [10,16]:

$$E^* = E' + iE''$$

$$E' = |E^*| \cos \delta$$

$$E'' = |E^*| \sin \delta$$

$$\tan \delta = E'' / E'$$

where i is $\sqrt{-1}$ and δ is the phase angle between the stress and the strain.

The glass transition temperature (T_g) is defined as the temperature corresponding to the maximum of the E'' peak position [17,18].

The master curves for E' , E'' , and $\tan \delta$ for each material at a reference temperature of 37 °C were produced based on the time-temperature superposition principle [17,31,32]. The curves for E' , E'' , and $\tan \delta$ at frequencies of 0.1, 0.2, 1, 5, and 10 Hz in the temperature range of -150 °C to 200 °C were superimposed on the respective curves obtained at 37 °C via horizontal shifts along the logarithmic frequency or time scale using the Williams-Landel-Ferry (WLF) method [16,17]. The analytical processing software for rheology measurement data Poly Dynamic Swing IRIS (Iris Development LLC, Amherst, MA, USA) was used for the analysis.

Differential scanning calorimetry (DSC)

DSC converts the difference in temperature between a reference material and specimens into a moving quantity of heat per unit time by changing the temperature of both substances according to a given program. DSC can be used to evaluate phase transition phenomena such as the glass transition, crystallization, and melting of polymeric materials. DSC was carried out using a differential scanning

calorimeter (DSC-60, Shimadzu Corp., Kyoto, Japan) to measure T_g . Five specimens were prepared in accordance with the manufacturer's instructions for each material. Each specimen was placed into an aluminum pan. The test was conducted 1 hour after the preparation of specimens under a nitrogen purge with a flow rate of 50 mL/min [28]. The scan speed for thermal heating was 10 °C/min and the temperature range was -150 °C to 100 °C. The heat flow for each material was plotted against the temperature (time) to obtain the DSC curve. Measurements were performed 5 times for each material. The T_g values for the materials were determined according to JIS K7121 [33].

Shore A0 hardness test

Specimens were placed into a metal mold cavity (diameter: 50 mm; thickness: 8 ± 0.5 mm) according to ISO 10139-1 [30]. 15 minutes after mixing, the specimens were immersed into a water bath at 37 °C. They were removed from the mold 1 hour after mixing. Five points were measured for each specimen using a type E durometer (GS-721G, Teclock, Nagano, Japan) with a stand (GS-615, Teclock, Nagano, Japan).

Statistical analysis

The T_g values obtained from DMA and DSC, the three rheological parameters, and Shore A0 hardness values for the three tested materials were subjected to a one-way analysis of variance (ANOVA). The

mean values were compared using Tukey's method at a 5% level of significance. Regression analyses were conducted to determine the correlation between the three rheological parameters and Shore A0 hardness. For all statistical analyses, the statistical analysis software SPSS Statistics version 17.0 (SPSS Inc., Chicago, IL, USA) was used.

Results

Figures 1-3 show the temperature dependence of the storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$) for the three tested materials, respectively, at various frequencies. No large differences were found in the positions of the curves for E' , E'' , and $\tan \delta$ for GC RELINE II among the frequencies. At approximately -50 °C, E' and E'' for GC RELINE II rapidly decreased and $\tan \delta$ exhibited a sharp peak. Furthermore, the peak for E'' (*i.e.*, the glass transition temperature, T_g) appeared at approximately -125 °C. A small peak for $\tan \delta$ also appeared at approximately -125 °C (Fig. 1). The T_g values for GC RELINE II changed from -127.5 ± 1.0 °C to -120.1 ± 2.9 °C when the frequency was increased from 0.1 to 10 Hz (Table 2). BIO LINER and SOFT-LINER exhibited almost the same tendencies in terms of the temperature dependence of E' , E'' , and $\tan \delta$. The curves for E' , E'' , and $\tan \delta$ significantly shifted to the right with increasing frequency. E' and E'' values for BIO LINER gradually decreased from approximately 0 °C. Small peaks in the E'' curves appeared at approximately -20 °C. Large peaks in $\tan \delta$

appeared between approximately 10 °C and 40 °C in the frequency range tested (Fig. 2). The T_g values for BIO LINER changed from -27.3 ± 9.4 °C to -9.6 ± 5.4 °C when the frequency was increased from 0.1 to 10 Hz (Table 2). E' and E'' values for SOFT-LINER gradually decreased from approximately -40 °C. Small peaks in the E'' curves appeared at approximately -50 °C. Large peaks in $\tan \delta$ appeared between approximately -20 °C and 0 °C (Figs. 3). The T_g values for SOFT-LINER changed from -61.3 ± 5.0 °C to -44.3 ± 9.2 °C (Table 2).

Figure 4 shows the E' , E'' , and $\tan \delta$ values for the three materials obtained at 1 Hz and 37 °C. The values at 1 Hz are clinically important because this frequency simulates the masticatory rhythm. Significant differences were found among the E' values for the materials ($p < 0.05$); the order was GC RELINE II > BIO LINER > SOFT-LINER. BIO LINER had higher E'' values than those for the other two materials; there was no significant difference in the E'' values between GC RELINE II and SOFT-LINER. The values of $\tan \delta$ for BIO LINER were higher than those for SOFT-LINER, which in turn were higher than those for GC RELINE II ($p < 0.05$).

Figure 5 shows an example of the relationship between frequency and a rheological parameter for various temperatures. The curves shifted toward the curve obtained at the reference temperature (37 °C) one by one along the log frequency scale until they were superposed. Curves obtained at temperatures above 37 °C shifted to the left, and those obtained at below 37 °C shifted to the right. The master curves for E' , E'' , and $\tan \delta$ at 37 °C for the tested materials are shown in Fig. 6. The master curves for GC

RELINER II, BIO LINER, and SOFT-LINER cover the frequency ranges of 10^{-5} to 10^{16} , 10^{-2} to 10^{21} , and 10^{-1} to 10^{17} rad/s, respectively. The changes in the three rheological parameters for GC RELINER II with frequency were not larger compared with those for the acrylic materials (BIO LINER and SOFT-LINER). The E' and E'' values for the acrylic materials increased rapidly in the frequency range of approximately 10^{-2} to 10^4 rad/s with increasing frequency; thereafter, E' increased gradually and E'' decreased gradually. The $\tan \delta$ values for the two acrylic materials increased with increasing frequency, peaked at approximately 10^2 to 10^3 rad/s, and then decreased.

Significant differences in T_g values obtained using DSC (differential scanning calorimetry) were found among the materials. The value of T_g for GC RELINER II was lower than that for SOFT-LINER, which in turn was lower than that for BIO LINER ($p < 0.05$) (Table 2). The T_g values obtained using DSC were within those obtained using DMA (dynamic mechanical analysis).

The Shore A0 hardness for the three materials is shown in Figure 7. GC RELINER II had a higher Shore A0 hardness than that for BIO LINER, which in turn was higher than that for SOFT-LINER ($p < 0.05$). Figure 8 shows the correlation between the three rheological parameters and the Shore A0 hardness for the three materials. A positive linear relationship was found between E' values and Shore A0 hardness ($R=0.854$, $F=35.076$, $p < 0.0005$). There was no significant correlation between the other two rheological parameters and the Shore A0 hardness.

Discussion

The present findings confirm our hypotheses that the time-temperature superposition principle could be applied for the analysis of the dynamic viscoelastic properties of soft denture liners and tissue conditioners, and that there would be differences in viscoelasticity among the materials. In addition, it was found that the glass transition temperature (T_g) values for the acrylic permanent soft liner and tissue conditioner vary between DMA (dynamic mechanical analysis) and DSC (differential scanning calorimetry) even though the T_g values for the silicone permanent soft liner obtained using these two methods were almost the same. We also found a positive linear relationship between Shore A0 hardness and the storage modulus (E') for the tested materials.

In the present study, DSC was selected to measure T_g values because it is simple and the endothermic and exothermic reactions of the tested materials generally occur at the glass transition temperature [19, 34]. DMA was also used to determine the viscoelastic properties, namely storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$), which are important for the clinical evaluation of materials.

A soft denture liner and a tissue conditioner applied to the intaglio surface of a denture are subjected to instantaneous stress such as mastication. This is reflected by the behavior at 1 Hz in DMA. The viscoelastic properties at 1 Hz in the temperature range of approximately 0 °C to 60 °C, and especially at 37 °C, are important for the evaluation of the clinical behavior of materials [14]. Large differences in the

dynamic viscoelasticity were found among the tested materials. E' and E'' represent the elastic and viscous components of a material, respectively. Higher E' and E'' at 1Hz would indicate a greater ability of the dentures to crush food instantaneously [14]. $\tan \delta$ is the ratio of the elastic and viscous components, and thus shows their relative contributions. The damping that results from a higher value of $\tan \delta$ is likely to produce a degree of stress relief [14]. The acrylic permanent soft liner had higher $\tan \delta$ values than those for the other two materials at 1 Hz and 37 °C. The E' values for the silicone and acrylic permanent soft liners were higher than those for the tissue conditioner. A previous study found that the application of a soft denture liner with relatively high values of $\tan \delta$ and E' to a denture improved masticatory function [14]. Therefore, the improvement will be greater with BIO LINER, which is an acrylic permanent soft liner, than with the other 2 materials. However, it has been reported that the durability of acrylic permanent soft liners is lower than that of silicone permanent soft liners due to the loss of the low-molecular-weight plasticizer and water absorption [10, 35, 36]. It was also found that acrylic materials are temperature-dependent in the temperature range of the oral environment. Therefore, dentists should select a soft denture liner according to the clinical situation (*e.g.*, alveolar ridge resorption).

In the present study, the curves for E' , E'' , and $\tan \delta$ for a given frequency (0.1, 0.2, 1, 5, and 10 Hz) in the temperature range of -150 °C to 200 °C were superimposed to create master curves at the reference temperature of 37 °C using the time-temperature superposition principle [16] for each material, enabling the evaluation of viscoelasticity over a very large range of times and frequencies. In a clinical situation,

permanent soft liners and tissue conditioners are subjected to rapidly applied forces, caused by mastication. Furthermore long-term forces caused by functional pressure or changes in the oral supporting tissues are also subjected. Thus it is also important to evaluate viscoelastic properties at as lower frequencies as possible in addition to 1 Hz. Such master curves cover times and frequencies outside the range easily accessible by practical experiments. Without the application of the time-temperature superposition principle, an extremely long time would be necessary to measure material properties, especially at lower frequencies. The master curves obtained in the present study show that the rheological parameters for the silicone permanent soft liner exhibited almost no sensitivity to frequency, whereas those for the acrylic permanent soft liner and tissue conditioner were frequency-dependent, especially at lower frequencies. It was also found that the tissue conditioner and acrylic permanent soft liner had lower E' and higher $\tan \delta$ values than those for the silicone permanent soft liner at lower frequencies. Materials with lower E' and higher $\tan \delta$ values flow under continuous weak pressure [14]. Therefore, tissue conditioners would flow under continuous weak functional pressure to allow mucosal tissues to change shape during tissue healing, whereas silicone permanent soft liners would not flow to maintain the dimensional integrity of the lining. The dynamic viscoelastic properties for each material were found to be reasonably in line with what is required for clinical application.

Differences in T_g and dynamic viscoelastic properties were found among the tested materials. The T_g value for the acrylic permanent soft liner (BIO LINER) was higher than that for the tissue conditioner

(SOFT-LINER), which in turn was higher than that for the silicone permanent soft liner (GC RELINE II) for both DSC and DMA. These differences are due to the compositional and structural differences among the materials. The autopolymerized silicone permanent soft liner used in this study comprised polydimethyl siloxane and silica. It has been reported that the T_g value of polydimethyl siloxane is about -120 °C and that melting of the crystal occurs at about -50 °C [37]. The T_g values for the silicone permanent soft liner obtained from DSC and DMA in the present study are consistent with a previous report [37]. The melting temperature of the crystal observed in DMA in this study is also consistent with the above report. Although no differences in T_g have been found among silicone materials, the rheological parameters, such as E' , E'' , and $\tan \delta$, vary due to differences in the degree of polymerization and the content and size of silica [37]. The silicone permanent soft liner was found to have a lower T_g than those for the other two materials. The non-cross-linked structure and the plasticizer in the tissue conditioner lead to lower T_g values compared with those for the acrylic permanent soft liner that contains monomer and is cross-linked amorphous polymer.

Our findings demonstrate that the evaluation method (DSC or DMA) and the frequency in DMA have a significant influence on the T_g value for an acrylic permanent soft liner or tissue conditioner even though they only slightly influence the T_g value for a silicone permanent soft liner. In DMA, higher frequencies tended to produce higher T_g values in all materials. When the frequency was changed from 0.1 to 10 Hz, the increase in T_g for the acrylic materials (acrylic permanent soft liner and tissue conditioner) was about

18 °C and 17 °C, respectively, and that of the silicone permanent soft liner was about 7 °C. The influence of the testing frequency on the dynamic mechanical properties of the acrylic products was higher than that on those of the silicone permanent soft liner. A higher frequency led to higher values of E' and E'' , especially for the acrylic products because polymeric materials generally exhibit stiffer properties at higher frequencies. Therefore, the T_g values that exhibit relaxation of the molecular of the materials shifted to a higher temperature at higher frequency. A similar phenomenon has been observed with autopolymerized hard direct denture reline resins [28]. Significant differences were found in the T_g values for the acrylic permanent soft liner and tissue conditioner between DSC and DMA (with 6 frequencies), whereas no significant differences were found for the silicone permanent soft liner. The T_g values for silicone are thus not influenced by the measurement method. The method for the determination of T_g for acrylic materials should be selected according to the experimental purpose, and T_g should be analyzed in consideration of the selected method. Furthermore the measurable temperature range in DMA of acrylic materials is dependent on the softness because the specimens are pulled during measurement and are snapped at different temperatures. DTA and TMA can also be employed for the thermal analysis of polymeric materials, and thus future studies should utilize these methods for the rigorous evaluation of dental materials such as soft denture liners and tissue conditioners.

The hardness requirements for soft denture liners and tissue conditioners are specified in terms of Shore hardness in ISO 10139 [29, 30]. The relationship between Shore hardness and viscoelasticity was

analyzed in the present study. A positive linear relationship was found between Shore A0 hardness and E' values, but not E'' and $\tan \delta$ values. Shore hardness thus reflects the elastic component, which is represented by E' , for materials. Shore hardness is generally measured using a durometer. The rod of the durometer is pressed against the material of interest and is stopped at the position where the force of the durometer spring is equal to that on the material. Shore hardness thus evaluates only elasticity; viscosity cannot be evaluated. The clinical efficacy of soft denture liners and tissue conditioners depends on viscosity as well as elasticity. A revision of the ISO specification for soft denture liners and tissue conditioners that includes the evaluation of both elasticity and viscosity might be necessary.

Conclusions

Within the limitations of the present study, the following conclusions can be drawn:

1. For soft denture liners and tissue conditioners, the curves of storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$) versus frequency obtained at various temperatures could be superimposed to create master curves by applying the time-temperature superposition principle.
2. The acrylic permanent soft liner and tissue conditioner exhibited viscoelastic properties and were frequency-dependent, especially at lower frequencies. In contrast, the silicone permanent soft liner exhibited elastic properties and almost no sensitivity to frequency.

3. The glass transition temperature (T_g) for the acrylic permanent soft liner was higher than that for the tissue conditioner, which in turn was higher than that for the silicone permanent soft liner for both dynamic mechanical analysis (DMA) and differential scanning calorimetry (DSC).
4. In DMA, the application of a higher frequency to the soft denture liners and tissue conditioner tended to produce higher T_g values.
5. A positive linear relationship was found between Shore A0 hardness and E' values, but not E'' and $\tan \delta$ values.

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Conflict of interest The authors declare that they have no conflict of interest.

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Figure Legends

Fig 1. Variations in storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$) values with temperature for GC RELINE II at various frequencies.

Fig 2. Variations in storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$) values with temperature for BIO LINER at various frequencies.

Fig 3. Variations in storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$) values with temperature for SOFT-LINER at various frequencies.

Fig 4. Storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$) values for the three tested materials at 1 Hz and 37 °C.

Fig 5. Relationship between frequency and $\tan \delta$ for BIO LINER at various temperatures.

Fig 6. Master curves for storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$) for the three tested materials at a reference temperature of 37 °C.

Fig 7. Shore A0 hardness for the three tested materials.

Fig 8. Correlation between storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$), and Shore A0 hardness.

Table 1 Information on tested soft denture liners and tissue conditioner

| Material | Manufacturer | Type | Batch no. | Composition* | |
|---------------------|--|---|--|---|--|
| GC RELINE II (Soft) | GC Corp., Tokyo, Japan | Soft denture liner (silicone permanent soft liner) | 1401151 | Base: vinylpolysiloxane, silicon dioxide, platinum catalyst | Catalyst: vinylpolysiloxane, silicon dioxide |
| BIO LINER | Nissin Dental Products Inc., Kyoto, Japan | Soft denture liner (acrylic permanent soft liner) | Powder: 515369000 Liquid: 515369010 | Powder: poly(n-butyl methacrylate), benzoyl peroxide | Liquid: methacrylic ester, fatty acid ester |
| SOFT-LINER | GC Corp., Tokyo, Japan | Tissue conditioner (acrylic temporary soft liner) | Powder: 1303261 Liquid: 1303272 | Powder: methacrylate polymer | Liquid: phthalate plasticizer, ethanol |

*From manufacturer

Table 2 Glass transition temperature (T_g) values obtained by dynamic mechanical analysis (DMA) at various frequencies and differential scanning calorimetry (DSC) for the 3 tested materials.

| Material | DSC | DMA | | | | | |
|---------------------|--------------------------------|--------------------------|-----------------------------|--------------------------------|--------------------------------|-----------------------------|--------------------------|
| | | 0.1 | 0.2 | 0.5 | 1 | 5 | 10 (Hz) |
| GC RELINE II (Soft) | -122.9 (0.3) | -127.5 (1.0) | -124.9 (1.0) | -124.3 (3.5) | -122.8 (3.5) | -120.3 (7.8) | -120.1 (2.9) |
| BIO LINER | -17.0 (2.0) ^{a, b, c} | -27.3 (9.4) ^a | -24.5 (9.3) ^{a, b} | -22.6 (8.4) ^{a, b, c} | -19.0 (6,2) ^{a, b, c} | -11.6 (7.7) ^{b, c} | -9.6 (5.4) ^c |
| SOFT-LINER | -66.4 (2.5) ^a | -61.3 (5.0) ^a | -62.9 (5.9) ^a | -56.5 (4.4) ^{a, b} | -57.1 (4.2) ^{a, b} | -46.7 (10.0) ^b | -44.3 (9.2) ^b |

Unit: °C

(): SD

Matching letters indicate no significant difference.

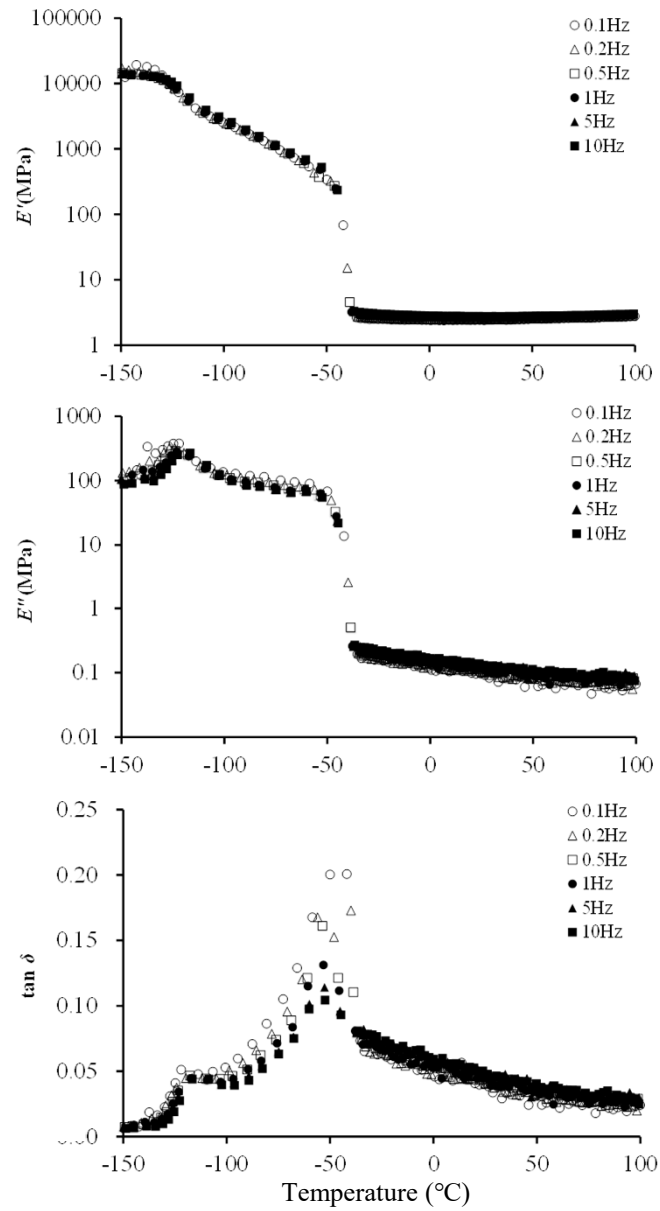


Fig 1. Variations in storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$) values with temperature for GC RELINE II at various frequencies.

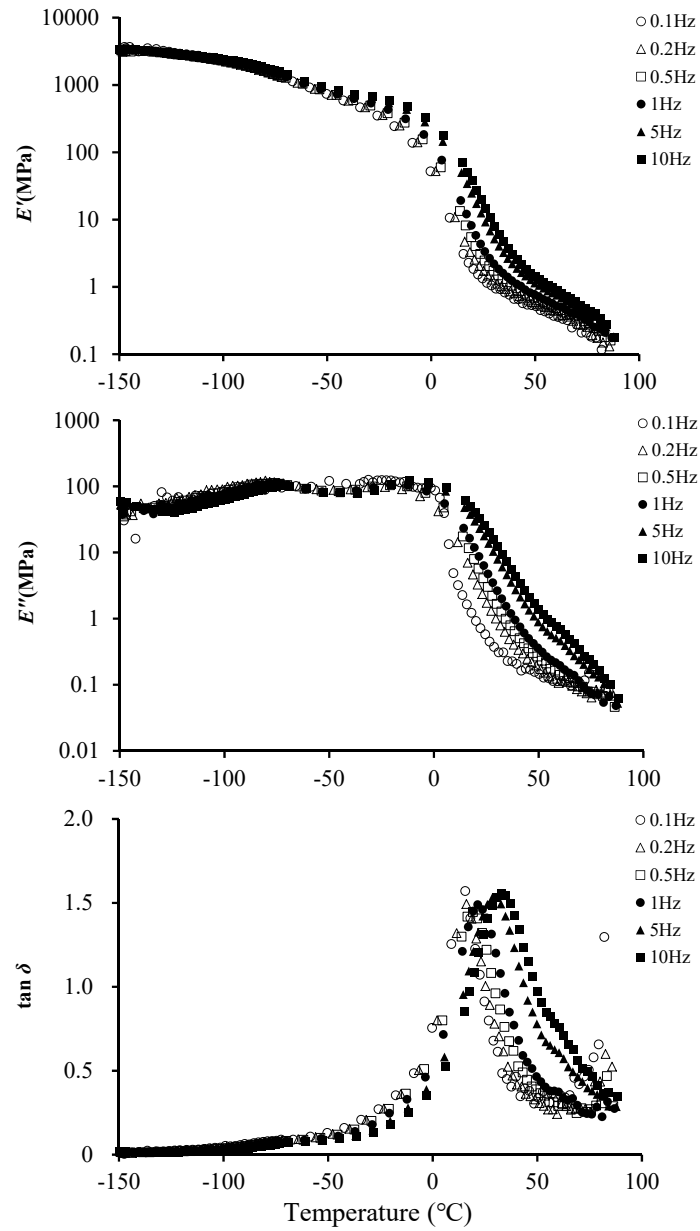


Fig 2. Variations in storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$) values with temperature for BIO LINER at various frequencies.

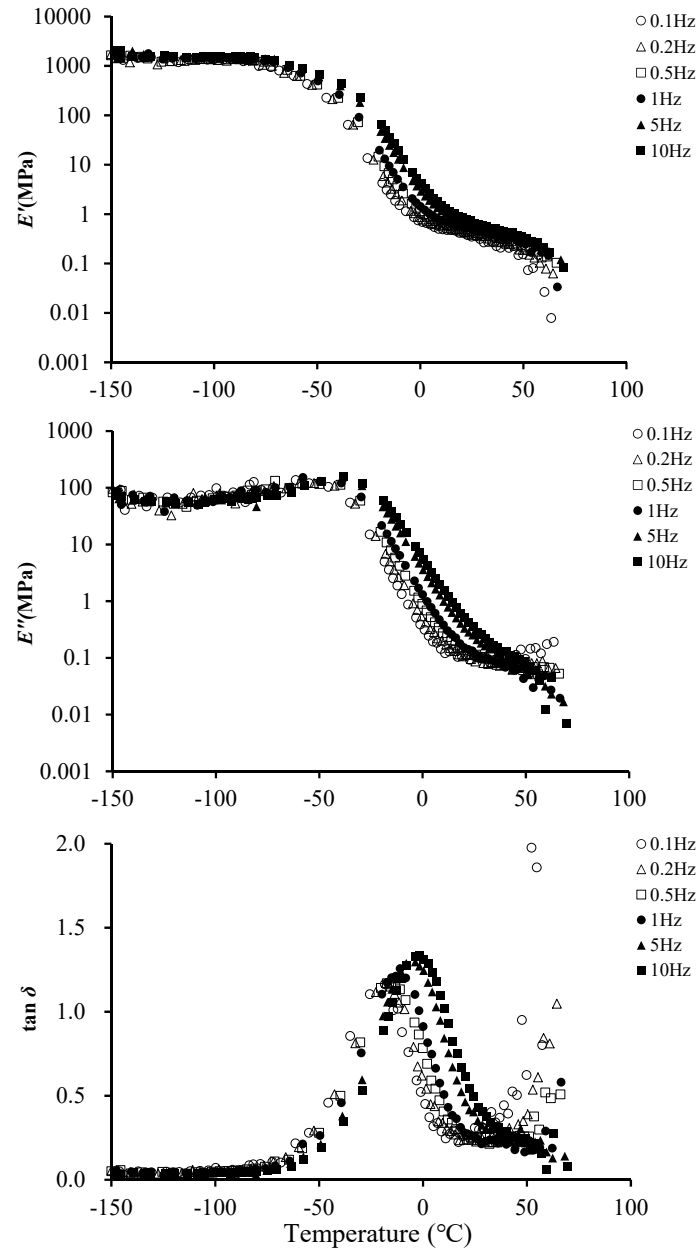


Fig 3. Variations in storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$) values with temperature for SOFT-LINER at various frequencies.

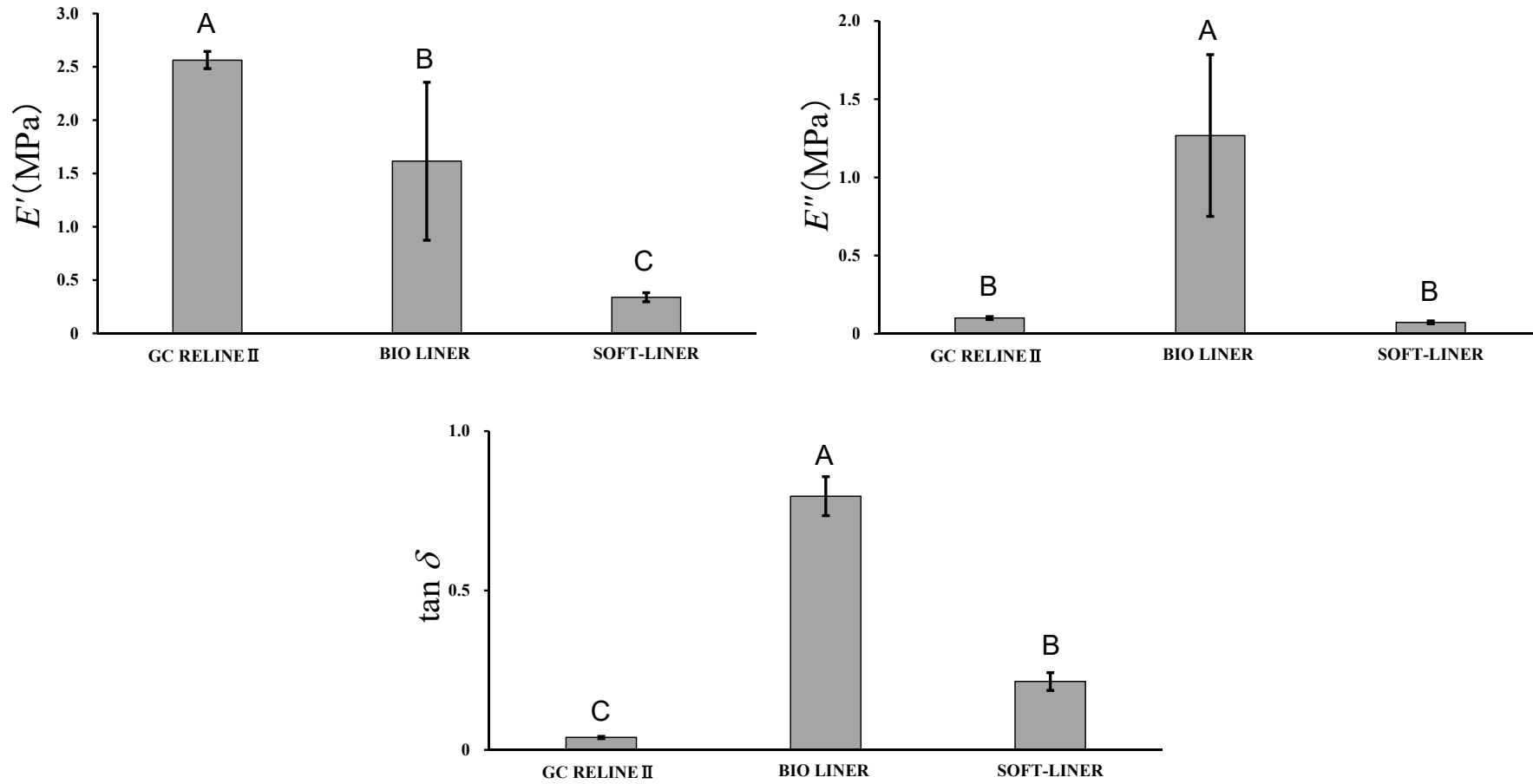


Fig 4. Storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$) values for the three tested materials at 1 Hz and 37°C.

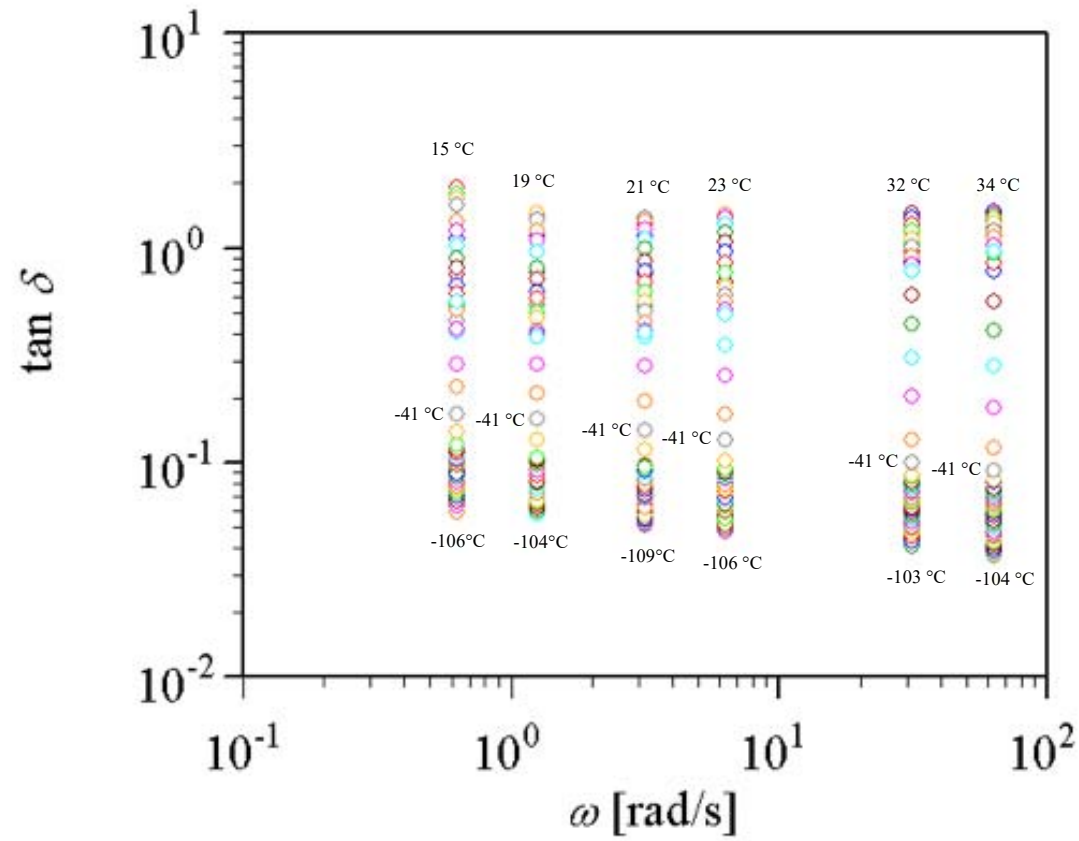


Fig 5. Relationship between frequency and $\tan \delta$ for BIO LINER at various temperatures.

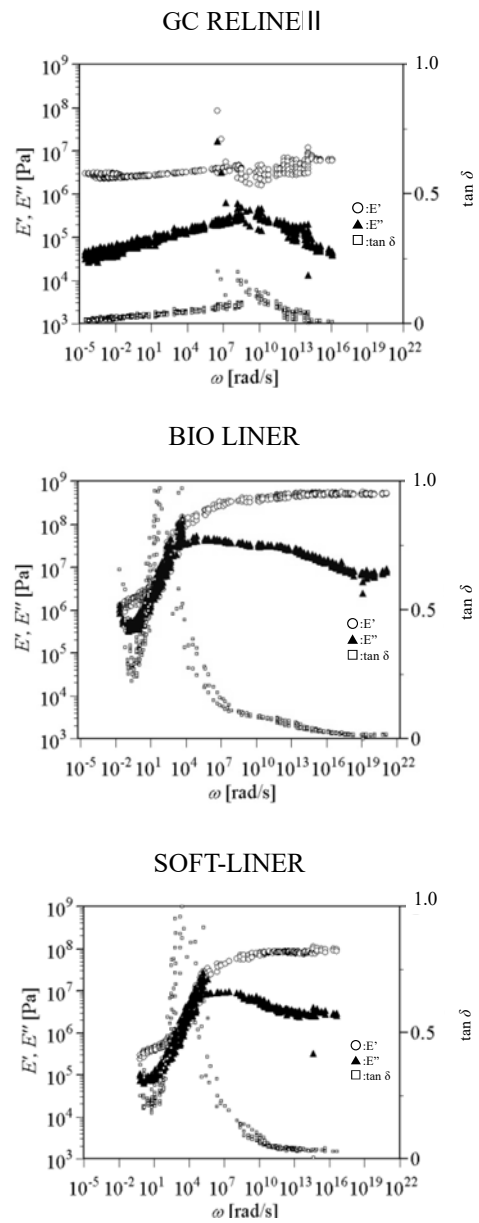


Fig 6. Master curves for storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$) for the three tested materials at a reference temperature of 37°C.

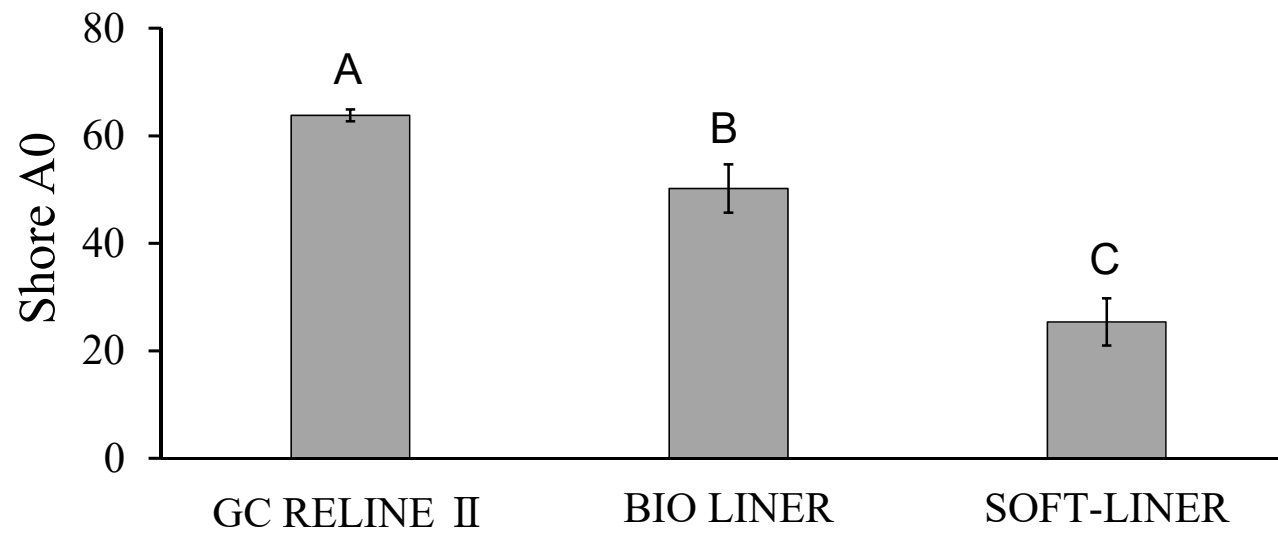


Fig 7. Shore A0 hardness for the three tested materials.

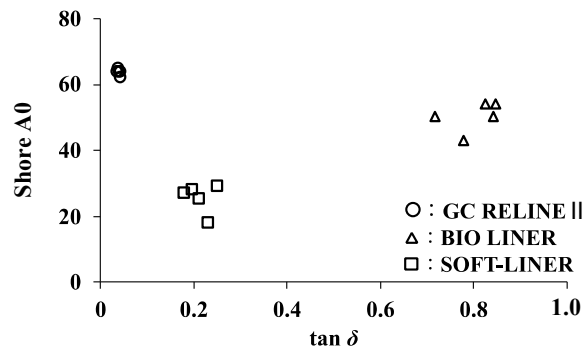
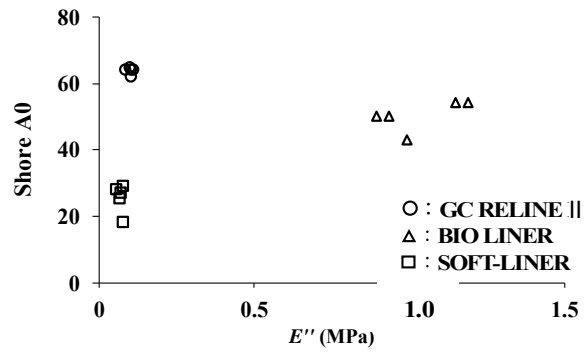
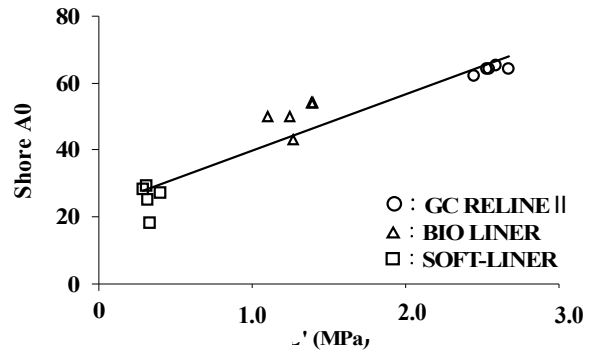


Fig 8. Correlation between storage modulus (E'), loss modulus (E'') and loss tangent ($\tan \delta$), and Shore A0 hardness.