Mechanical viscoelastic behavior of dental adhesives

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Short title: Viscoelastic behavior of dental adhesives

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Abstract

Objectives: The purpose of the study was to evaluate the mechanical viscoelastic properties of different dental adhesive materials after both dry and wet storage and under different testing temperatures.

Methods: Six materials were tested: Silorane Adhesive System (SL), Heliobond (HE), One-Step Plus (OS), Optibond Solo Plus (OP), cmf Adhesive System (CF) and Protobond (PR). Static and dynamic testing was performed after 24 h of dry and wet storage under temperatures from 21 $^{\circ}$C to 50 $^{\circ}$C. Shear and elastic moduli, Poisson's ratio, creep recovery and other viscoelastic parameters were calculated.

Results: Most of the materials were deleteriously affected by the presence of water and the increase of temperature, but not in the same magnitude. CF presented the highest shear and flexural moduli, while OS and OP were affected the most by the storage and testing conditions. OP showed the highest recovery under creep testing, while SL exhibited the highest permanent deformation.

Significance: The adhesives tested can perform satisfactorily under clinical conditions, as long as they do not come in contact with water. HEMA-containing adhesives are more affected by water.

Keywords: viscoelasticity; elastic modulus; dental adhesives; water storage; creep

1. Introduction

The need for restorative materials that could replace amalgam led to the introduction of composite resins that are now widely used by dentists. One of the most important steps that consequently led to the popularity of these materials was the breakthrough in adhesive technology. The finding that acid etching enamel led to higher bond strength between resin and enamel resulted in further research and the understanding of hybrid layer and dentin etching. Dental adhesives have evolved since then and many commercial products are available with different compositions and different approach to the way they deal with tooth tissue[1]. Their aim is to achieve strong bonding between dental tissues and restorative materials which will provide clinical longevity to the restoration.

Despite the advancements in bonding, the bonded interface of composite restorations is still the weakest area of the restoration and the main reason for failures such as marginal discoloration and poor marginal adaptation which may later lead to loss of retention[2]. This is apparent in the fact that various strategies and types of adhesives are used in order to achieve the most satisfactory bonding performance. Without regard to the steps required, the approaches contemporary adhesives use are two: etch-and-rinse and self-etch. In the former approach the tooth substrate is first etched and rinsed (conditioning stage), followed by a priming stage and the application of the bonding resin[3]. On the other hand, self-etch approach does not require a separate etch-and-rinse stage and uses acidic monomers that simultaneously condition and prime dentin and enamel[4].

The resulting composite restorations are a complex system that consists of different substrates and interfaces. The long-term performance of these restorations is the result of the behavior of the various components and their ability to withstand stress and deformation. Testing separately the various components of the composite restoration can help to identify which is the least stable under different conditions[5]. While the viscoelastic behavior of the restorative composite resins is often studied, adhesive resins are not commonly examined regarding their mechanical behavior. This phenomenon may be attributed to the fact that

research is focused mainly on their bonding properties and that some techniques cannot be applied directly to materials that are used in thin layers[6].

The aim of this study was to evaluate the mechanical properties of various commercial dental adhesives under different conditions both under static and dynamic testing. The null hypothesis was that the materials will not present differences in their properties and will not be affected by storage and testing conditions.

2. Materials and methods

Six commercially available materials were tested and are shown in Table 1. In the case the material consisted of more than one component only the bonding component was tested. Cylindrical specimens (d = 1 mm, l = 18 mm) from each material were made with the use of glass capillary tubes and were thoroughly light-cured according to the manufacturers' instructions (600 mW/cm^2, Coltulux 4 light, Coltene Whaledent, Altstätten Switzerland).

The materials were tested under four different conditions (n = 4 for each condition):

i) Tested dry at 21^{0}C, after 24 h of storage in room temperature of 21 ^{0}C.

ii) Tested wet at 21^{0}C, after 24 h of storage in distilled water at 21 ^{0}C.

iii) Tested wet at 37^{0}C, after 24h of storage in distilled water at 37 ^{0}C.

iv) Tested wet at 50^{0}C, after 24h of storage in distilled water at 50 ^{0}C.

The specimens were mounted using a jig for centering between a Plexiglas disc (0.5 mm thick) and a rod. The experiments were performed using an apparatus (Fig. 1), previously described by Lakes[7], that is capable of testing cylindrical specimens under torsion or bending and measuring viscoelastic behavior. A permanent Sm-Co magnet was attached to the end of each specimen and placed in the center of a Helmholtz coil producing a torque (M) of 3.53 x10^{-3} Nm/A controlled by the current in the coil. The weight of the magnet caused only a small constant axial tensile stress and there was no constraint on the specimen for

neither torsion, nor extension. The laser beam from a low power He–Ne laser was reflected by a small mirror (d = 1.55 mm) attached to the magnet to a calibrated chart at a distance $D =$ 944 cm. The rotation angle of the mirror is given by $\varphi = 2X / D$, where X is the displacement of the laser beam to the chart.

Static and creep experiments

The determination of the static shear modulus of the materials was achieved with the rapid application of a constant torque to the specimen for 10 s. The angular displacement was recorded and the torque was then released. The shear modulus $G = \sigma/\gamma$ was calculated from the equation $G = 2ML/ \pi R^4 \varphi$. The shear stress and shear modulus at 10 s show the short-time viscoelastic response of the material. Compliance J is the reciprocal of the shear modulus.

Young's modulus of elasticity E was obtained by repeating the experiment after the coil was rotated for 90° in order to achieve bending. Again, a constant torque was applied to the specimen for 10 s and then "instantaneously" released. The distribution of flexural strain ε, in a circular cylinder in bending is: $\varepsilon = r\varphi / L$. Young's modulus $E = \sigma / \varepsilon$ was calculated from the equation: $E = 64ML / \pi d^4 \varphi$. Poisson's ratio v was calculated from: $E = 2G(1 + v)$, using the values of G and E calculated in static measurements.

Creep measurements were also made, with the specimens being tested 24 h after fabrication. A constant torque was applied to the materials and the angular displacement was recorded for 3 h. The stress was then released and the recovery was recorded for 50 h. Three different torques were used corresponding to stresses of 2.03, 3.38 and 4.73 MPa.

Dynamic experiments – Storage modulus

Under dynamic experiments within linear viscoelastic behavior, stress and strain vary sinusoidally. Storage modulus (G_1) which is the real part of the complex modulus G* is in phase with strain, while the loss modulus G_2 (the imaginary part of the complex modulus G*, related to the dissipation of energy) is 90^0 out of phase with strain. In stiff solids usually the complex modulus G^* is approximately equal in magnitude to the storage modulus G_1 because G_2 is small compared to G_1. The ratio of the imaginary part to the real part (G_2/G_1) of the complex modulus G* is called the loss tangent (tan δ) and represents the phase angle between stress and strain sinusoids. Loss tangent is proportional to the energy loss per cycle while in the framework of linear viscoelasticity.

Frequencies ranging from 1 to 150 Hz were applied in the dynamic vibration of the specimens. A function generator connected to the Helmholtz coil creates a sinusoidal torque. The displacement or amplitude was measured on the chart for each frequency. Viscoelastic parameters were calculated from the resonance frequency v_0, corresponding to the peak amplitude and the resonance full width Δ_v which is the difference between the two frequencies at which the amplitude is half of the maximum. The loss tangent is equal to:

$\left(\dfrac{1}{\sqrt{3}}\right)\dfrac{\Delta v}{v_0}$, while shear storage modulus G_1 was calculated from the following equation: v_0

$=\left(\dfrac{1}{2\pi}\right)\sqrt{\dfrac{G_1\pi r}{2LI}}$, where r is the specimen's radius, L is its length and I is the moment of inertia of the magnet which was measured at 5.5 x 10^{-8} kg m^2. Dynamic viscosity was calculated from: $n^* =\left(\dfrac{1}{\omega_0}\right)\sqrt{\left(G_1^2+G_2^2\right)}$, where $\omega_o = 2\pi v_o$. The quality factor, Q, indicates the shape of the resonance curve. A high value of Q correlates with a peaked resonance curve and little damping. Q was calculated from the equation Q = $v_0/\Delta v$.

Statistical analysis

The results were analyzed statistically using two-way analysis of variance (ANOVA) and Bonferroni post-hoc tests ($\alpha = 0.05$). The independent parameters were the materials and the testing conditions for the static and dynamic testing, while for creep testing the independent parameters were the materials and the initial stress.

3. Results

The results of static testing are shown in Table 2 with mean values of G, E and ν, while creep results are shown in Table 3. Mean values of G_1, $\tan\delta$, n* and Q are shown in Table 4, while the effect of water and temperature on various properties is depicted in Fig. 2. Shear modulus values ranged from 0.19 to 1.99 GPa depending on testing conditions, while Young's modulus ranged from 0.67 to 5.69GPa among the different materials. Material OS became very soft when tested at 37°C and 50°C wet and the values obtained for its moduli were extremely low. For such low moduli, the weight of the magnet begins to contribute error and the corresponding values for OS result in unrealistic Poisson's ratio values and consequently the values for these conditions were omitted.

CF was the material exhibiting the highest G and E values under static testing (p <0.005) in all testing conditions. HE showed the lowest values when tested dry at 21°C, while OP had the lowest G and E when tested wet in all temperatures. Most materials were affected by the different testing conditions exhibiting a decrease in their moduli with the presence of water and the increase of temperature.

Under dynamic testing the results were similar, with CF presenting the highest G_1 values, while HE and OP presented the lowest values when tested dry at 21°C. Under the other testing conditions it was OS that showed the lowest values. Loss tangent ($\tan\delta$) increased with the presence of water and the increase of temperature, contrarily to the quality factor (Q) that changed in the opposite way.

The adhesives tested were susceptible to creep deformation as can seen in Fig. 3 and Fig. 4. Depending on the torque applied and the material, different initial strains were recorded.

Under the lowest stress some of the adhesives managed to recover fully after 3 hrs. In the two highest stresses all adhesives exhibited varying permanent deformation.

Among the materials tested OP showed the least permanent deformation after 50 hrs of recovery, while SL had the highest deformation percentage and did not manage to fully recover in any of the three stresses applied.

4. Discussion

Based on the results of the study the null hypothesis has to be rejected; the materials tested differed significantly in their behavior and they were affected by storage and testing conditions

Adhesives are under the influence of both mechanical and chemical factors that lead to changes in their mechanical properties[8]. They are one of the components of the complex system of tooth substance- adhesive – composite resin. As most contemporary composite resins shrink during polymerization it has been suggested that a low modulus adhesive could be of importance[6]. The flexible thin adhesive layer could absorb the polymerization stresses caused by shrinkage diminishing the undesirable effects of setting strain. However, finite element analysis has shown that this low modulus layer does not help dissipate the stresses, but leads to stress concentration at the stiffer regions of the restoration[9]. Moreover, adhesives are placed in thin layers and even if the material is compliant, may not add much to the structural compliance of the restoration, hence to its deformability in the clinical setting.

Storage modulus G_1 defines the energy stored elastically in the material, while loss modulus corresponds to the energy loss as heat. A material with high enough storage modulus is able to better withstand stress, creating a high-modulus hybrid layer that may reduce the stress concentration zone and the magnitude of the stress[9]. On the other hand, an extremely high modulus would result in a brittle material that would not perform in a satisfactory way under clinical conditions. The dental adhesives tested however possess moduli much lower than dentin, enamel and the composite resins that are used in the restorations. CF presented

the highest values among the materials tested with PR and SL following. CF contains a relatively stiff resin backbone as it consists mainly of bulky monomers Bis-GMA and Bis-EMA resulting in less flexible polymer chains. CF also presented the highest moduli G and E under static testing. On the other hand, the lowest moduli when the materials were tested dry were presented by HE which is an unfilled material, containing only resin. The inclusion of fillers is known to increase the modulus of a composite material providing stiffness and the fact that the unfilled material HE was the least stiff supports this theory. Unfortunately, the fact that filler composition was not available for all materials did not made it possible to perform correlations between filler content and the mechanical properties tested. When dry, the flexural modulus for the materials ranged from 2.77 GPa to 5.69 GPa, values that can be considered satisfactory for low-filled materials such as dental adhesives. However it must be pointed that in the case of adhesives that require the use of a separate primer, it has been shown that the presence of the primer affects adversely the mechanical properties of the bonding agent/primer mixture[10] and the values found here may be lower in clinical conditions for the whole adhesive layer than the ones for only the bonding component.

The mechanical properties of most dental adhesives examined in the study were deleteriously affected by storage in water for 24 hrs. The decrease of G ranged from 6% to 73% after 24 hrs of storage in water compared to the values of dry storage for 24 hrs. OP and OS were the most affected materials from the presence of water, while PR was not affected by storage in water. Decrease of E was found to be between 1.5% and 58% with SL and PR not showing any significant difference. The reduction in the mechanical properties after only 24 h of storage is considerable for some of the materials. Moreover, it has been shown that prolonged storage time in water leads to further deterioration of the properties [5, 11], so the difference between mechanical properties under dry and water storage may increase for longer periods of storage time. It should be noted that these conditions ideally are not close to the clinical conditions as the adhesives do not come to immediate contact with the oral environment after placement. The rate of change in the mechanical properties will probably

be slower *in vivo* and cannot be directly extrapolated to clinical conditions as the adhesive is between the tooth and the composite resin[12]. However, factors as rinsing, air-drying and incomplete evaporation of solvents may play an important role on the properties of the adhesives as has been shown in some cases between dry and wet bonding[13]. Although the presence of a modest amount of moisture is needed in the case of wet bonding and has been reported to not affect much the properties prior to curing[14], contamination of the cured adhesive after polymerization should be avoided in the next stages of the restoration as the current findings show it can jeopardize the mechanical behavior of the adhesive. With possible microleakage and the presence of water inside the dentinal tubules, a deterioration of the mechanical properties of the adhesives over time can be expected.

The degradation caused by water is also observed in composite resin polymers [15] and can be attributed to the plasticization caused by water. The molecules of water enhance the movement of the polymer chain segments by increasing free volume, thus increasing the flexibility of the material. Water is attracted to polar groups forming hydrogen bonds, resulting to "bound water" which is responsible for plasticization[16]. The highest percentage of modulus decline was observed in the materials containing HEMA as one of their monomers, namely OS and OP. It should be noted that SL contains HEMA in its primer, but in the present study only the bonding components were tested and SL bond does not contain HEMA. Despite the fact that the unfilled bonding resin HE had the lowest G and E when tested dry, it presented higher values compared to OS and OP in other conditions, being less affected by water than the other two filled materials that contain HEMA. The absence of acidic monomers in HE makes it hydrophobic and less sensitive to the effect of water.

HEMA is a small molecule with significant hydrophilicity used as an adhesion-promoting monomer[17]. It is used as a solvent for monomers that are less miscible and improves the miscibility of adhesive blends[18], while preventing phase-separation[19]. It can absorb water both as a monomer and when it is part of the polymer chains, leading to water uptake that influences the mechanical strength. HEMA-containing adhesives have been found to present

higher water sorption values than other adhesives and be more flexible with inferior qualities[20]. The increase of HEMA concentration is accompanied by an increase of the plasticization action of absorbed water and results in lowering of both glass transition temperature and stiffness[12]. After polymerization poly-HEMA attracts water and creates hydrogels which weaken the mechanical strength of the polymer[18]. The current findings show that HEMA-containing adhesives are more prone to the effects of water, even when compared to resin-based materials without fillers.

Most materials were affected by the increase of temperature as the parameters investigated are temperature dependent. Increase of temperature increased the mobility of polymer chains leading to more flexible materials. However not all materials exhibited the same magnitude of changes in their properties. Materials OS and OP were the ones affected the most, with OS reaching extremely low values of modulus at 50 $^{\circ}$C and its behavior becoming more rubbery. Such low values in clinical conditions could lead to debonding under constant loading and are not satisfactory, but high temperatures in the oral conditions are not continual and occur only during the consumption of hot food and beverages. Moreover, the adhesive layer is covered by the bulk of the composite resin and longer contact times would be needed in order for properties to be affected that much. Material PR was the one that exhibited the most stable behavior, being the material least affected both by water and increase of temperature and showing the most desired performance under storage and testing conditions.

All adhesives tested exhibited linear viscoelastic behavior under creep testing as indicated by the superimposition of their respective creep curves. Creep compliance was a function of time only and not of the stress magnitude. Initial loading caused an initial elastic response and was subsequently followed by a time-dependent viscoelastic creep deformation. After load removal the recovery phase begins and consists of two stages: the first is an instantaneous elastic recovery and then a slower retarded elastic recovery.

Restorations are subjected to occlusal forces during mastication under repeating loading cycles. In this study static creep was used, however a strong correlation has been reported between static creep and the theoretically more clinically relevant dynamic creep[21]. Recently, this has been confirmed as a strong correlation between dynamic creep strain and maximum static creep strain and an even stronger correlation between dynamic creep strain and static permanent deformation was reported[22].

The increase of the applied stress resulted into an increase in the ineleastic, irreversible strain component for all the adhesives. Material HE presented the highest initial elastic strain due to its absence of fillers, but after 3 h of loading it was OP that showed the highest viscoelastic deformation and total strain. On the other hand, OP showed a remarkable ability to recover as it was the material with the lowest permanent deformation after 50 h despite being the most susceptible material to creep loading. CF also exhibited satisfactory recovery values, same or close to OP, but also managed to be the most resistant material to deformation with the lowest strain values after loading. Exhibiting low creep-strain values can be helpful under clinical conditions as these materials can be dimensionally more stable for longer periods of time and under aggressive conditions such as acid or enzyme attacks in the oral environment[22]. SL showed a relatively high deformation and moreover showed the least recovery after 50 h which makes for least satisfactory performance overall among the materials tested.

Creep testing was performed under dry conditions and this should be taken into account, as creep is known to be increased by water sorption[23] and the materials tested here are affected in different magnitude by the presence of water as seen in the results of the other experiments. The obtained values could be different under other conditions, as apart from compositional factors like resin volume and filler volume and size, the presence of water plays an important role in creep resistance.

5. Conclusions

The dental adhesives tested varied in their mechanical behavior mainly because of their different compositions. While in general they showed satisfactory mechanical properties, the effects of water and temperature were significant, resulting in some cases in materials with very low mechanical properties that could influence negatively the restoration in long term. During adhesive application, clinicians should be very careful after curing to take all necessary measures in order to create an adhesive layer with optimal mechanical performance. Especially in the case of HEMA-containing adhesives which were found to be the most prone to testing conditions, presence of water after polymerization should be avoided.

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Table 1. The materials used in the study.

Table 2. Results of static testing (mean values and standard deviation).

Table 3. Results of torsional creep testing (mean values and standard deviation).

Table 4. Results of dynamic testing (mean values and standard deviation).

Fig. 1. The apparatus used in the study.

Fig. 2. G_1, E, n* and tanδ for the adhesives tested under the different testing conditions.

Fig. 3. Typical creep and recovery curves of the materials tested under stress of 4.73MPa.

Fig. 4. Creep compliance curves under constant stress of 4.73 MPa for 3 h and 50 h of recovery.

Table 1

Material	Composition	Type
Silorane Adhesive System (SL) 3M ESPE - USA	Hydrophobic dimethacrylate, phosphorylated methacrylates, TEGDMA, initiators, stabilizers Fillers: Silane treated silica	Two-step self-etch: Self-etch primer and bond
Heliobond (HE) Ivoclar Vivadent - FL	Bis-GMA, TEGDMA, initiators, stabilizers	Bonding resin
One-Step Plus (OS) Bisco - USA	Bis-GMA, HEMA, BPDM, acetone Fillers: 8.5% wt. glass ionomer	Two-step etch and rinse: Etching gel and bond
OptiBond Solo Plus (OP) Kerr - USA	Bis-GMA, HEMA, GDM, GPDM, ethanol Fillers: 15% wt. 0.4 μm barium glass, fumed silica, sodium hexafluorosilicate	Two-step etch-and-rinse: Etching gel and bond
cmf adhesive system (CF) Saremco - SW	Bis-GMA, Bis-EMA Fillers: Silanized barium glass	Three-step etch-and-rinse: Etching gel, primer and bond
Protobond (PR) Dental Co-operative - GR	Bis-GMA, Bis-EMA, HPMA, initiators, stabilizers	Two-step etch-and-rinse: Etching gel and bond

Table 2

		SL	HE	OS	OP	CF	PR
Shear modulus G (GPa)	21^0C Dry	1.28 (.02)	0.98 (.03)	1.35 (.02)	1.06 (.04)	1.99 (.05)	1.18 (.07)[b]
	21^0C Wet	1.15 (.04)	0.76 (.01)	0.36 (.06)[a]	0.39 (.03)[a]	1.87 (.05)	1.16 (.06)[b]
	37^0C Wet	1.02 (.05)	0.58 (.04)	0.19 (.03)	0.29 (.05)	1.35 (.03)	1.09 (.04)[b]
	50^0C Wet	0.80 (.01)	0.45 (.03)	---	0.18 (.02)	0.94 (.04)	0.84 (.05)
Young's modulus E (GPa)	21^0C Dry	3.61 (.04)[c]	2.77 (.03)[d]	3.92 (.03)	3.12 (.04)	5.69 (.06)	3.45 (.08)[f]
	21^0C Wet	3.61 (.02)[c]	2.73 (.05)[d]	1.66 (.05)[e]	1.61 (.03)[e]	5.38 (.04)	3.41 (.03)[f]
	37^0C Wet	2.95 (.03)	1.70 (.04)	0.72 (.04)	0.99 (.05)	3.95 (.07)	3.33 (.05)
	50^0C Wet	2.34 (.05)	1.55 (.02)	---	0.67 (.02)	2.96 (.05)	2.59 (.06)
Poisson's ratio v	21^0C Dry	0.414 (.004)[g]	0.409 (.008)[g]	0.448 (.011)	0.466 (.008)[h]	0.426 (.006)[i]	0.466 (.01)[h]
	21^0C Wet	0.415 (.009)[g]	0.448 (.005)	0.550 (.007)	0.619 (.004)	0.431 (.007)[i]	0.473 (.009)
	37^0C Wet	0.452 (.007)	0.463 (.006)	0.859 (.005)	0.722 (.009)	0.449 (.008)	0.523 (.007)[k]
	50^0C Wet	0.470 (.01)	0.717 (0.009)	---	0.811 (.007)	0.578 (.011)	0.533 (.006)[k]

*mean values with same superscript letters are not significantly different ($p > 0.05$)

Table 3

Materials	Applied torque $(Nm/A \times 10^{-5})$	Initial Stress (MPa)	Initial strain γ_0 $(rad \times 10^{-4})$	% residual strain of initial strain (γ_t/γ_0)	% recovery after 3 hrs of creep	Initial shear modulus at 10s (GPa)
SL	5.29	2.03	1.75 (.11)[a]	3.77 (.32)[g]	97.41 (.15)	
	8.82	3.38	2.70 (.19)[d]	4.27 (.28)	96.49 (.14)	1.24 (.05)
	12.35	4.73	3.69 (.23)[e]	6.01 (.45)	95.82 (.22)	
HE	5.29	2.03	2.06 (.18)[b]	3.73 (.24)[g]	98.48 (.21)	
	8.82	3.38	3.81 (.15)[c]	3.80 (.21)[g]	97.53 (.24)[k]	0.85 (.11)
	12.35	4.73	6.36 (.20)	3.86 (.29)[g]	97.47 (.19)[k]	
OS	5.29	2.03	2.03 (.12)[b]	0	100[i]	
	8.82	3.38	3.70 (.14)[c]	1.51 (.26)	98.98 (.10)[j]	1.06 (.05)
	12.35	4.73	4.36 (.17)	7.04 (.33)	98.01 (.13)	
OP	5.29	2.03	2.04 (.17)[b]	0	100[i]	
	8.82	3.38	2.93 (.21)[d,f]	13.80 (.28)	99.1 (.16)[j]	1.09 (.13)
	12.35	4.73	4.00 (.16)	18.20 (.39)	98.81 (.14)	
CF	5.29	2.03	1.78 (.18)[a]	0	100[i]	
	8.82	3.38	3.14 (.22)[f]	1.51 (.23)[h]	98.89 (.12)[j]	1.15 (.10)
	12.35	4.73	3.75 (.21)[e]	3.15 (.31)	97.78 (.17)[l]	
PR	5.29	2.03	1.30 (.16)	0	100[i]	
	8.82	3.38	2.02 (.14)	1.57 (.32)[h]	98.89 (.18)[j]	1.59 (0.04)
	12.35	4.73	2.97 (.17)	7.48 (.19)	97.8 (.13)[l]	

*mean values with same superscript letters are not significantly different (p > 0.05)

Table 4

		SL	HE	OS	OP	CF	PR
Storage modulus G_1	21^0C Dry	1.36 (.04)^a	1.15 (.04)^b	1.36 (.05)^a	1.14 (.07)^b	2.12 (.06)	1.27 (.06)
	21^0C Wet	1.30 (.08)^a	1.13 (.04)^b	0.54 (.09)^c	0.49 (.11)^c	1.94 (.05)	1.18 (.08)^b
	37^0C Wet	1.14 (.03)^b	0.95 (.07)	0.19 (.06)	0.33 (.08)^d	1.6 (.10)	1.11 (.07)^b
	50^0C Wet	0.99 (.09)	0.80 (.04)	---	0.25 (.07)^d	1.29 (.07)	0.99 (.09)
Loss tangent $\tan\delta$	21^0C Dry	0.026 (.005)^e	0.040 (.005)	0.053 (.006)	0.064 (.004)	0.016 (.005)	0.028 (.006)^e
	21^0C Wet	0.039 (.004)	0.071 (.004)	0.139 (.007)^f	0.139 (.008)^f	0.056 (.008)	0.046 (.004)^g
	37^0C Wet	0.055 (.008)	0.091 (.006)	0.248 (.007)	0.192 (.007)	0.079 (.006)	0.050 (.007)^g
	50^0C Wet	0.071 (.006)	0.103 (.007)^h	---	0.249 (.01)	0.101 (.005)^h	0.105 (.009)^h
Dynamic viscosity n*	21^0C Dry	1.47 (.08)	1.15 (.12)	1.82 (.09)	2.44 (.13)^i	4.23 (.14)	2.43 (.08)^i
	21^0C Wet	2.80 (.12)^j	2.53 (.09)	1.64 (.08)	2.83 (.15)^j	7.53 (.12)	4.62 (.13)^k
	37^0C Wet	2.62 (.09)	3.30 (.14)	0.78 (.11)	2.32 (.13)	6.85 (.15)	4.60 (.09)^k
	50^0C Wet	2.46 (.10)	2.11 (.11)	---	2.07 (.10)	6.17 (.11)	4.20 (.14)
Quality factor Q	21^0C Dry	38.40 (1.34)	25.31 (1.62)	18.69 (1.22)	15.61 (1.48)	60.65 (1.51)	35.36 (1.61)
	21^0C Wet	25.78 (1.18)	14.05 (1.49)	7.18 (0.98)^l	7.18 (0.69)^l	18.01 (1.16)	21.79 (1.52)^m
	37^0C Wet	18.18 (1.43)^m	10.94 (1.50)^{n,p}	4.03 (1.33)^o	5.22 (1.25)^o	12.62 (1.32)^n	19.95 (1.18)^m
	50^0C Wet	14.11 (0.88)	9.72 (1.27)^p	---	4.02 (0.76)^o	9.85 (1.24)^p	9.52 (0.97)^p

*mean values with same superscript letters are not significantly different (p > 0.05)

Support rod

Specimen

rsion coil

Bending co

Laser beam

Mirror

Magnet