

# A correlation between the optical and mechanical properties of novel polysilane/polysiloxane nanocomposites

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Received: 9 February 2009 / Revised: 20 April 2009 / Accepted: 22 April 2009 /  
Published online: 5 May 2009  
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**Abstract** Poly(di-*n*-hexyl)silanes (PDNHS), which are  $\sigma$ -conjugated conductive polymers were synthesized by the conventional Wurtz-type coupling reaction. PDNHS were incorporated into polymer networks obtained by cross-linking vinyl terminated poly(dimethyl)siloxanes and methylhydrosiloxane-dimethylsiloxane copolymers. From the UV spectra of polysilane/polysiloxane composites, it can be derived that direct correlations exist between the structure of the polysiloxane matrix and the conformation of small amounts of added polysilanes. The degree of cross-linking plays an important role to understand the structure of the polysiloxane matrix. In addition, rheological measurements indicate that the mechanical properties and the degree of cross-linking in the PDNHS/polysiloxane composites are directly correlated. This implies that the optical properties of small amounts of added PDNHS can be correlated to the mechanical properties of the surrounding polymer matrix. Furthermore, this correlation confirms that PDNHS are suitable to probe the properties of complex polymeric structures.

**Keywords** Polysilane · Polysiloxane · Nanocomposite

## Introduction

Conductive polymers have been developed for many uses, such as in corrosion inhibitors [1], compact capacitors [2, 3], antistatic coatings [4], electromagnetic shielding of computers [5], and in smart windows [6] that can change colors and transparency. Furthermore, research is being done on the more sophisticated applications of conductive polymers in transistors [7], light-emitting diodes [8, 9],

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lasers [10], and solar cells [11]. Low cost and flexibility are some of the reasons why conductive polymers are attractive. However, for commercial applications, the poor processing abilities and the low conductivity of many accessible conductive polymers have inhibited their widespread use. In order to avoid these disadvantages, composites of conductive polymers and other polymers [2, 5] have been developed. In this paper, polymer composites of polysilane as a conductive polymer and polysiloxane are described.

Polysilanes, which are  $\sigma$ -conjugated polymers with a one-dimensional Si backbone and organic substituted side chains, have been used in this study as the conductive polymer [10]. Polysilanes have several advantages over other conductive polymers, making them very suitable for the preparation of polymer composites. First of all, functionalized polysilanes are readily accessible. Such polysilanes can be deposited in well-organized layers with suitable electrical and optical properties [7, 8, 12–14]. Another interesting property of polysilanes is that the relationship between the structure and optical properties of polysilanes has been well established [10]. Therefore, the optical spectroscopy of polysilanes under given conditions can be used to obtain information about the backbone conformations of polysilanes. Finally, polysilanes are transparent in the visible region of the spectrum [7, 8], thus they do not affect the visible optical characteristics of the matrix.

Polysilanes absorb radiation in the ultraviolet due to a  $\sigma$ - $\sigma^*$  electronic transition associated with the extensive delocalization of charges along the silicon backbone. This  $\sigma$ - $\sigma^*$  electronic transition is dependent on the polysilane backbone conformation, which itself is sensitive to external stimuli such as temperature and pressure. Poly(di-*n*-hexyl)silane (PDNHS) exhibits four types of conformations depending on the surrounding medium as well as physical properties such as temperature and pressure. In solution at room temperature, the wavelength maximum of PDNHS is 317 nm, as a result of the presence of a helix conformation. With decreasing temperature, solutions of PDNHS exhibit thermochromism, which is evident as an abrupt red shift to 370 nm of the wavelength maximum at  $-30\text{ }^\circ\text{C}$  [15]. This wavelength shift indicates that in low temperature solutions the conformation of PDNHS is dominated by a *trans* planar conformation. In the solid state, PDNHS exhibits a *trans* planar conformation with the wavelength of 370 nm at room temperature. Furthermore, it has been observed that the application of high pressure in the solid state results in a shift of the wavelength maximum of PDNHS to 350 nm signifying a conformational change from *trans* planar to TG $TG'$  [16]. As a result, UV spectroscopy gives an excellent indication of the backbone conformation of polysilanes in general and PDNHS in particular (Table 1). Whereas such stimulus responsive behavior has been studied in detail, the effect of the mechanical properties of the surrounding medium has been so far unexplored.

Polysiloxanes have been used as the second component of the composites. Polysiloxanes are used for many industrial applications such as cosmetics, food processing materials and medicinal preparations [17]. In addition, polysiloxanes exhibit excellent mechanical, chemical and thermal stabilities [18]. The structure and synthetic chemistry of polysiloxanes as well as the curing processes of polymer networks are well known [18]. Furthermore, the degree of crosslinking of polysiloxanes can be easily controlled by adjusting the amount of starting materials.

**Table 1** The correlation between the conformation and the wavelength maximum of the  $\sigma$ – $\sigma^*$  transition in alkyl substituted polysilanes

Conformation	Wavelength maximum (nm)
<i>trans</i> planar	~ 370
TGTG'	~ 350
7/3 helix	~ 317
Random coil	<300

The transparency of polysiloxanes and polysiloxane networks also allows the observation of optical properties of the conductive polymers.

In this paper, polymer composites of polysilane synthesized as a conductive polymer and polysiloxane as matrix have been prepared. Various polysiloxane matrices have been used and their effect on the  $\sigma$ – $\sigma^*$  electronic transition has been studied. The goal of this research is to examine whether polysilanes can be employed to probe the mechanical properties of the surrounding matrix.

## Experimental

### Materials

The conventional Wurtz-type coupling reaction was used for preparing PDNHS and purified by reprecipitation of toluene with methanol. The purified polymer had the molecular weight of  $1.43 \times 10^6$  and the polydispersity of 2.1.

For the preparation of the polymer nanocomposites, a suitable matrix was chosen in which the PDNHS were embedded. The silicone matrix was prepared from vinyl terminated polydimethylsiloxanes (**A**) and methylhydrosiloxane-dimethylsiloxane copolymers with 25–30% of methylhydrosiloxane repeating units (**B**), or methylhydrosiloxane-dimethylsiloxane copolymers with 0.5–1.0% of methylhydrosiloxane repeating units (**C**). In general, vinyl terminated polymers are used in addition cure systems with a platinum catalyst. The platinum catalyzed hydrosilylation reaction takes place between the vinyl groups of **A** and the Si–H functionalities of **B/C**. A platinum (platinum-divinyltetramethyldisiloxane complex in xylene) was employed for curing silicones at room temperature.

In the studies presented here, PDNHS with methoxyl end caps ( $-\text{OCH}_3$ ) (**D**) were added before curing of the silicone network. Six different polysiloxane networks were prepared with different degree of cross-linking: Three networks were prepared from **A** and **B** and another three networks were prepared from **A** and **C** in different ratios. PDNHS **D** were incorporated into the six different polysiloxane networks respectively, giving a total of six composites (Tables 2, 3).

In order to prepare a composite, first, **A** and **B/C** were combined. Subsequently, dissolved PDNHS in hexane (30 ml) was added. After adding dilute platinum catalyst complex (0.01  $\mu\text{l}$  with hexane 5 ml), the resulting mixture was stirred to obtain a homogeneous mixture. Experiments were done at room temperature. It was noticed that incorporation of extra hexane reduced viscosity of mixtures, leading to disappearance of bubbles developed during mixing within a few hours. Therefore, it

**Table 2** A, B, and D network compositions

Composite	A (g)	B (g)	A:B	D (g)	wt.% of D in network
1	5.86	0.38	4:0.25	0.0076	0.122
2	4.01	1.00	4:1	0.0065	0.130
3	4.00	2.01	4:2	0.0075	0.124

**Table 3** A, C, and D network compositions

Composite	A (g)	C (g)	A:C	D (g)	wt.% of D in network
4	1.97	2.02	4:4	0.0047	0.118
5	1.04	4.05	4:16	0.0063	0.124
6	0.28	4.02	4:57	0.0053	0.123

was not necessary to use vacuum system during curing. For complete curing process, networks with **B** needed 2 days. However, in case of networks with **C**, at least ten days were needed due to lower mole percentages of methylhydrosiloxanes in **C** (0.5–1.0%) in comparison to **B** (25–30%) and the high viscosity of the starting compounds. Dynamic viscosities were measured daily to ensure the status of curing. When dynamic moduli were saturated, the optical and mechanical properties of composites were investigated.

### Characterization

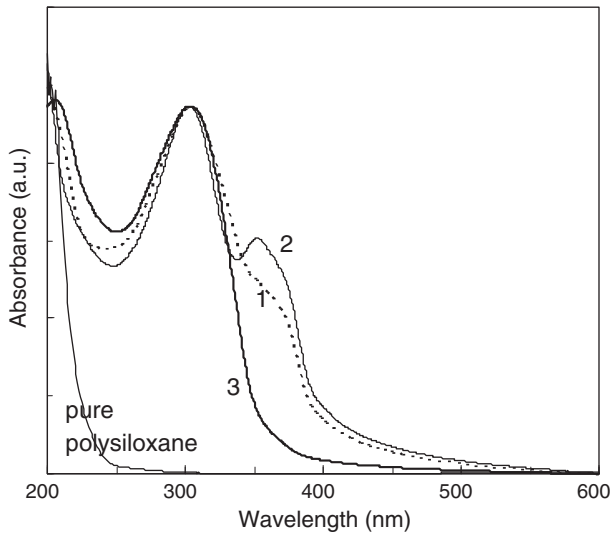
For absorption experiments at room temperature, polysiloxanes network films (0.5–1.0 mm, thickness) were sandwiched in between two quartz plates after which they were mounted in the optical path of the UV/VIS/NIR spectrophotometer (V-570 Jasco Corporation).

A Bohlin VOR rheometer (Bohlin Instruments Inc.) with parallel plate measuring system was used for measuring the mechanical properties. For dynamic viscosity, frequency sweep was performed at frequency of 0.1–30 rad/s and strain of 1%. All measurements have been performed with a 90 g-cm transducer and 30 mm diameter plates at room temperature. Sample thickness ranges from 0.5 to 1.0 mm.

## Results and discussion

### UV absorption of PDNHS/polysiloxane composites

The UV spectroscopy of the PDNHS/polysiloxane composites gives a direct indication of the conformation of PDNHS in the polysiloxane matrix. Figure 1 shows the UV spectra of **1–3**. Furthermore, the UV spectrum of polysiloxane



**Fig. 1** The UV spectra of pure polysiloxane and polysilane/polysiloxane composites **1**, **2**, and **3**

matrix, which has the same composition of **1** without PDNHS, was provided to ensure that no peak was observed from polysiloxanes. It was evident that no spectrum from polysiloxanes was superposed to peaks or shoulders from PDNHS. The UV absorption spectrum of **1** displays a large band ( $\lambda_{\text{max}} = 300 \text{ nm}$ ) and a shoulder at longer wavelength. Apparently various distinct conformations are present (cf. Table 1). The oscillator strength of each transition is dependent on the relative fraction of polymer repeating units in a particular conformation. The dominant 300 nm absorption maximum of **1** is due to a contribution of the random coil (290 nm) and 7/3 helix (317 nm) conformations. This reflects the presence of a distorted helical conformation, i.e. a mixture of 7/3 helix and random coil segments. The 355 nm shoulder is clearly composed of two absorption maxima, indicating that segments with TGTG' (350 nm) and *trans* planar (370 nm) conformation are present. The difference in intensity between the larger peak and the shoulders indicate that the conformation of in **1** is predominantly a distorted helix (random coil and 7/3 helix) and that smaller portions of the backbone have a TGTG' and a *trans* planar conformation. This presence of multiple conformations of the PDNHS in the polysiloxane matrix is remarkable since usually only one conformation (absorption maximum) of PDNHS is observed both in solution and in the solid state at room temperature. As a result, these unusual multiple conformational contributions have to be an effect of the surrounding matrix. However, the exact “concentrations” of the various conformations are difficult to establish, as the contributions are highly sensitive to the external temperature and thermal history of the sample.

The UV absorption spectrum of **2** shows a large resemblance with that of **1**. The only observable difference is a minor change in the shape of the shoulder. In the UV spectrum of **3**, only the  $\sigma\text{-}\sigma^*$  transition, which is associated with the distorted

helical conformation, can be observed without a shoulder. Apparently, whereas for **1** and **2** the conformational freedom of the PDNHS in the matrix is limited and the backbone is “forced” in a variety of conformations, **3** offers sufficient conformational freedom to obtain a distorted helical conformation only. In all cases the PDNHS concentration is approximately equal, indicating that the differences in the optical characteristics arise from differences in the structures of the surrounding matrix.

Tables 4 and 5 show the composition of the starting components and the degree of cross-linking for composites **1–6**. Only very small amounts (below 0.13 wt.%) of PDNHS are incorporated into all composites (see Tables 2, 3). Therefore, the added PDNHS is expected to have virtually no effect on the network composition/structure as a result of the very low concentration of PDHNS. Hence, only **A** and **B/C** are considered to calculate the degree of cross-linking.

The degree of cross-linking can be defined as the number of theoretical cross-links divided by the total number of repeating units. In this experiment, it is assumed that the hydride groups from **B** are in excess and all vinyl groups from **A** react. From Table 4, it is evident that the degree of cross-linking slightly decreases upon going from **1** to **3**, i.e. from 0.50 to 0.35%.

In view of the UV-absorption spectra of **1–3** (Fig. 1), this suggests that at degrees of cross-linking in the 0.4–0.5% range, multiple conformations exist, whereas at lower degree of cross-linking, i.e. 0.35% the additionally available conformational freedom allows the polymer to adapt a distorted helical conformation. Apparently the overall PDNHS conformation is not affected by cross-linking with the matrix. For **4–6**, **C**, methylhydrosiloxane-dimethylsiloxane copolymers with 0.5–1.0% of methylhydrosiloxane, are used instead of **B**. In addition to a lower

**Table 4** **A** and **B** composite compositions

Composites	Moles of <b>A</b> <sup>a</sup>	Moles of <b>B</b> <sup>b</sup>	Moles of vinyl group in <b>A</b>	Moles of hydride groups in <b>B</b>	Theoretical degree of cross-linking (%)
1	0.0002089	0.00019	0.0004178	0.00141	0.499
2	0.0001432	0.00050	0.0002864	0.00371	0.425
3	0.0001429	0.00101	0.0002857	0.00764	0.353

<sup>a</sup> Molecular weight of **A**: 28,000

<sup>b</sup> Molecular weight of **B**: 2,000

**Table 5** **A** and **C** composite compositions

Composites	Moles of <b>A</b> <sup>a</sup>	Moles of <b>C</b> <sup>b</sup>	Moles of vinyl group in <b>A</b>	Moles of hydride groups in <b>C</b>	Theoretical degree of cross-linking (%)
4	0.0000704	0.000037	0.0001407	0.000204	0.262
5	0.0000371	0.000074	0.0000743	0.000410	0.108
6	0.0000100	0.000073	0.0000200	0.000407	0.034

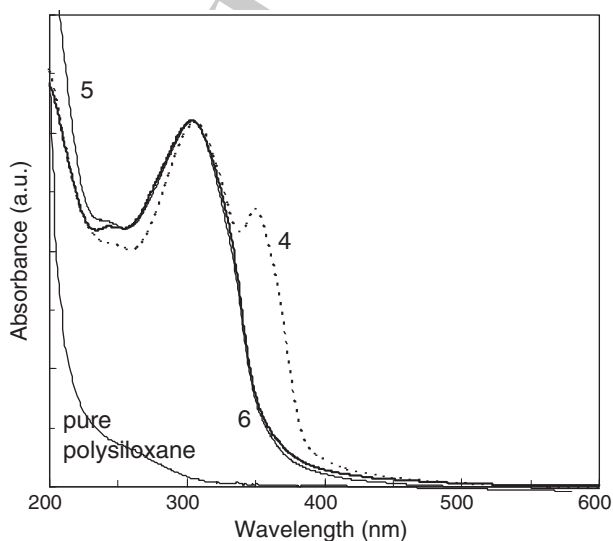
<sup>a</sup> Molecular weight of **A**: 28,000

<sup>b</sup> Molecular weight of **C**: 55,000

methylhydrosiloxane ratio, **C** has a higher molecular weight (55,000) than **B** (2,000). The compositions of **4–6** are shown in Table 3. Figure 2 shows UV spectra of composites of **4–6**. It should be noticed that the UV spectrum of polysiloxane matrix containing the same composition of **4** without PDNHS showed no significant peak or shoulder. **4** shows one peak at 300 nm with a shoulder at 355 nm. This fact indicates that the composite of **4** predominantly has a distorted helical conformation, with some of the segments in a TGTG' conformation. Only the 300 nm absorption maximum is visible in the remainder of the composites (**5** and **6**) implying that the silicon backbone of **5** and **6** adapts a distorted helical conformation.

It is interesting to rationalize the results in terms of the degree of cross-linking. In going from **4** to **6**, the degree of cross-linking reduces significantly (see Table 5). This indicates that for composites **4–6** a similar relationship exists between the degree of cross-linking and the optical properties as observed for **1–3** (vide supra). Apparently at degrees of cross-linking around 0.26% two conformations exist, at lower degrees of cross-linking (i.e. below 0.11%) the additionally available conformational freedom again allows the polymer to adapt a distorted helical conformation and no other conformations are present.

It is also interesting to note that the relationship observed for **4–6** is not completely in agreement with the one observed for **1–3**. This can have various reasons. For example, it is possible that not only the degree of cross-linking but also the distribution of the cross-linking throughout the matrix plays a role. However, in view of the high molecular weight of the PDNHS, such an effect is unlikely (i.e. the average degree of polymerization of PDNHS is much larger than that of **A**, **B** and **C**). Instead it is more likely that the addition of large excesses of **B** (in the case of



**Fig. 2** The UV spectra of pure polysiloxane and polysilane/polysiloxane composites **4**, **5**, and **6**

**Table 6** Relationship between the conformation of small amounts of added PDNHS and the degree of cross-linking of the surrounding matrix

Degree of cross-linking of matrix (%)	PDNHS conformation	$\lambda_{\max}$ (nm)
0–0.1	Distorted helix	300
0.1–0.3	Distorted helix and TGTG'	300, 350
0.3 and higher	Distorted helix, TGTG' and <i>trans</i> planar	300, 350, 370

composite **3**) causes the observed behavior. In composite **3**, **B** is present in over 25% excess, which indicates that a large amount of **B**, which has only a low molecular weight of 2,000, remained uncross-linked in the matrix. This uncross-linked **B** will act as a plasticizer, thus accounting for the increased conformational freedom of the PDNHS, as observed in **3**.

Based on the UV–Vis spectroscopic data, it can therefore be concluded that direct correlations exist between the structure of the polysiloxane matrix (i.e. the degree of cross-linking) and the conformation of small amounts of added PDNHS (see Table 6). Hence, PDNHS are suitable to probe the structure of other polymers. To further understand this relationship, the mechanical properties of the composites have been studied in more detail. It is expected that the mechanical properties correlate with the degree of cross-linking.

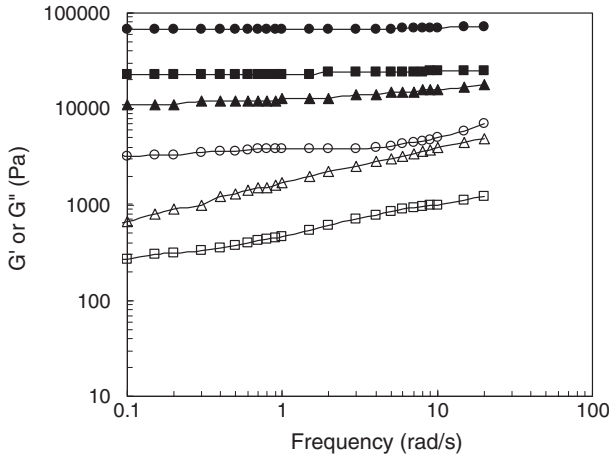
#### Mechanical properties of composites

Visco-elastic materials are characterized by two dynamic moduli, an elastic (or storage) modulus  $G'$  and a viscous (or loss) modulus  $G''$ . Both moduli have been measured for **1–6** using oscillatory mode with the parallel plate measuring system. Sample thickness ranges from 0.5 to 1.0 mm. The results of  $G'$  and  $G''$  of **1–6** are plotted against the frequency  $\omega$  with logarithmic scales (Figures 3, 4). Table 7 shows  $G'$  values and  $G''$  values at the lowest and the highest frequency.

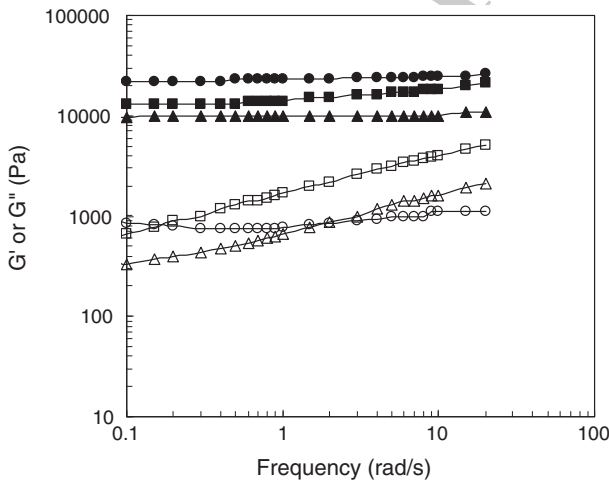
According to this definition,  $G'$  quantifies the energy stored and recovered in the elastic response per cycle and  $G''$  quantifies the energy dissipated or lost as heat in the viscous response per cycle. All composites reveal the viscous response as well as the elastic response. In all composites over the entire range of frequencies,  $G'$  is relatively flat ( $G'$  (at 20 rad/s)/ $G'$  (at 0.01 rad/s)  $\leq$  1.80). In contrast,  $G''$  exhibits substantial changes over the frequency range ( $G''$  (at 20 rad/s)/ $G''$  (at 0.01 rad/s) ranges from 0.58 to 17). In addition,  $G'$  is always larger than  $G''$ , indicating that the behavior of the composites is much more elastic than viscous, i.e. the elastic properties dominate.

In going from **1** to **3**, values of  $G'$  decrease over the entire range of shear rates, indicating that composites are less elastic with increasing addition of **B** (see Table 2). It can be expected that **3** is less elastic and **1** is more elastic. The  $G''$  graphs are also different for **1**, **2** and **3** (Fig. 3). The  $G''$  graph of **1** has small changes. However, the  $G''$  of **3** is dependent on frequency over an entire frequencies region with the largest changes of  $G''$  among three composites (**1–3**). The behavior





**Fig. 3** Dynamic moduli:  $G'$  (filled); 1, filled circle; 2, filled square; 3, filled triangle.  $G''$  (open); 1, open circle; 2, open square; 3, open triangle



**Fig. 4** Dynamic moduli:  $G'$  (filled); 4, filled circle; 5, filled square; 6, filled triangle.  $G''$  (open); 4, open circle; 5, open square; 6, open triangle

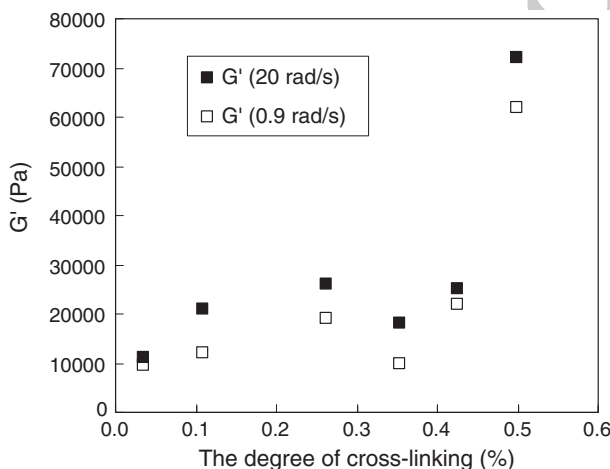
of 2 is similar to 1 and 3 at the low frequency range and at the high frequency range respectively.

Figure 4 illustrates dynamic moduli for composites 4–6. A and C are employed for composites 4–6. From 4 to 6, decreasing values of  $G'$  are observed over the entire range of shear rates with increasing C composition (see Table 4). This indicates that composites are again less elastic with increasing addition of C. For  $G''$ , the larger changes of 5 than those of 4 exhibit with the adding of C. However, the  $G''$  of 6 deviated from what has been observed in previous series composites.

Fig. 5.

**Table 7** The  $G'$  values and  $G''$  values of **1–6** at low and high frequency

	Frequency (rad/s)	1	2	3	4	5	6
$G'$ (Pa)	0.01	62,000	22,000	10,000	19,000	12,000	9,500
	20	72,000	25,000	18,000	26,000	21,000	11,000
$G' (20)/G' (0.01)$		1.16	1.14	1.80	1.37	1.75	1.16
$G''$ (Pa)	0.01	2,800	280	300	1,900	300	1,000
	20	7,000	1,200	4,900	1,100	5,100	2,100
$G'' (20)/G'' (0.01)$		2.50	4.29	16.33	0.58	17	2.10

**Fig. 5** The relationship between  $G'$  and the degree of cross-linking

It is interesting to relate the degree of cross-linking and the optical properties to the above results. It appears that in **1–6** the degree of cross-linking is correlated to  $G'$ . A decrease in the degree of cross-linking is reflected in a decrease in  $G'$  (see Tables 4, 5, 7). An exception on the above relationship forms **3**. In the case of **3**, the discussion as presented in “[UV absorption of PDNHS/polysiloxane composites](#)” that the large excess of **B** acts as a plasticizer gives a reasonable explanation of why **3** deviates. As a result, it is evident that the mechanical behavior can be correlated with the degree of cross-linking.

It is noteworthy that the fact that the mechanical properties and the degree of cross-linking can be correlated directly implies that the optical properties of small amounts of added PDNHS can be correlated to the mechanical properties of the surrounding polymer matrix. For example, a value for  $G'$  higher than 25,000 Pa (i.e. **1** and **2**) correlates to the presence of a distorted helical, a TGTG' and a *trans* planar conformation of the PDNHS probe backbone. Around 20,000 to 25,000 Pa is a critical point (i.e. **4**), at which the PDNHS backbone exhibits only a distorted helical and a TGTG' conformation. Furthermore, when  $G'$  is below 20,000 Pa (i.e. **3**, **5**, and

**Table 8** Relationship between the conformation of small amounts of added PDNHS and the mechanical properties of the surrounding matrix

$G'$ of matrix (Pa)	PDNHS conformation	$\lambda_{\max}$ (nm)
Below 20,000	Distorted helix	300
Around 25,000	Distorted helix and TGTG'	300, 350
Above 25,000	Distorted helix, TGTG' and <i>trans</i> planar	300, 350, 370

6) the PDNHS has only a distorted helical conformation. It is also an interesting fact that this correlation is valid for all composites (1–6) without any exceptions in Table 8. 3 is exceptions in the relationship between the conformation of small amounts of added PDNHS and the degree of cross-linking. This is in contrast to the relationship between the degree of cross-linking and the PDNHS probe backbone conformation, as observed from UV–Vis spectroscopy. UV–Vis spectroscopy indicated that 3 did not follow this relationship due to plasticizer effects (cf. “UV absorption of PDNHS/polysiloxane composites”). Hence it is clear that it is better to correlate the PDNHS probe backbone conformation to the mechanical properties (i.e.  $G'$ ) than directly to the degree of cross-linking.

Therefore, it can be concluded that the backbone conformation of small amounts of added PDNHS and the mechanical properties of the polysiloxane matrix have a direct correlation. In addition, this correlation between the optical properties of the PDNHS and the mechanical properties of matrix supports our claim that PDNHS are suitable to probe the properties of complex polymeric structures.

## Conclusion

In the UV spectra of PDNHS/polysiloxane composites, the more ordered conformation such as TGTG' and *trans* planar from PDNHS was observed with composites possessed the higher degree of cross-linking. It can be derived that direct correlations exist between the structure of the polysiloxane matrix and the conformation of small amounts of added PDNHS. The degree of cross-linking plays an important role to understand the structure of the polysiloxane matrix. Rheological measurements indicate that the mechanical properties and the degree of cross-linking in the PDNHS/polysiloxane composites are directly correlated. This implies that the optical properties of small amounts of added PDNHS can be correlated to the mechanical properties of the surrounding polymer matrix. Furthermore, this correlation confirms that PDNHS are suitable to probe the properties of complex polymeric structures.

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