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## International Journal of Polymer Analysis and Characterization

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713646643>

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**To cite this Article** Ioan, Silvia , Lungu, Voligona and Harabagiu, Valeria(2005) 'Optical Properties of Poly(vinyl-g-dimethylsiloxane) Copolymers', International Journal of Polymer Analysis and Characterization, 10: 5, 361 – 372

**To link to this Article:** DOI: 10.1080/10236660500479569

**URL:** <http://dx.doi.org/10.1080/10236660500479569>

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## Optical Properties of Poly(vinyl-g-dimethylsiloxane) Copolymers

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**Abstract:** Refractive index is a key criterion in the evaluation of optical polymers. The continuous development of new materials for biomedical applications such as optical fibers or lenses raises questions on the fundamental physical limits of this class of materials. Using the Lorenz-Lorentz equations, we have investigated the theoretical refractive index and specific refractive index increments of some copolymers obtained by radical copolymerization of methyl methacrylate or styrene with allyl methacrylate, followed by the hydrosilation of the resulting copolymers with mono hydro-terminated polydimethylsiloxanes, starting from the refractive increments of the functional groups.

**Keywords:** Poly(vinyl-g-dimethylsiloxane) copolymers; Refractive index; Specific refractive index increment

### INTRODUCTION

In the search for materials for new applications, the design of polymers with diverse architectures for specific properties is required. Polydimethylsiloxane (PDMS)-based materials have been widely used in many products due to the diversity of their properties and processing technologies. The unique combination of siloxane-containing copolymer properties, such as surface activity, physiological inertness (biocompatibility), high-oxygen permeability, hydrophobicity, extremely low temperature flexibility, and

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atomic oxygen resistance, offers a wide range of special applications in many fields.<sup>[1]</sup> Thus, due to their excellent blood compatibility (low interaction with plasma proteins) and high-oxygen and moisture permeability, siloxane-containing copolymers and networks have been extensively evaluated and used for blood-containing devices and contact lenses.<sup>[2-4]</sup> After the discovery that maximum comfort in contact lenses is achieved when the eye has sufficient oxygen, siloxanes became primary polymeric moieties for these types of applications due to their extremely high oxygen permeability.<sup>[5]</sup>

There are currently three types of contact lenses in use: hard, soft, and rigid gas-permeable lenses. Each of them has very distinct material properties, and due to their different properties, contact lenses not only interact with the body in different ways, but also have different life spans. Common interactions of contact lenses include protein deposits, binding of lipids, and water absorption. Less common and more severe interactions include giant papillary conjunctivitis, contact-lens-induced acute red eye, contact-lens-induced peripheral ulcer, microbial keratitis, and corneal melt.

Hard contact lenses have become rather obsolete and are made of a hard plastic, more particularly poly(methyl methacrylate) (PMMA). PMMA has very good light transmittance, toughness, and stability. However, PMMA is hydrophobic and therefore has low oxygen permeability and is very brittle, thus making it a nonideal contact-lens material.

Soft contact lenses are made of hydrogels and contain 25–79% water. Hydrogels were first used in contact lenses due to their relatively good mechanical stability, favorable refractive index, and oxygen permeability. However, oxygen permeability is limited by the water content and cannot supply more than 50% of the oxygen required for normal metabolism. These contact lenses were found to be more comfortable than hard contact lenses and more difficult to dislodge. However, several problems can be associated with soft contact lenses. They have a high level of lipid and protein binding, tear easily, dry out eyes, have a shorter life span, and are more difficult to take care of.

Rigid gas-permeable lenses (RGPs) are made with silicone and are much more flexible than typical hard contact lenses. RGPs are an excellent option for contact lenses as they are durable and oxygen-permeable, retain shape, provide crisper vision, last longer, and have less binding of proteins and lipids. The disadvantages associated with this type of lense are that they are less comfortable and slightly more expensive.

In lens applications, the refractive index of the materials has different values. To treat high myopia, the refractive index of lens materials is around 1.43, with a relative density of 1.16, and for other special lenses the refractive index is higher than 1.50.<sup>[6]</sup>

It is known that optical properties, ideal transparency, and good resistance to shock, breaking, and bending of the copolymers with minimum allyl methacrylate content slightly differ from the properties of the copolymers with methyl methacrylate.<sup>[7]</sup>

Development of various siloxane-containing copolymers for contact lens applications has received widespread attention after the synthesis and characterization of siloxane-modified methacrylate copolymers by Gaylord.<sup>[3]</sup> The toughness of these copolymers increases with PMMA content, but a corresponding loss in tensile strength occurs.<sup>[4]</sup> The transparency of PMMA could be retained by using an aromatic substituent on silicon to match refractive indices of the components.

These investigations are important because one of the features of organic polymers used in optical applications is the refractive index. Lenses, optical waveguides, and nonlinear optical devices are just three examples where this material parameter plays a key role in system design. With the widespread use of contact lenses and the close interaction they have with the human eye, there is a great need for biomaterials research in this area.

Siloxane copolymers with styrene and/or methacrylates have been also used as gas separation membranes with good selectivity or as pressure-sensitive adhesives.<sup>[3-5]</sup>

In previous articles<sup>[8]</sup> we described the synthesis of poly(methyl methacrylate) or styrene-g-dimethylsiloxane copolymers by radical copolymerization of methyl methacrylate or styrene with allyl methacrylate (AMA) followed by hydrosilation of the allyl ester side units with monofunctional hydro-terminate polysiloxane, in order to incorporate the attractive characteristics of each of the above-mentioned polymers. The structure and morphology of these copolymers were also investigated by infrared (IR) absorption, nuclear magnetic resonance spectra (<sup>1</sup>H-NMR), and gel permeation chromatography (GPC). The advantage of the synthesis of MMA/AMA copolymers consists in the applicability to obtain graft copolymers with siloxane units.

The influence of these copolymer structures on the refractive index and the specific refractive index increments is analyzed in this article in order to obtain materials with potential applications.

Accurate values of the refractive index or the specific refractive index increment ( $dn/dc$ ) of these copolymers in different solvents must be determined also in order to obtain the weight-average molecular weight ( $M_w$ ) by light scattering or to estimate the molecular weight distribution by size exclusion chromatography. For copolymers, the specific refractive index increments depend on the refractivity and content of components. This dependence becomes complicated for copolymers comprising three or more components differing in refractivity.

## EXPERIMENTAL SECTION

The synthesis of poly(vinyl-g-dimethylsiloxane) copolymers was achieved by a multistep procedure.<sup>[8]</sup> The radical copolymerization of methyl

**Table I.** Weight fractions,  $M_n$ , and polydispersity index of the poly(methyl methacrylate-allyl methacrylate) and poly(styrene-allyl methacrylate) copolymers

Sample <sup>a</sup>	Code	Weight fraction (MMA or St/AMA)	$M_n \cdot 10^{-2}$ (GPC)	$M_w/M_n$ (GPC)
P(MMA/AMA)	1	0.853/0.147	433	2.8
P(MMA/AMA)	2	0.929/0.071	561	2.2
P(MMA/AMA)	3	0.963/0.037	384	2.9
P(MMA/AMA)	4	0.981/0.019	498	2.8
P(St/AMA)	5	0.880/0.120	370	2.2
P(St/AMA)	6	0.938/0.062	304	2.0
P(St/AMA)	7	0.978/0.022	385	2.4
P(St/AMA)	8	0.986/0.014	402	2.3

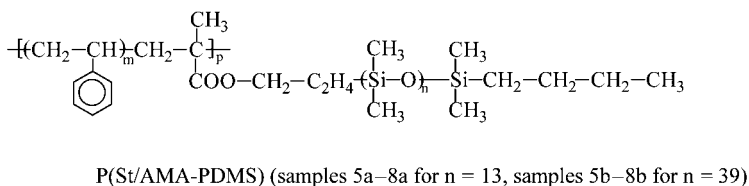
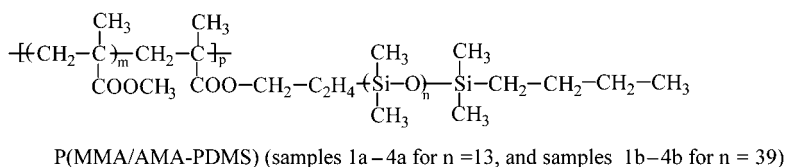
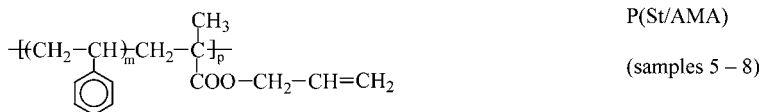
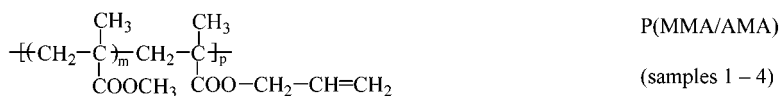
<sup>a</sup>Reaction conditions: solvent, toluene; total monomer concentration, 20%; AIBN, 0.2% vs. monomers; temperature, 80°C; duration, 8 h.

methacrylate (MMA) or styrene (St) with allyl methacrylate (AMA) allowed the synthesis of vinyl copolymers containing different proportions of unsaturated (allyl ester) groups attached to the main chain. The characteristics of the poly(methyl methacrylate/allyl methacrylate) (P(MMA/AMA)) and poly(styrene/allyl methacrylate) (P(St/AMA)) copolymers are listed in Table I. The  $M_n$  and the polydispersity index ( $M_w/M_n$ ) were determined in chloroform by GPC using an evaporative light scattering detector (PL EMD-950) equipped with two PL gel 5  $\mu\text{m}$ -C 300  $\times$  7.5 mm columns (Polymer Laboratories, UK); calibration was performed with narrow polydispersity polystyrene standards.

P(MMA/AMA) and P(St/AMA) copolymers were further hydrosilated.<sup>[8]</sup> The general chemical structures of P(MMA/AMA), P(St/AMA) copolymers and poly(vinyl-g-dimethylsiloxane) copolymers, and P(MMA/AMA-PDMS) and P(St/AMA-PDMS) resulting from hydrosilation with two mono hydro-terminated polydimethylsiloxanes ( $M = 1000$  ( $n = 13$ ) or  $M = 3000$  ( $n = 39$ )) are illustrated in Scheme 1.

The weight fractions of MMA or St and of hydrosilated groups (AMA-PDMS) in poly(vinyl-g-dimethylsiloxane) copolymers and  $M_n$  values are presented in Table II. For poly(vinyl-g-dimethylsiloxane) copolymers the GPC investigations show narrower polydispersity indexes, as compared to the corresponding precursors, of around 1.8.

Refractive indices ( $n_2$ ) were measured with an Abbé refractometer at 25°C, and specific refractive index increments ( $dn/dc$ ) in chloroform and benzene were measured with a Zeiss interferometer at 25°C and at 436 nm wavelength in the standard way. The theoretical values of the refractive index and of the specific refractive index increments of poly(vinyl-g-dimethylsiloxane) copolymers in chloroform and benzene were also



Scheme 1.

determined using the Lorenz-Lorentz equation<sup>[7,8]</sup> and the corresponding group contributions to the molar refraction and to the molar volume.

## RESULTS AND DISCUSSION

### Refractive Index Contribution of Substructure from Copolymer Repeating Units

The Lorenz-Lorentz equation (1) can be used to calculate the refractive index of a polymer if its molar refraction ( $R$ ) and molar volume ( $V$ ) are known.<sup>[9–11]</sup> Thus, it is possible to predict a chemical composition and to impose synthesis conditions of polymers depending on the required specific properties.

$$R = V \frac{n_2^2 - 1}{n_2^2 + 2} \quad (1)$$

**Table II.** Weight fractions and number-average molecular weights of the poly(vinyl-g-dimethylsiloxane) copolymers

Sample	Code <sup>a</sup>	Weight fraction (MMA or St and AMA-PDMS)	M <sub>n</sub> · 10 <sup>-3</sup> (GPC)
P(MMA/AMA-PDMS)	1a	0.853/0.147	109
P(MMA/AMA-PDMS)	2a	0.929/0.071	98
P(MMA/AMA-PDMS)	3a	0.963/0.037	53
P(MMA/AMA-PDMS)	4a	0.981/0.019	60
P(MMA/AMA-PDMS)	1b	0.853/0.147	226
P(MMA/AMA-PDMS)	2b	0.929/0.071	173
P(MMA/AMA-PDMS)	3b	0.963/0.037	80
P(MMA/AMA-PDMS)	4b	0.981/0.019	78
P(St/AMA-PDMS)	5a	0.880/0.120	41
P(St/AMA-PDMS)	6a	0.938/0.062	33
P(St/AMA-PDMS)	7a	0.978/0.022	60
P(St/AMA-PDMS)	8a	0.986/0.014	52
P(St/AMA-PDMS)	5b	0.880/0.120	81
P(St/AMA-PDMS)	6b	0.938/0.062	57
P(St/AMA-PDMS)	7b	0.978/0.022	79
P(St/AMA-PDMS)	8b	0.986/0.014	64

<sup>a</sup>From Scheme 1, n = 13 for samples 1a–8a and 5a–8a; n = 39 for samples 1b–8b and 5b–8b.

According to Equations (2) and (3), the molar refraction R and the molar volume V can be regarded as a sum of refraction increments R<sub>i</sub> and volume increments V<sub>i</sub>, each corresponding to a particular function group within the polymer repeating unit.

$$V = \left( \sum_i a_i \right) V_i \quad (2)$$

$$R = \left( \sum_i a_i \right) R_i \quad (3)$$

where a<sub>i</sub> is the number of groups *i* in the repeating unit.

The incremental values<sup>[12]</sup> of various polymer substructures are presented in Table III.

R and V calculations are illustrated in the following example for styrene:

Substructure	R <sub>i</sub>	V <sub>i</sub>
CH <sub>2</sub>	4.504	15.528
CH	3.412	8.085
C <sub>6</sub> H <sub>5</sub> (arom)	25.824	74.129
	R = 33.740	V = 97.742

**Table III.** Increments of various polymer substructures

Increment	$R_i$ (cm <sup>3</sup> /g)	$V_i$ (cm <sup>3</sup> )	$R_i/V_i$
CH <sub>2</sub>	4.50	15.53	0.290
CCH <sub>3</sub>	7.88	22.65	0.347
CO <sub>2</sub>	6.29	22.76	0.276
CH <sub>3</sub>	5.90	25.80	0.229
CH	3.41	8.09	0.422
C <sub>6</sub> H <sub>5</sub> (arom)	25.82	74.13	0.348
O (ether)	1.63	9.05	0.180
Si	9.00	31.20	0.288

From Equation (1),  $n_2$  for styrene is 1.606, in very good agreement with the experimental values of 1.59–1.60.<sup>[13]</sup>

The molar volume, molar refractivity, and refractive index of MMA, St, AMA-PDMS, P(MMA/AMA), P(St/AMA), and poly(vinyl-g-dimethylsiloxane) copolymers determined with Equations (1)–(3), where the increments of various substructures are those in Table III, are presented in Tables IV and V. The tables also contain the experimental values of  $n_2$ , the molar weight of the repeating units, and the densities calculated from the molar weight of the repeating units and molar volume ratio.

One can see that the theoretical values of  $n_2$  slightly differ from the experimental values and are in good agreement with literature data.<sup>[13,14]</sup>

**Table IV.** Molar weight of repeating units ( $m_0$ ), molar refraction (R), molar volume (V), density ( $\rho$ ), refractive index ( $n_2$ ), and specific refractive index increments (dn/dc) in chloroform (C) and benzene (B) of the constituent homopolymers and of AMA-PDMS groups

Sample	$m_0$	R (cm <sup>3</sup> /g)	V (cm <sup>3</sup> )	$\rho$ (g/cm <sup>3</sup> )	$n_2$	dn/dc (cm <sup>3</sup> /g)	
						C	B
MMA	100	24.57	86.73	1.153	1.478	0.029	-0.017
					1.489 <sup>a</sup>	0.042 <sup>a</sup>	0.002 <sup>a</sup>
AMA	126	31.09	100.08	1.259	1.534	0.069	0.028
St	104	33.74	97.74	1.064	1.606	0.143	0.097
					1.600 <sup>a</sup>	0.155 <sup>a</sup>	0.111 <sup>a</sup>
AMA-PDMS, n = 13	1209	363.95	1457	0.830	1.414	-0.037	-0.104
AMA-PDMS, n = 39	3136	947.05	3845	0.816	1.407	-0.046	-0.115

<sup>a</sup>Experimental values.

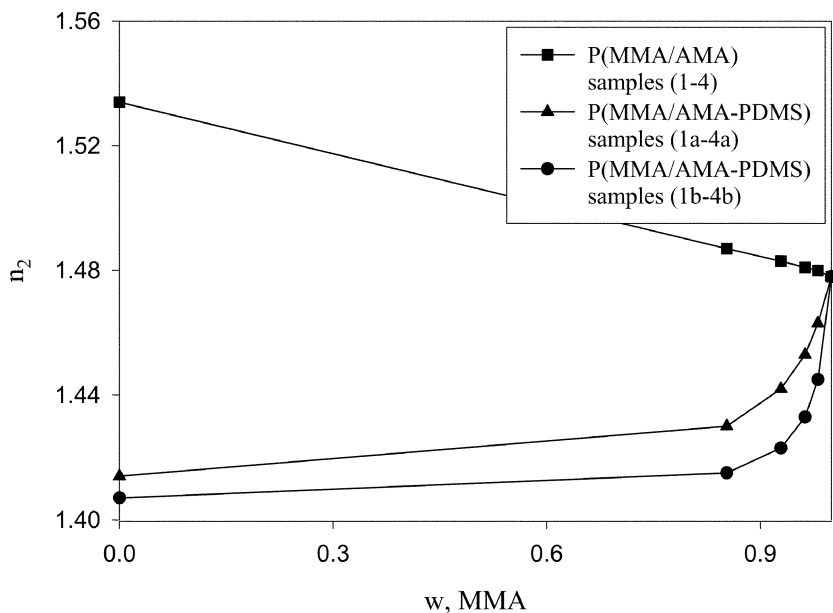


**Table V.** Molar weight of repeating units, molar refraction, molar volume, density, experimental and theoretical values of refractive index, and theoretical specific refractive index increments in chloroform (C) and benzene (B) of the poly(methyl methacrylate-allyl methacrylate), poly(styrene-allyl methacrylate), and poly(vinyl-g-dimethylsiloxane) copolymers

Sample	$m_0$	R ( $\text{cm}^3/\text{g}$ )	V ( $\text{cm}^3$ )	$\rho$ ( $\text{g}/\text{cm}^3$ )	$n_2$		dn/dc ( $\text{cm}^3/\text{g}$ ) Eq. (6)	
					Exp.	Eqs. (1)–(3)	C	B
1	710	173.59	603.12	1.177	1.494	1.487	0.035	-0.011
2	1440	352.94	1236.26	1.165	1.490	1.483	0.032	-0.014
3	2760	677.25	2381.13	1.159	1.482	1.481	0.030	-0.015
4	5410	1328.33	4679.53	1.156	1.480	1.480	0.030	-0.016
1a	1789	506.45	1960.05	0.913	1.434	1.430	0.019	-0.030
2a	2519	685.80	2593.19	0.971	1.444	1.442	0.024	-0.023
3a	3839	1010.11	3738.05	1.027	1.454	1.453	0.027	-0.020
4a	6489	1661.19	6036.45	1.075	1.468	1.463	0.028	-0.019
1b	3716	1089.55	4348.05	0.855	1.414	1.415	0.018	-0.031
2b	4446	1268.90	4981.19	0.893	1.423	1.423	0.024	-0.024
3b	5760	1593.21	6126.05	0.941	1.432	1.433	0.026	-0.021
4b	8416	2244.29	8424.45	0.999	1.446	1.445	0.028	-0.019
5	889	277.39	813.59	1.093	1.599	1.597	0.134	0.089
6	1690	537.19	1566.21	1.079	1.601	1.602	0.138	0.093
7	4862	1566.26	4547.34	1.069	1.604	1.604	0.141	0.095
8	7244	2338.90	6785.63	1.067	1.606	1.605	0.142	0.096
5a	1968	610.25	2170.52	0.907	1.473	1.474	0.121	0.073
6a	2769	870.05	2913.13	0.947	1.508	1.507	0.132	0.085
7a	5941	1899.12	5904.26	1.006	1.554	1.556	0.139	0.093
8a	8323	2671.76	8142.55	1.022	1.571	1.570	0.140	0.094
5b	3895	1193.35	4558.52	0.854	1.436	1.437	0.120	0.072
6b	4696	1453.15	5311.13	0.884	1.460	1.459	0.131	0.084
7b	7868	2482.22	8292.26	0.949	1.512	1.511	0.139	0.092
8b	10250	3254.86	10530.55	0.973	1.529	1.530	0.140	0.094

In Figures 1 and 2 the influence of the chemical structure of poly(vinyl-g-dimethylsiloxane) copolymers on the refractive index was taken into account.

Increasing the proportion of allyl methacrylate slightly increases the refractive index of the P(MMA/AMA) copolymers (samples 1–4, Figure 1) and slightly decreases the refractive index of the P(St/AMA) copolymers (samples 5–8, Figure 2). Larger refractive index differences of these copolymers were obtained than for poly(vinyl-g-dimethylsiloxane) copolymers. The presence of siloxane components in P(MMA/AMA-PDMS) and P(St/AMA-PDMS) decreases the refractive index.



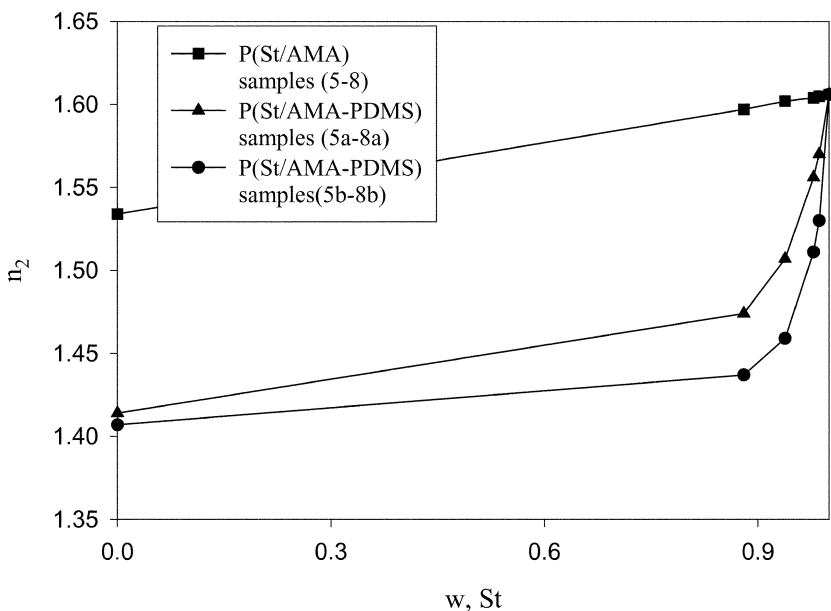
**Figure 1.** Refractive index versus weight fraction of MMA for P(MMA/AMA) and P(MMA/AMA-PDMS) copolymers with  $n = 13$  and  $n = 39$ . The dots correspond to experimental data and the lines are the theoretical curves calculated from Equations (1)–(3).

All  $n_2$  values are situated in the ranges of refractive index that are acceptable for different optical materials. P(St/AMA-PDMS) was also evaluated for use as gas-separation membranes for selectivity for or as pressure-sensitive adhesives.<sup>[3–5]</sup> Thus, by adjusting the refractive index and other properties through the synthesis of these copolymers, one can prepare materials with predicted applications.

### Specific Refractive Index Increments of Poly(vinyl-g-dimethylsiloxane) Copolymers

The  $dn/dc$  values of PMMA, PSt, PAMA, and AMA-PDMS groups were calculated with Lorenz-Lorentz equation (4),<sup>[9,11]</sup> where the refractive index of polymers,  $n_2$  (Table IV), was determined from Equation (1), and the refractive index of chloroform and benzene are  $n_1 = 1.444$  and 1.498, respectively.

$$\frac{dn}{dc} = \bar{v} \cdot \left[ \frac{(n_2^2 - 1)}{(n_2^2 + 2)} - \frac{(n_1^2 - 1)}{(n_1^2 + 2)} \right] \cdot \frac{(n_1^2 + 2)^2}{6 \cdot n_1} \quad (4)$$



**Figure 2.** Refractive index versus weight fraction of St for P(St/AMA) and P(St/AMA-PDMS) copolymers with  $n = 13$  and  $n = 39$ . The dots correspond to experimental data and the lines are the theoretical curves calculated from Equations (1)–(3).

$\bar{V}$  is the partial specific volume, which, in most cases, has been approximated by the specific volume  $v_2$  of polymer in the solid state. These values were calculated with the equation:

$$v_2 = \frac{V}{m_0} \quad (5)$$

The specific refractive index increments of P(MMA/AMA), P(St/AMA), and poly(vinyl-g-dimethylsiloxane) copolymers presented in Table V were determined considering that  $dn/dc$  values of mixed components are additive functions of composition expressed in terms of weight fraction,  $w$ .<sup>[15]</sup>

$$\frac{dn}{dc} = w_1 \cdot \left(\frac{dn}{dc}\right)_1 + w_2 \cdot \left(\frac{dn}{dc}\right)_2 \quad (6)$$

It was observed that for all copolymers the  $dn/dc$  values are lower in benzene than those in chloroform. The presence of AMA-PDMS sequences in the copolymers resulted in a decrease of  $dn/dc$  values in the following order: P(MMA/AMA) > P(MMA/AMA-PDMS) for  $n = 13$  > P(MMA/AMA-PDMS) for  $n = 39$ , and P(St/AMA) > P(St/AMA-PDMS) for  $n = 13$  > P(St/AMA-PDMS) for  $n = 39$ .

Experimental values of the specific refractive index increment for PMMA, PSt, and poly(vinyl-g-dimethylsiloxane) copolymers are in very good agreement with the theoretical data. A small discrepancy can arise if the partial specific volume  $\bar{V}$  is substituted with the specific volume  $v_2$  of solid polymers in Equation (4).

The absolute weight-average molecular weights and absolute radii of gyration for binary copolymers can be determined from apparent molecular weights and apparent radii of gyration in different solvents, by light scattering. If the copolymers are heterogeneous by composition then the molecular weight will depend on the refractive index of the solvent. For multicomponent copolymers, the determination of the structural parameters by light scattering is more difficult. High errors in determination of absolute weight-average molecular weights and absolute radii of gyration by light scattering are also observed for small specific refractive index increments of copolymers. From this reason, one can observe from Table V that benzene is an inadequate solvent for light scattering measurement of copolymers with MMA.

## CONCLUSIONS

The synthesized poly(vinyl-g-dimethylsiloxane) copolymers show structural features in correlation with their composition and preparation history. The synthesis was achieved by radical copolymerization of methyl methacrylate (MMA) or styrene (St) with allyl methacrylate (AMA) followed by hydrosilation with mono hydro-terminated polydimethylsiloxanes.

The molar properties, such as molar weight, molar refraction, and molar volume, were used to determine density, refractive index, and specific refractive index increments.

The experimental values of the refractive index for these copolymers slightly differ from those calculated with the Lorenz-Lorentz equation, considering the corresponding group contributions to the molar refraction and to the molar volume. The presence of siloxane components in P(MMA/AMA-PDMS) and P(St/AMA-PDMS) decreases the refractive index. Thus, adjusting the refractive index and other properties from synthesis of these copolymers allows the formulation of materials with expected applications.

Specific refractive index increments of MMA, St, AMA-PDMS, and AMA-PDMS groups in chloroform and benzene were theoretically obtained by the Lorenz-Lorentz equation. Experimental values slightly differ from those calculated. For poly(vinyl-g-dimethylsiloxane) copolymers the  $dn/dc$  values were determined also in chloroform and benzene considering that  $dn/dc$  values of mixed components are additive

functions of composition. It was observed that the presence of AMA-PDMS sequences in the copolymers determines a decrease of  $dn/dc$  values.

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