

ORIGINAL ARTICLE

## Influence of intermediate resin on the bond strength of light-curing composite resin to polymer substrate

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### Abstract

**Objectives.** The aim of this study was to examine the effect of intermediate resin (IMR) of different monomer compositions and viscosities on the shear bond strength between polymer substrate and light-curing composite. **Methods.** The substrate used in the study was an autopolymerizing polymethyl methacrylate (PMMA) based polymer. The substrate was treated with the IMR for 3 min before application of light polymerizable particulate filler composite resin. The monomers of the IMR were either bisphenol-A-glycidyl dimethacrylate (BisGMA) and triethyleneglycol dimethacrylate (TEGDMA) or BisGMA and methyl methacrylate (MMA). The shear bond strength of the IMR treated substrate to the particulate filler composite was evaluated after storing the specimens dry and after thermocycling the specimens in water. Light microscope examination was accomplished to determine the swelled layer of the substrate. **Results.** Significant differences were found between the shear bond strength values of the IMRs ( $p < 0.001$ ). The bond strengths were generally higher in the BisGMA-MMA groups than in the BisGMA-TEGDMA groups. Two-way ANOVA revealed significant effects of type of IMR ( $p < 0.001$ ) and thermocycling ( $p = 0.017$ ) on the shear bond strength. No interaction was found between these variables ( $p > 0.05$ ). **Conclusions.** The results suggest that the monomer composition and ratio of the IMRs used in the study influence the shear bond strength of the polymer substrate to the new resin.

**Key Words:** *intermediate resin, monomer composition, monomer ratio, shear bond strength*

### Introduction

The adequate adhesion of new resin to polymer substrate is essential when the polymeric appliances such as fiber-reinforced composite fixed partial dentures are luted to teeth with composite luting cements. Also, when polymeric appliances of removable and fixed prosthodontics are repaired, adhesion of new resin to the old one is needed. It is known that the bond between the polymer substrate and the new resin can be formed in two different ways. The presence of unreacted pendant functional groups with carbon-carbon double bonds on the surface of the polymer substrate can allow a free radical polymerization between the substrate and the new resin. However, it has been studied that the greatest reactivity of the substrate to the formation of covalent bonding can be found on the surface during the first 24 h after polymerization of the substrate [1]. Thus, the possibility to

obtain covalent bonding between the polymer substrate repaired with new resin is relatively low if the polymer substrate is older than 24 h, which is the situation in most of the repairs.

Another approach to adhere the new resin on the polymer substrate is based on interdiffusion of monomers of the new resin to the linear polymer phases of the substrate [2–4]. The bonding based on the interdiffusion of the monomers can be obtained only if the polymer substrate contains linear polymer such as polymethyl methacrylate (PMMA) and if the monomers of the new resin have a dissolving capability against the linear polymer phases of the substrate. If the diffused monomers are bifunctional, such as bisphenol-A-glycidyl dimethacrylate (BisGMA) or triethyleneglycol dimethacrylate (TEGDMA), which are commonly used in dental monomer systems, the bonding inter-phase after polymerization is a combination of cross-linked polymer of BisGMA-TEGDMA

Table I. Materials used in the study.

Code	Description of material	Manufacturer	Lot no.	Type of material
BisGMA	Bisphenol-A-glycidyl dimethacrylate	Chemotechnique Diagnostics, Tygelsjö, Sweden	400631	Monomer
TEGDMA	Triethyleneglycol dimethacrylate	Sigma-Aldrich, Steinheim, Germany	07304-067	Monomer
MMA	Methylmethacrylate	Sigma-Aldrich, Steinheim, Germany	398474/1 31899	Monomer
DMAEMA	2-dimethylaminoethyl methacrylate	Sigma-Aldrich, Steinheim, Germany	363172/1 43598	Catalyst
	Camphorquinone	Fluka Chemie, Buchs, Switzerland	395656/1 399	Initiator
Sinfony dentin A4	Octahydro-4,7-methano-1H-indenediyl-bis methylenediacyrylate 10–30 wt% + fillers	3M-ESPE Dental-Medizin GmbH & Co., Seefeld, Germany	FW 0052367	Veneering composite
PalaXpress	Polymethyl methacrylate (powder) Methyl methacrylate-Butanediol dimethacrylate (liquid)	Heraeus Kulzer, Wehrheim, Germany	393 powder 282 liquid	Autopolymerizing acrylic polymer

and linear PMMA [4]. This kind of multiphase polymer structure is a so-called semi-interpenetrating polymer network (semi-IPN) and, if the semi-IPN is located at the bonding interface, it is called secondary-IPN to distinguish it from that of bulk material's semi-IPN [5,6]. If the new resin contains monofunctional monomers like methyl methacrylate (MMA) in a combination of bifunctional monomers such as BisGMA the bonding interphase after polymerization is a semi-IPN of cross-linked BisGMA-PMMA and linear PMMA. However, in this case, the BisGMA-PMMA network, the new polymer is not as highly cross-linked as the BisGMA-TEGDMA network, which may have some influence on bond strength between the substrate and the polymerized new resin. If the new resin contains only mono-functional monomers, the formed bonding interphase is not, according to definition, the semi-IPN [4,5].

In removable prosthodontics the interdiffusion of monomers for adhering the new resin on the polymer substrate has been used for years to repair fractured removable dentures [7,8] and to adhere acrylic denture teeth to denture base polymers [2,9,10]. However, in fixed prosthodontics the methods based on the interdiffusion of monomers and especially on formation of the IPN bonding have been suggested recently [4,11,12] due to the increased use of polymer-based appliances, e.g. fiber-reinforced composite (FRC) fixed partial dentures with multiphase polymer matrix [12].

It has been reported recently that different chemical compositions of the intermediate resins (IMRs) influenced the shear bond strength of aged composite substrates repaired with new resin [4]. It was supposed that bifunctional BisGMA-TEGDMA resins and BisGMA containing monofunctional HEMA resin had a relatively good dissolving capability of the linear PMMA phases of the used substrate, while one of the IMRs that contained only bifunctional diacyrylate did not have a similar effect [4]. These results are supported by a study in which it was found

that high shear bond strengths were related to specific IMRs used [13]. It has also been reported that relative amounts of certain monomers such as BisGMA and TEGDMA had a significant effect on the mechanical properties of the resin composition [14] as well as on the solubility parameter and the fractional polarity of the resin composition [15,16]. Thus, it can be assumed that the use of intermediate resins (IMRs) with different monomer compositions and ratios may also influence shear bond strength of the polymer substrate to the polymerized new resin. Because some of the low viscosity IMRs used are only based on a mixture of bifunctional monomers such as BisGMA and TEGDMA, and the other IMRs contain both bifunctional and monofunctional monomers such as MMA or 2-hydroxyethyl methacrylate (HEMA), it is important to study the effect of different monomer compositions and ratios of the IMR on shear bond strength between the substrate and new polymer or composite.

The aim of this study was to investigate the effect of experimental IMR of different monomer compositions and ratios on bonding between polymer substrate and light-curing composite. The existence of a possible bonding interphase based on the interdiffusion of the monomers was also examined. In addition, the viscosities of different monomer compositions were measured to determine the practicability of the used IMRs.

## Materials and methods

The materials used in this study are listed in Table I. The substrate was fabricated from autopolymerizing two-component acrylic resin (PalaXpress) with a powder-to-liquid ratio of 10 g:4 ml. The polymer of the powder beads was linear PMMA and the monomers of the liquid were MMA and bifunctional monomer as a cross-linking agent. A stainless steel cylinder was filled with unpolymerized denture base resin and the filled cylinder was polymerized in water

Table II. Intermediate resin (IMR) combinations and viscosities.

Code	Material combination, percentage by weight	Viscosity cP at 40.2°C	Spindle speed, rpm
B70/M30	BisGMA 70%/MMA 30%	46.3	1.5
B50/M50	BisGMA 50%/MMA 50%	6.7	12.0
B20/M80	BisGMA 20%/MMA 80%	1.0	30.0
B0/M100	BisGMA 0%/MMA 100%	0.5	60.0
B70/T30	BisGMA 70/TEGDMA 30%	287.8	0.3
B50/T50	BisGMA 50/TEGDMA 50%	45.2	1.5
B20/T80	BisGMA 20/TEGDMA 80%	10.3	6.0
B0/T100	BisGMA 0/TEGDMA 100%	1.1	30.0

at  $(55 \pm 1)^\circ\text{C}$  for 15 min under air pressure of 300 kPa (Ivomat-type IP2, Ivoclar AG, Schaan, Liechtenstein). After the polymerization the substrates were wet ground with 1200 grit (FEPA) silicon carbide grinding paper (Struers, Copenhagen, Denmark) to expose the linear polymer phases (powder beads) of the substrate. The substrates were cleaned in distilled water with an ultrasonic cleaning device (Quantrex 90, L&R Ultrasonics, NJ) for 15 min. Before application of new resin, the substrates were stored in a desiccator at room temperature for 2 days.

The monomers used in the IMRs were either BisGMA and TEGDMA or BisGMA and MMA (Table II). TEGDMA was chosen because of it has been widely used in commercial adhesive resins and MMA because of its good dissolving capability of PMMA. The IMRs contained 0.4% camphorquinone initiator and 0.4% 2-dimethylaminoethyl methacrylate (DMAEMA) activator by weight. The viscosities of the IMRs were measured by means of a rotating spindle rheometer (Brookfield syncro-lectic viscometer, Stoughton, MA) at  $40.2^\circ\text{C}$ . The test was run at spindle speeds varying from 0.3–60 rpm. The IMRs were tempered for 5 min in the viscometer before torque was recorded and expressed in centipoises (cP). The IMRs contained 0.4% camphorquinone initiator and 0.4% 2-dimethylaminoethyl methacrylate (DMAEMA) activator by weight.

The substrates were treated with the IMR for 3 min and protected from light before application of the particulate filler composite resin (Sinfony dentin) on the unpolymerized IMR layer. The composite resin was applied on the IMR treated substrate surface by using a translucent tubular polyethylene mold with an inner diameter of 3.6 mm. The composite resin was injected into the mold from a syringe and initially polymerized with a visible light-curing unit (Elipar, 3M-Espe, Seefeld, Germany) for 40 s. The wavelength of the light of the unit was between 380–520 nm and the light intensity was  $800 \text{ mW/cm}^2$ . After the initial cure of the IMR and the composite resin, the mold was gently removed and the specimens

were polymerized in a light-curing oven (Liculite, Dentsply, Dreieich, Germany) for 5 min.

The polymerized test specimens were stored in a desiccator at room temperature of  $25 \pm 1^\circ\text{C}$  for 2 days or thermocycled in a thermocycling unit (custom made by NIOM- Scandinavian Institute of Dental Materials, Haslum, Norway) for 12 000 cycles between  $5\text{--}55^\circ\text{C}$  in deionized grade 3 water before testing the samples. The dwell time at each temperature was 30 s and the transfer time from one bath to the other was 2 s. The test specimens were divided into 16 different groups according to type of the IMR and the aging of the adhesive joint (dry storage or thermocycling fatigue). Each group contained five test specimens.

The universal testing machine (Lloyd LRX, Lloyd Instruments Ltd, Fareham, UK) with Bencor Multi-T shear assembly jig (Danville Engineering Inc., San Ramon, CA) was used to measure the shear force applied until fracture occurred. The specimens were loaded at a crosshead speed of 1.0 mm/min and the stress strain curve was analyzed with Nexygen 2.0 software (Lloyd Instruments Ltd, Fareham, UK). Univariate Analysis of Variance (ANOVA) and Tukey's post-hoc test with a significance level of 0.05 were conducted with the type of IMR and thermocycling as independent variables and shear bond strength as dependent variable.

The effect of each IMR on the swelling of the substrate was studied with a light microscope (Orthoplan, Ernst Leitz Wetzlar GmbH, Germany), a magnification of  $\times 40$ . For a comparison the B0/M100 group was used as a control IMR because of its high MMA content (100 wt%). Eight test specimens were fabricated for microscopic examination. The surface of the substrate was wet ground with 4000 (FEPA) grit silicon carbide grinding paper and cleaned in distilled water in the ultrasonic cleaning device as described above. The cleaned substrates were stored in a desiccator at room temperature of  $25 \pm 1^\circ\text{C}$  for 2 days and the substrate surface was treated for 3 min with the IMRs before adding the particulate filler

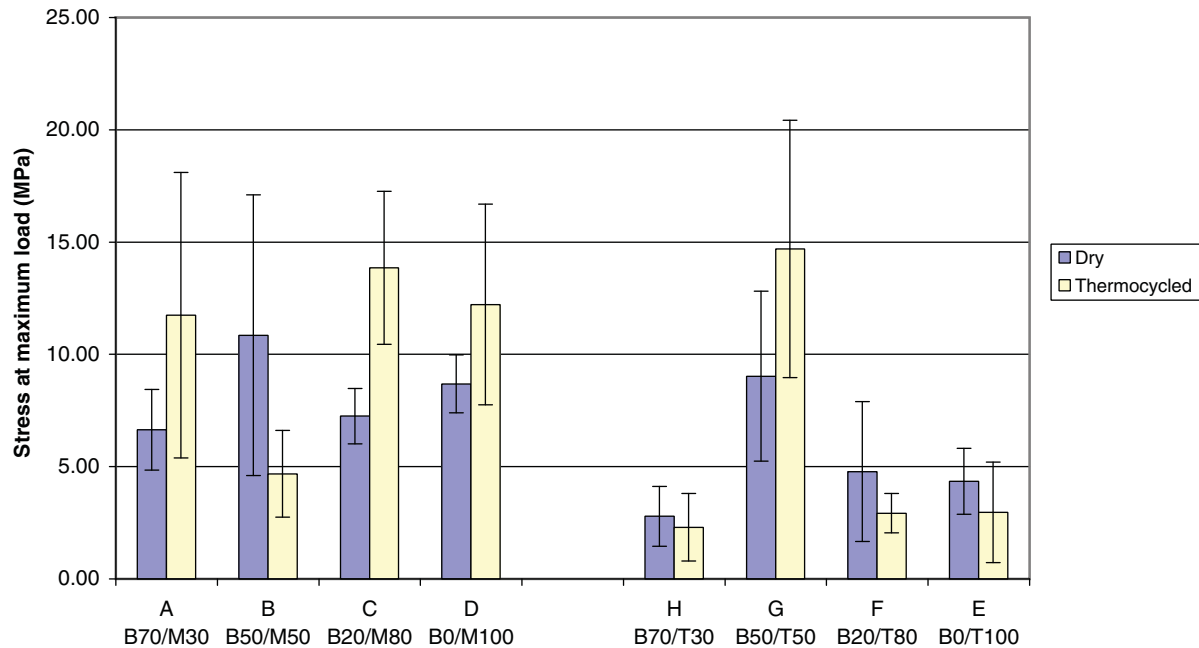


Figure 1. Mean shear bond strengths of different groups. Vertical lines indicate standard deviations.

composite resin. The treatment time for control IMR (B0/M100) was 60 min. The test specimens were wet ground to the thickness of 0.15 mm with 4000 (FEPA) grit silicon carbide grinding paper and rinsed with water before light microscope examination in translucent light.

## Results

The viscosities of the different IMRs are presented in Table II. The result of the viscosity measurement showed that, with the BisGMA-MMA resins, the viscosities were lower when compared to the corresponding values of the BisGMA-TEGDMA resins (Table II). The bond strengths were generally higher in the BisGMA-MMA groups than in the BisGMA-TEGDMA groups, except the B50/T50 group

(Figure 1). In the BisGMA-MMA group the highest shear bond strength was achieved with the B20/M80 combination when the test specimen were thermocycled ( $13.9 \pm 3.4$  MPa) (Figure 1). The lowest mean shear bond strength was measured with the B80/M20 group without thermocycling fatigue ( $6.6 \pm 1.8$  MPa). Thermocycling increased the bond strengths in three BisGMA-MMA groups but slightly decreased it in the B50/M50 group (Figure 1). In the BisGMA-TEGDMA groups the highest bond strength was achieved with the thermocycled B50/T50 group ( $11.8 \pm 5.3$  MPa) and the lowest bond strength with the thermocycled B70/T30 group ( $2.3 \pm 1.5$  MPa). In the BisGMA-TEGDMA groups the thermocycling decreased the bond strength in three groups but increased it in the B50/T50 group (Figure 1).

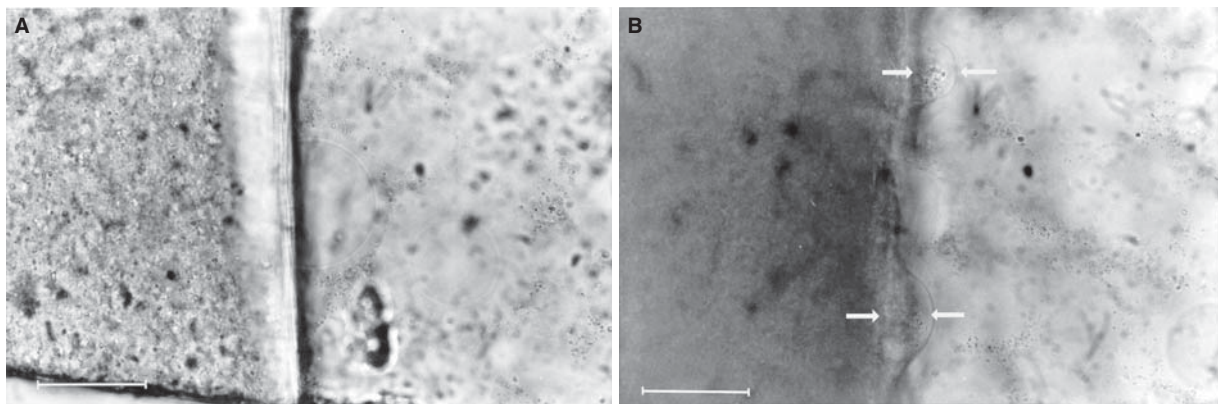


Figure 2. Micrographs of interphase of the used substrate and the polymerized new resin. The 3 min treatment time with all eight IMRs did not show any signs of swelling in the polymer substrate (A). Diffusion of monomers of the new resin into the polymer substrate was noticed after 60 min treatment with B0/M100 IMR (B). The white arrows indicate the swelled interphase by monomer diffusion. Magnification = 40. Bar = 50  $\mu$ m.

Table III. The effect of the intermediate resin (IMR) and thermocycling (TC) on shear bond of the polymer substrate to new resin compared by two-way ANOVA.

Source	Type III sum of squares	df	Mean square	F	Sig.
IMR	808.957	7	115.565	8.234	< 0.001
TC	83.845	1	83.845	5.974	0.017
IMR*TC	161.080	7	23.011	1.639	0.140

Significant differences were found between the shear bond strength values of the IMRs ( $p < 0.001$ ). Two-way ANOVA revealed significant effects of type of IMR ( $p < 0.001$ ) and thermocycling ( $p = 0.017$ ) on the shear bond strength. No interaction between these variables was found ( $p > 0.05$ ) (Table III). Homogeneous sub-sets of mean shear bond strengths of IMRs are presented in Table IV.

When 3 min treatment time was used, the light microscope examination of the adhesive interphase showed no visible swelling of the linear phases of the substrate caused by different monomer combinations or ratios (Figure 2A). On the other hand, the control IMR (100% MMA) caused swelling of the thermoplastic PMMA beads of the substrate when the treatment time was increased to 60 min (Figure 2B).

## Discussion

This study demonstrated the effect of two IMR compositions with different monomer ratios and viscosities on the shear bond strength of new resin to polymer substrate. The multiphase acrylic resin was chosen as the polymer substrate because it should allow the bonding based on the interdiffusion of the monomers of the new resin, as has been previously reported [2,4,11,12]. The results of this study support the earlier findings that high shear bond strengths were related to type of IMR [4,13]. On the other hand, it needs to be emphasized that the substrate in this study was PMMA based acrylic, which is basically not a cross-linked polymer, in contrast to cross-linked composite resin which has been extensively studied in terms of composite-composite bond strength [17–19]. Studies have demonstrated that mechanical roughening and additional treatment by tribochemical silica coating considerably enhance bonding properties. This reflects poor dissolving capacity of the cross-linked polymer matrix by the monomers of new resins.

It was also found in the present study that the monomer ratio had an influence on the bond strength, especially when the monomers of the IMR were bifunctional. The highest bond strength results may also have clinical relevance. The result of the viscosity measurement showed that with the BisGMA-MMA resins the viscosities were lower when compared to the corresponding values of the BisGMA-TEGDMA resins (Table II). If the values are compared to the

viscosity of water (1 cP at 20°C) it can be noted that the viscosities of the B20/M80, B0/M100 and B0/T100 IMRs were near the viscosity value of water. Only the B70/T30 IMR was found to be impractical because of its very high viscosity.

According to the results, the proportion of the monomers did not have much influence on the mean shear bond strengths in the B/M group. However, in the B/T group, the B50/T50 IMR gave much higher bond strength than the other three IMRs of the B/T group. One reason for this can be a favorable solubility parameter of the B50/T50 IMR, which can be near to the solubility parameter of linear PMMA phases of the polymer substrate. The solubility parameter is a measure of the ability of the liquid to solubilize and soften solid surfaces. A softening occurs when the liquid and the solid have solubility parameters that are not too far apart and when the liquid and the solid have about the same polarity [6,15,20]. According to this fact, it can be assumed that one reason for different bond strength values of B50/T50 IMR might be the right solubility and polarity parameter which allow the swelling of the linear phase of the substrate and further on a semi-IPN bonding between the polymer substrate and new resin. However, it must be emphasized that the same solubility and polarity parameters of the substrate and solvent (IMR) do not mean that swelling of the substrate occurs. Because a cross-linked three-dimensional polymer can be dissolved very poorly or cannot be dissolved at all, it is likely that the B50/

Table IV. Homogeneous sub-sets of mean shear bond strengths of the groups according to the monomer composition and ratios of the intermediate resins (IMRs). Comparison made by Tukey's post-hoc analysis with a significance level of 0.05.

Intermediate resin (IMR)	n	Mean (SD) MPa
B70/T30	10	2.5 <sup>a</sup> (1.3)
B20/T80	10	3.0 <sup>a</sup> (1.6)
B0/T100	10	4.5 <sup>ab</sup> (2.3)
B70/M30	10	9.2 <sup>bc</sup> (5.2)
B50/T50	10	9.4 <sup>bc</sup> (5.3)
B0/M100	10	9.4 <sup>bc</sup> (3.1)
B50/M50	10	10.2 <sup>c</sup> (6.0)
B20/M80	10	10.6 <sup>c</sup> (4.2)

Superscript font indicates the homogeneous subsets of mean values. For codes of different groups, see Table II.

T50 IMR has swollen only the linear PMMA phases of the used substrate. If the solubility parameter and polarity parameter of the polymer substrate is known, it can be calculated what proportion of certain monomer or monomers gives the parameter values near the corresponding values of the substrate. This method has been reported to be practical help for strong adhesive bonding to dentin [21]. However, if the substrate is multiphase polymer such as autopolymerizing acrylic resin consisting cross-linked and linear phases, the calculation of the solubility and polarity parameters can be difficult because the different phases are not evenly distributed and, thus, the parameters vary in different parts of the polymer. Therefore, experimental studies are needed to define the effect of IMRs with different compositions and proportions on bond strength between the multiphase polymer substrate and the new resin.

According to statistical analysis it was found that thermocycling had an effect on the mean shear bond strengths (Table III). When BisGMA-MMA monomers were used, the thermocycling increased the shear bond strengths in three cases but slightly decreased the bond strength in the B50/M50 group. In the BisGMA-TEGDMA group the shear bond strength generally decreased after thermocycling, whereas in the B50/T50 IMR thermocycling increased the bond strength. Statistically, high standard deviations of the B50/M50 and B50/T50 groups explain the differences from the general trend. High standard deviations might be related to techniques to fabricate and test the specimens. The shear bond test has been a routine procedure for the determination of bonding effect. However, it has been reported that the shear bond test produced significantly more failures within dentin and composite than the micro tensile method [21]. On the other hand, it has also been stated that either the tensile or the shear test method could be used for quality testing of dentin adhesive [22]. Although the shear bond test has been criticized, the simplicity of the shear bond test has been its advantage [23,24]. However, the explanations mentioned above do not explain the thermocycling general effects in both groups.

In a previous study it was reported that IMR with bifunctional BisGMA and monofunctional HEMA monomers resulted in higher shear bond strengths after thermocycling when compared to values where IMR with bifunctional monomers BisGMA and TEGDMA was used [4]. One explanation was that, after being plasticized by water, the less rigid adhesive joint of the BisGMA-HEMA IMR might work as a stress breaker between the two different materials such as autopolymerized acrylic polymer and particulate filler composite [4]. This can also explain the results of the present study. The increased bond strength values after thermocycling could be explained by a less cross-linked structure, i.e. a structure of lower

modulus of elasticity, of the BisGMA-MMA resins than of the BisGMA-TEGDMA resins.

An interesting finding was the fact that the amount of MMA in BisGMA-MMA IMRs did not seem to have a significant effect on bond strength. One reason could be the used 3 min influence time of the IMR. In a previous study, the effect of monomer liquid treatment (MMA) on the surface of the autopolymerized PMMA-based polymer substrates was clearly seen after 30 min treatment [6]. In that study, no significant differences were found between 0 min, 0.5 min and 3 min treatment times. In another previous study, 5 min treatment time with BisGMA-HEMA and BisGMA-TEGDMA resulted in good bond strengths between the fiber-reinforced polymer substrate and the new composite resin [4]. Our study was based on 3 min treatment time, which could be the reason for the relatively weak bond strengths and for absence of swollen layers at the adhesive joint (Figure 2A). When the micrograph of the control group was studied it was noticed that the monomers of the IMR had diffused to the polymer substrate (Figure 2B). The finding that microscopic changes were found when the polymer substrate was treated with the B0/M100 IMR for 60 min supports the importance of the treatment time on shear bond strength (Figure 2B). This may indicate that the 3 min treatment time was insufficient for formation of durable bonding based on the interdiffusion of the monomers of the new resin. On the other hand it must be emphasized that longer IMR treatment times are not necessarily practical in the dental office but might be used in a dental laboratory. However, further investigations are needed to determine the effect of the treatment time of the IMR with dissolving capability of the linear phases on the shear bond strength.

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