

## EFFECT OF BLEACHING GELS ON THE ELUTION OF MONOMERS FROM METHACRYLATE BASED NEWER COMPOSITES-AN IN VITRO STUDY

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### ABSTRACT

The aim of this study was to evaluate the amount of elution of BISGMA and UDMA from nanocomposites following bleaching with 15% and 35% bleaching gel over a period of 24 hours and 7 days. Filtek Z250XT (3M, ESPE;India) and IPS Empress (Ivoclar Vivadent; Schaan) were treated with an Opalescence PF15 % (Ultradent Products, Inc.; South Jordan) for 5 hours and Opalescence PF35% (Ultradent Products, Inc.; South Jordan) for 30 min. For each of the two composite resins, 10 samples were prepared and immersed in 2 ml of 99.99% ethanol (Hayman limited, Eastways Park, Witham, Essex, CM88YE, and England) and were stored at room temperature. The medium was replenished after 24 hours and 7 days. The composite blocks were measured using reverse phase High Performance Liquid Chromatography (SHIMADZU, Model SPD 20A, Shimadzu Corporation, and Kyoto, Japan.) unit. One-way ANOVA, Post hoc Test, paired 't' test and student 't' test were used for statistical analysis of the acquired data. The results of the study exhibited that at 24hrs, the mean value of the elution of BISGMA and UDMA from Filtek Z250XT was higher compared to that of IPS Empress (i.e.  $P<0.001$ ). The mean values increased significantly from 24 hours to 7 days ( $p<0.001$ ) for all the groups. On comparing the elution of BISGMA (Bis[4-(2-Hydroxy Methacryloxypropoxy) Phenyl]Propane) and UDMA(Urethane Dimethacrylate), significantly higher amount of UDMA was eluted at 24 hours and at the end of 1-week (i.e.  $P<0.001$ ). Opalescence PF15% eluted higher amount of monomers than 35% at 24 hours.

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## Introduction

Harmonizing an esthetic smile requires a perfect integration of the facial and dental composition. The current trend towards cosmetic dentistry has generated more interest in bleaching. In general, bleaching is a conservative treatment and has been shown to be both efficient and safe. <sup>(1)</sup>

Bleaching techniques are based on hydrogen peroxide or carbamide peroxide gel namely, (1) home bleaching methods containing 15% peroxides; (2) in-office-bleaching methods containing 35% peroxides; (3) over-the-counter products containing maximum 10% peroxides and (4) chairside-bleaching methods containing 38% peroxides. <sup>(2)</sup>

The mechanism of bleaching is a complex phenomenon. Carbamide peroxide and hydrogen peroxide deliver oxidizing agents, which tend to degrade the extracellular matrix of the enamel & dentin and additionally causing the oxidation of chromophores situated in the enamel and/or dentin. However, a few studies are available regarding its interaction with the 3-D polymer network of methacrylate based composite resin restorations. <sup>(3,4)</sup>

Control of contour during restoration, increased colour stability, increased polymerization and rapid setting compared to chemically activated materials is some of the primary advantages of composites. However, inadequate polymerization, which results because of the high amount of residual monomers, is one of the most common drawbacks. <sup>(5)</sup>

Various factors are known to be responsible for the elution of monomers from resin composites. Ferracane attributed that the released components are directly related to the degree of double bond conversion. Also, it depends upon the chemistry and size of the released monomers. Enhanced mobility is exhibited by small monomers, which elute at a faster rate as compared to larger, bulkier molecules. <sup>(6)</sup>

Unpolymerized monomers are known to cause various detrimental biological reactions, such as systemic toxicity, local toxicity, pulp reactions, allergies and estrogenic responses. According to proven studies, the cytotoxicity positioning of the monomers is as follows:

BISGMA>UDMA>TEGDMA>>HEMA. <sup>(7)</sup>

The distinction in the chemistry of the composites and the variation in the filler proportion and monomer

significantly affect the materials component discharge and level of cytotoxicity. <sup>(8)</sup>

Ferracane and Condon advocated immersion of specimens in ethanol to mimic the clinical situation. This was attributed to the capacity of ethanol to promote the release of entangled monomers from the set composite materials. Hence, in the present study, 99.99% ethanol was utilized as a solvent to immerse the samples.

In the present study, HPLC is used for the analysis of monomer elution as it is a significant and prevalent method for investigation, not restricted by the volatility or stability of the sample compound. HPLC is preferred to gas chromatography since it gives a greater level of control over the separation procedure, as monomers are soluble in the mobile phase.

The degree of the monomer-polymer conversion of dental resin composites varies between approximately 35% and 77%. <sup>(5)</sup>

Limited information exists regarding the bleaching effect on the monomer elution of resin composites. Since bleaching materials have an influence on the physicochemical properties, this could also influence the elution of monomers. Bleaching treatment influences the three-dimensional polymer network of dental resin composites. <sup>(8)</sup>

Therefore the need of this study was to find out whether different bleaching gels affect the time-related elution of components from various dental resin composites.

## Materials and Methods

The present study was conducted at Department Of Conservative Dentistry and Endodontics,

A.B. Shetty Memorial Institute of Dental Sciences, Mangaluru in association with the

Department of Pharmacology, NGSIM Institute of Pharmaceutical Sciences, Paneer Deralakatte.

## Sample Preparation

Two nanohybrid dimethacrylate composites: Filtek Z250XT (3M, ESPE;India) and IPS Empress (Ivoclar Vivadent; Schaan) were used in the present study and further sub-divided into:

**GROUP 1a:** Filtek Z250XT samples bleached with Opalescence PF15% for 5 hrs.

**GROUP 1b:** Filtek Z250XT samples bleached with Opalescence PF 35% for 30 min

**GROUP 1c:** Filtek Z250XT Control group

**GROUP 2a:** IPS Empress samples bleached with Opalescence PF15% for 5 hrs.

**GROUP 2b:** IPS Empress samples bleached with Opalescence PF 35% for 30 min

**GROUP 2c:** IPS Empress Control group

10 samples each were prepared (N=10) and then treated with bleaching gels Opalescence PF15%(Ultradent Products, Inc.;South Jordan) for 5 hours and Opalescence PF 35% (Ultradent Products, Inc.;South Jordan) for 30 min.

1. A standard Teflon mould of dimension 2x2x2 mm was used to prepare the samples. A glass plate was used to position the mould on a transparent plastic matrix strip to avoid oxidation of the superficial layer. Each sample was cured using ‘LED’ curing unit (Bluedent LED smart, BG light, Bulgaria LTD). Using a 1cm glass plate the distance was standardized between the light source and the sample. After curing with the LED, the specimens were then bleached and immersed in 2 ml of 99.99% ethanol (Hayman limited, Eastways Park, Witham, Essex, CM88YE, and England). These samples were immediately stored at room temperature for a time period of 24 hours. The storage medium was replenished after 24 hours and then stored for 7 days. The composite blocks were then removed from the storage medium (99.99% ethanol) after 24hrs and 7 days, and were thus prepared for measurements

### Sample Evaluation

After 24 hours and 7 days the samples were measured using a reverse phase HPLC unit (SHIMADZU, Model SPD 20A, Shimadzu Corporation, and Kyoto, Japan.) was used for the detection of monomer release. The separation of monomers took place with a CC125/4 Nucleodur 100-5 C18ec HPLC-Column. The mobile phase was acetonitrile/water (75/25% v/v) at a flow rate of 1 mL/min and detection performed at a wavelength of 254 nm for 30 minutes. For the analysis of extracted residual monomers a reference standard of BISGMA (CAS No. 261548, Sigma Aldrich Chemical Co., USA) and UDMA (CAS No. 72869, Sigma Aldrich Chemical Co., USA) were purchased and stock solutions were prepared by appropriate quantitative dilution. 20µl from the solution was loaded into the HPLC system and

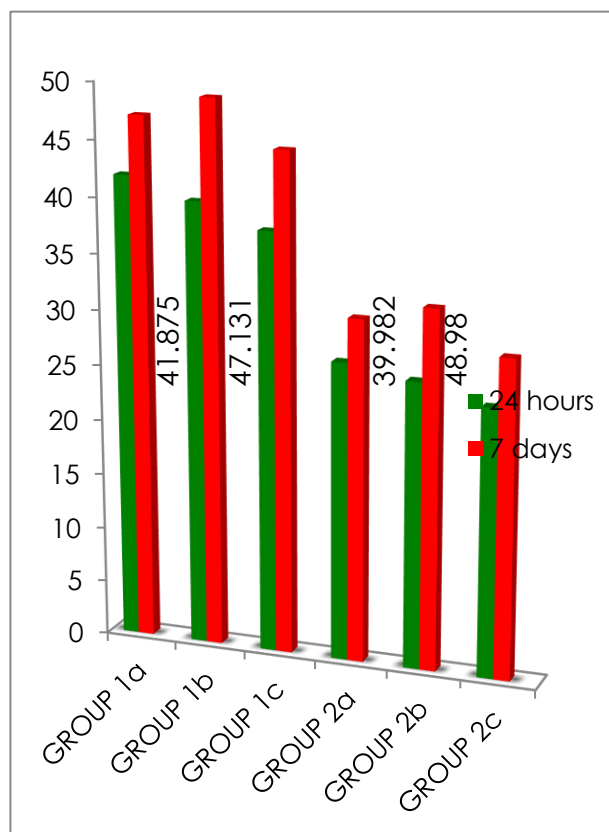
standard chromatograms were obtained for both the monomers individually.

### Statistical Analysis

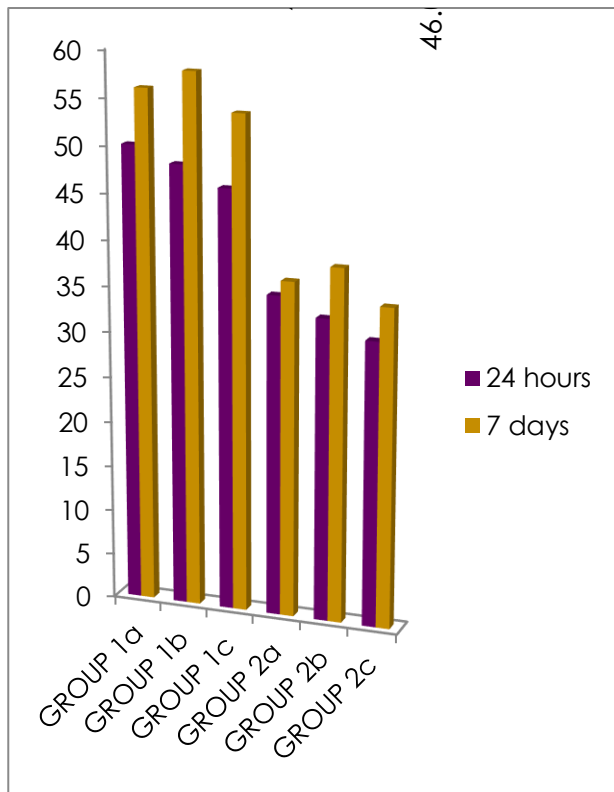
The results were tabulated under each group and were statistically analyzed using One-way ANOVA, Post hoc Test (Tukey’s HSD test), paired ‘t’ test and student ‘t’ test based on the data. SPSS (Statistical Package for Social Science) software version 15 was used. Level of significance was set at  $p < 0.05$

### Results

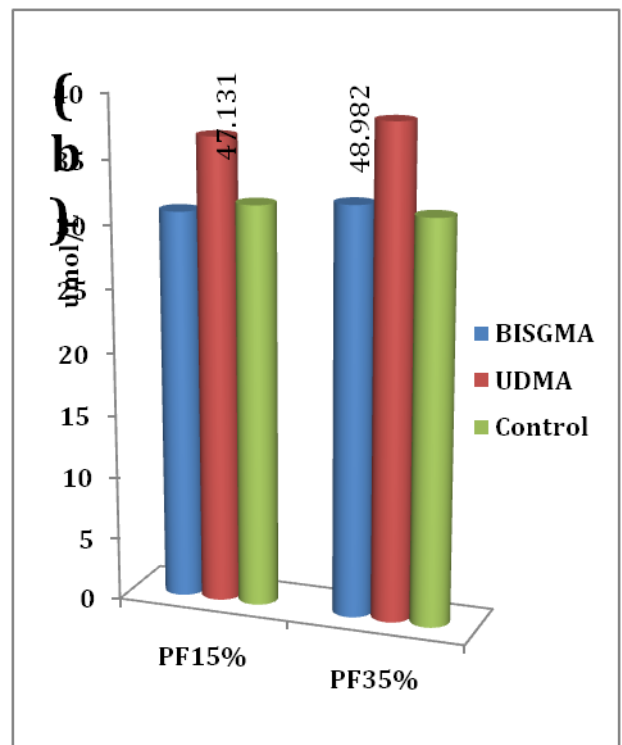
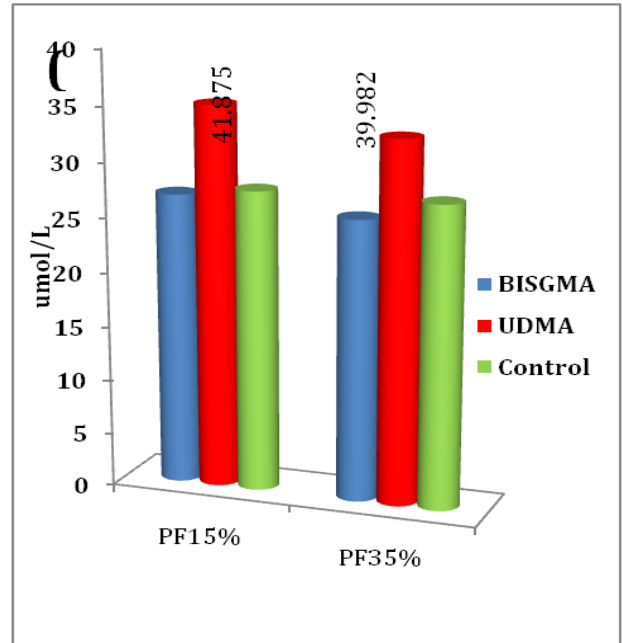
**GRAPH 1: Bar graph depicting the mean amount of eluted BISGMA when bleached with Opalescence PF 15% and 35% based on different storage period.**



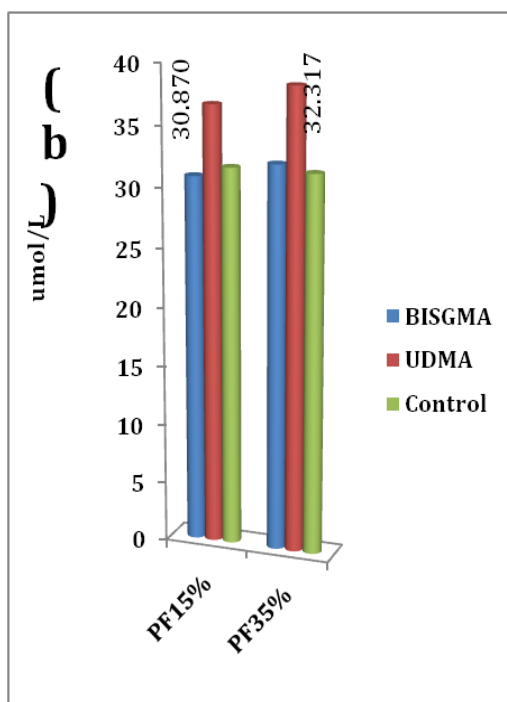
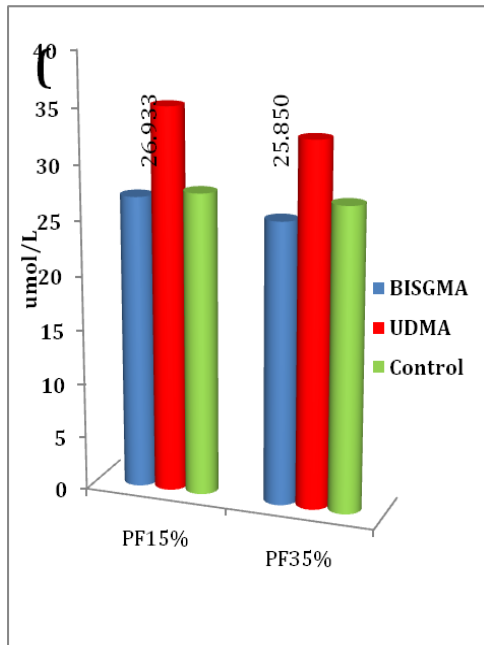
**GRAPH 2:** Bar graph depicting the mean amount of eluted UDMA when bleached with Opalescence PF 15% and 35% based on different storage period.



**GRAPH 3:** Bar graph depicting the mean amount of eluted monomers from Filtek Z250 XT when bleached with Opalescence PF 15% and 35% for (a) 24hrs and (b) 7 days.



**GRAPH 4: Bar graph depicting the mean amount of eluted monomers from IPS Empress Direct when bleached with Opalescence PF 15% and 35% for (a) 24hrs and (b) 7 days.**



### Discussion

The results obtained in the present study revealed increased elution of monomers after bleaching and a significantly larger amount of UDMA elution compared to BISGMA after 24 hours and 7 days. The elution of monomers with Opalescence PF 15% was higher at 24 hrs compared to 35%. At both time periods of 24 hours and 7 days highest elution of BISGMA and UDMA was seen in Filtek Z250XT composite resins.

Ferracane advocated the increased elution of monomers to be due to the low values of double bond conversion, which results due to the combination of high viscosity and high glass transition temperature. Hence resins with a lower degree conversion, result in the increased monomer release due to increased free monomers. <sup>(3)</sup>

The cytotoxicity of aromatic BISGMA is more than the aliphatic monomer UDMA. The BISGMA molecule, having a firm Bisphenol-A core and two pendant hydroxyl groups which are able to form strong hydrogen bonds, is the biggest and has the most reduced concentration of double bonds. <sup>(2,5,7)</sup> However, the UDMA molecule, with its flexible aliphatic core and two urethane links, is additionally ready to form hydrogen bonds, is smaller in size and therefore has an increased concentration of double bonds. <sup>(2,7)</sup>

### Elution of Monomers After Bleaching

The outcome of the present study demonstrates that in every single tested specimens, treated with Opalescence PF 15% for home bleaching or 35% for in-office bleaching, increased elution was seen in the bleached samples (graph 1,2), compared to the control samples ( $p < 0.01$ ) <sup>(4,13)</sup>

The increased elution of monomers after bleaching may be attributed to; the oxidative cleavage of the surface of the 3-D polymer network and the degree of conversion of monomers and additives during polymerization. <sup>(3,4,16)</sup>

The bleaching effect of carbamide peroxide is advocated to be due to its breakdown into urea and hydrogen peroxide. Eventually the hydrogen peroxide breaks into free radicals leading to bleaching. <sup>(10)</sup>

Superoxide molecules ( $O_2^-$ ), hydroxyl ions ( $OH^-$ ), perhydroxyl ions ( $HOO^-$ ), hydroxyl radicals ( $HO^\bullet$ ) or hydroperoxy radicals ( $HOO^\bullet$ ) formed from hydrogen peroxide are reactive species which may respond and oxidize the C-C single or double bonds and in addition, with ester bonds of the 3-D polymer network on the surface of the polymerized composite resin. This causes the bond strength of enamel and dentin to change. <sup>(4)</sup>

Hence the bleached specimens elute more monomers in comparison to unbleached specimens. <sup>(4)</sup>

The present study is in contrast to the study by Polydorou et al. who concluded that the bleaching of composite resin had no impact on the monomer release. Nonetheless, Polydorou et al. expelled the bleaching gel under the continuous water flow and stored in 75vol% ethanol. Thus, by this expelling process the monomers were diluted and washed out. <sup>(4)</sup>

#### **Elution of Bisgma and Udma Increases Gradually**

Yap et al in his investigation demonstrated that hydrolysis process be the main reason for monomer release after 24 hours. <sup>(7,14)</sup>

The paired 't' test applied to demonstrate the mean values for both the monomers to increase significantly at 7<sup>th</sup> day ( $p<0.01$ ). The more concentrated carbamide peroxide and increased immersion duration caused more elution of BISGMA (graph 1) and UDMA (graph 2) which could be one of the possibilities and is in accordance with Olga Polydorou et al. <sup>(3,11,15)</sup>

#### **Increased Elution of Udma than Bisgma**

The fundamental concern is the amount of monomer leaching and the time required for the entire elution. These parameters are influenced by the measure of unreacted monomers, controlled by the chemistry of the monomer and the polymerization conditions; the solvent utilized for the elution and the elution conditions; and the size and the chemistry of the monomers. It is known that small molecules like TEGDMA and UDMA have improved mobility and will be eluted at a quicker rate than bigger, bulkier molecules like BISGMA. <sup>(3,17)</sup>

In the present investigation, the outcome demonstrates that the bulky and rigid BISGMA, with the solid capacity to form hydrogen bonds through its hydroxyl groups, is eluted more gradually than the small and flexible UDMA from their copolymers at  $p<0.05$  (graph 3,4) <sup>(3)</sup>

This perception was in concurrence with Izabela M, Jürgen Durner et al, Hegde at al, Olga Polydorou et al, Tabatabaee et al. and Nathanson et al who stated in their investigation that smaller molecules are discharged faster and more than the larger ones on account of their mobility and hydrophilic nature. <sup>(11,18)</sup>

#### **Elution from Opalescence Pf 15% and 35%**

The findings from the present investigation propose that the subjected time of the bleaching gels (15%: 5 h, 35%: 30 min), irrespective of the distinctive concentrations of peroxides in the bleaching gels, caused an increased release of UDMA and BISGMA from the composite resins, subsequent to bleaching (graph 3,4) which is as per the outcomes acquired by Lena Schuster et al, Durner et al, and Jürgen Durner et al. <sup>(2,20)</sup>

This increased elution may be attributed to the longer duration of the contract (5 h of treatment with Opalescence PF 15% and 0.5 h of treatment with 35%) of the hydrogen peroxide with the 3-D polymer system of the samples. This can be interpreted as a positive connection between the presence of  $H_2O_2$  and probability of monomer discharge. <sup>(4,11)</sup>

#### **Elution from Filtek Z250 Xt and IPS Empress Direct**

The physical and mechanical properties of the composites are influenced by the filler content, the filler size, and the distribution of the filler particles. As indicated by Salazar et al, the filler content is as follows: IPS Empress Direct =72.7% and FILTEK Z250 XT =82%. It has been demonstrated that the filler volume fraction and filler load level of the composite resins correspond to the material strength and elastic modulus, and additionally the fracture toughness of the material. <sup>(12,19)</sup>

The outcomes from the present investigation reveal that the mean estimations of the release of BISGMA and UDMA at 24hrs and 7 days were higher in Filtek Z250 XT (graph 3) compared to IPS Empress Direct (graph 4) and were statistically significant ( $p<0.01$ ) <sup>(12)</sup>

Only qualitative outcomes on the monomers elution from Filtek Z250 XT after bleaching have been reported in the literature. An interesting outcome of the investigation done by Irini. D. et al states that the UDMA release, from all Filtek Z250 XT samples, are considerably higher than the alternate monomers; concluding, the resin matrix of this composite contains higher measures of UDMA than BISGMA and BISEMA. <sup>(3,20,22)</sup>

As indicated by Santos et al, water accumulated at the aggregated zirconium/silica cluster filler-organic matrix interface in the Nano composite, can make way for water dispersion towards the inside of aggregates, where microvoids are most likely present. Some portion of the absorbed water in composites diffuses through the network and is caught in polymer nanovoids. Accordingly the aggregate void volume in the polymer network dictates the measure of ingested water. <sup>(11,21)</sup>

At first, water sorption causes polymer softening because of reducing the frictional forces between the polymer chains. After relaxation process, the unreacted monomers trapped in the polymer network discharge at a rate controlled by the polymers swelling and relaxation capacity. It is certain that the more water absorbed, the more components leach out of resin composites. This phenomenon clarifies the increased release of monomers from Filtek Z250 XT in this study. <sup>(11)</sup>

### Limitations:

The limitations of the present study would be the usage of 99.9% ethanol as a solvent to immerse the cured composite samples, as it did not simulate the environment of the oral cavity. Further clinical studies need to be conducted for the same.

### Conclusion

The present study exhibited the concentration and the contact time of hydrogen peroxide to have a vital impact on the amount of the elution products and 3-D polymer system of the composite resins.

The outcomes from the present investigation propose that bleaching prompts a higher elution of monomers, additives & unspecific oxidation products from composites.

The higher concentration of carbamide peroxide and the more immersion time in our study caused more monomer release. Since bleaching can both soften and roughen the composite surface, it is profoundly possible that it influences monomer release from composite restorations and thus affects the biocompatibility of the resins.

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