

Detection of HEMA in Self-etching Adhesive Systems Using High Performance Liquid Chromatography

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One of the factors that can decrease hydrolytic stability of self-etching adhesive systems (SEAS) is 2-hydroxyethylmethacrylate (HEMA). Due to hydrolytic instability of acidic methacrylate monomers in SEAS, HEMA can be present even if the manufacturer did not include it in original composition. The aim of the study was to determine the presence of HEMA because of decomposition by hydrolysis of methacrylates during storage, resulting with loss of adhesion strength to hard dental tissues of the tooth crown.

Three most commonly used SEAS were tested: AdheSE ONE, G-bond and iBond under different storage conditions. High performance liquid chromatography (HPLC) analysis was performed on a Nucleosil C₁₈-100 5 μm (250x4.6 mm) column, Knauer K-501 pumps and Wellchrom DAD K-2700 detector at 215 nm. Data were collected and processed by EuroCrom 2000 HPLC software. Samples of adhesives (~70 mg) were dissolved in 2.0 ml of methanol and filtered; 20 μl were injected and isocratically eluted with 20% aqueous methanol for 15 min, followed by gradient to 100% methanol in 5 min. Calibration curves were made related eluted peak area to known concentrations of HEMA (purchased from Fluka). The elution time for HEMA is 12.25 min at flow rate 1.0 ml/min.

Table 1: Tested materials and the HEMA concentration in the study.

Adhesive	Storage	HEMA content (%)
G-bond	New bottle	0.14%
G-bond	Opened bottle, 2 months rt, 3 months 4 °C	0.15%
G-bond	Opened bottle, 5 months rt	0.22%
G-bond	Unopened bottle, 3 months 38 °C	0.31%
iBond	New bottle	0.07%
iBond	Opened bottle, 5 months rt	0.13% (partially polymerized or dried)
iBond	Unopened bottle, 3 months 38 °C	(completely dried or polymerized)

rt - room temperature

Obtained results, shown in table 1 indicate that no HEMA was present in AdheSE ONE because methacrylates are substituted with methacrylamides that seem to be more stable under acidic aqueous conditions. In all other adhesive systems HEMA was detected.