

DENTAL ADHESIVE COMPOSITIONS AND METHODS

RELATED APPLICATION

This application of a division of U.S. patent application Ser. No. 12/653,251 filed on Dec. 10, 2009, now Pat. No. 8,022,114, which is a continuation of U.S. patent application Ser. No. 12/284,817 filed on Sep. 25, 2008, now abandoned, which is a continuation of U.S. patent application Ser. No. 11/899,124 filed on Sep. 4, 2007, now abandoned, which is a continuation of U.S. patent application Ser. No. 10/844,628 filed on May 13, 2004, which claims the benefit of Provisional Application No. 60/469,938 filed on May 13, 2003.

TECHNICAL FIELD

The present invention generally relates to dental adhesives. More particularly the invention relates to self-etching adhesives. Specifically the invention relates to adhesives containing polymerizable acidic monomers.

BACKGROUND OF THE INVENTION

Table of Abbreviations Used Herein

Abbreviation	Full term
4-META	4-methacryloxyethyltrimellitic Anhydride
AHPMA	3-(Acryloyloxy)2-hydroxypropyl methacrylate
BHT	Butylated Hydroxytoluene
BMAP	Bis(2-methacryloxyethyl) phosphate
CAF	Cetylamine Hydrofluoride
CQ	Camphorquinone
DHEPT	Dihydroxyethyl-p-toluidine
DMABA	Dimethylaminobenzoic acid
DMABN	Dimethylaminobenzonitrile
EDAB	4-ethyl-dimethylaminobenzoate
EGMP	Ethylene glycol methacrylate phosphate
HEMA	2-Hydroxyethyl methacrylate
L-TPO	Diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide
NaTs	Sodium salt of p-toluenesulfonic acid
OEMA	4,4'-Oxydiphenylether 1,1',6,6'-tetracarboxylic acid-1,1'-(2-methacryloxy) dimethacrylate
PENTA	Dipentaerythritol pentaacrylate phosphoric acid ester
PyroEMA	Tetra-methacryloxyethyl Pyrophosphate
SBS	Shear Bond Strength
SCA	Self-cure activator
SEA	Self-etching adhesive
2P-SEA	Two-Part Self-etching adhesive
1P-SEA	One-Part Self-etching adhesive
VLC	Visible Light Cure
SC	Self Cure (or anticure)
SUM	summation
TEGDMA	Triethyleneglycoldimethacrylate
TMPTMA	Trimethylolpropane Trimethacrylate
UDMA	1,6-Bis[methacryloyloxyethoxycarbonylamino]-2,4,4-tromethylhexane

As used herein all “%” and percents or the like are by weight.

With the use of composite resins as dental restorative materials, it is required to ensure firm adhesion between tooth structures and composite resins by a simple handling. A representative adhesive restoration procedure includes an acid etching on tooth substrate by phosphoric acid, followed by water rinsing, drying, application of a primer, drying, application of a bonding agent, light-curing and filially filling of a composite resin. It is apparent that it takes time in many bonding steps to accomplish such handling and that no adhesion with confidence is attained.

In order to reduce the number of constituents to be used, priming and bonding were combined in one-bottle, so-called one-bottle/two-step, exemplified by Prime & Bond® brand adhesive (Dentsply). Etching must still be carried out first, followed by the application of the single-bottle bonding at least once and then polymerization, before the filling materials is used. Another simplification of the process for the adhesive securing of filling materials is to combine priming and etching into one-component, so-called self-etching primers, such as SE Primer in ClearFil SE Bond system [a 2-component (SE Primer and Bond liquid), 2-step, sequentially applied (SE Primer, followed by Bond resin) self-etching adhesive by Kuraray]. ClearFil SE Bond is indicated for direct light cured composite restoration bonding only. For indirect restoration bonding, Kuraray recommends using ClearFil Liner Bond 2V that is a multi-component (Primers A and B, Bond Liquid A and B)/multi-steps application self etching adhesive system.

Adper Prompt L-Pop (3M ESPE), so-called 2-component/one-pack/one-step self-etching and self-priming adhesive, is supplied in a Single Unit Dose blister package that consists of two-predosed compartments, or in two separate bottles for the two liquids A and B. Prompt L-Pop is indicated for direct light cured composite restoration bonding only.

Ref. U.S. Pat. No. 6,387,979 by K. Hino (Kuraray Co. Ltd., Japan), issued May 14, 2002 describes a tooth treated with a bonding composition with high initial bonding strength and good bonding durability comprising a mixture of polymerizable compound having an acid group, a water-soluble film-forming agent, water, and a curing agent, in which the calcium salt of the acid is insoluble in water, and the film-forming agent is a polymerizable compound miscible with a physiological saline solution, does not require any pre-treatment such as acid-etching or priming treatment. It is stated that the active ingredients of the composition in a single package may degrade or polymerize while stored. To prevent this, the constituent ingredients of the composition may be divided into two or more parts. The plural parts are separately packaged and stored in different packages. For their use, the plural parts taken out of the individual packages may be applied to one and the same object in sequence; or they may be blended into one mixture just before use.

PREFERRED EMBODIMENTS FOR CARRYING OUT THE INVENTION

In a preferred embodiment of the present invention, a dental adhesive comprises:

- (i) from about 5 to about 70% by weight of polymerizable acids components selected from the group consisting of PENTA, OEMA and mixtures thereof;
- (ii) from about 1 to about 30% by weight of hydrophilic methacrylate;
- (iii) from about 1 to about 25% by weight of hydrophilic difunctional (meth)acrylate;
- (iv) from about 1 to about 35% by weight of Urethane methacrylate;
- (v) from about 1 to about 30% by weight of hydrophobic difunctional (meth)acrylate;
- (vi) from about 0.1 to about 5% photoinitiator of (phosphine oxide, and/or CQ/co-initiator selected from the group consisting of Lucerin TPO, CQ/EDAB and CQ/DMABN);
- (vii) from about 0.1 to about 5% curing additives selected from the group consisting of aromatic sulfinate salts;