

## COMPOSITE RESIN IN THE LAST 10 YEARS - LITERATURE REVIEW. PART 1: CHEMICAL COMPOSITION

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### ABSTRACT

**Introduction:** This is the first article of a series of six manuscripts about composite resins. Composite resins composition influence on their physico-chemical properties and increased survive rates of their clinical application in dentistry. **Objectives:** The purpose of this review was to perform a literature review about the main components found in composite resins and their clinical influence in the restorative process. **Methods:** A search using several terms was performed on the PubMed database and, after reading the title and abstract, 50 articles were selected. These were read in full and 30 were used in this review. There was also a search in the references of the selected articles. The manufacturer's technical profile was analyzed for 23 different composite resins. **Results:** The most of the composite resins are composed of diacrylate monomers (Bis-GMA, UDMA, TEGDMA); zirconium, silica and barium-based are the most cited inorganic particles; and camphorquinone was the most used photoinitiator. **Conclusion:** The improvements involving components such as organic matrix, inorganic matrix, bonding agent, photoinitiator and inhibitors provided an improvement in the aesthetic quality of the composite resins, which led to their popularization. The composition of the resins is variable and directly interferes with their mechanical and physical properties, also influencing the quality and clinical success of the restorative treatment. Therefore, it is important that this information is clear and available by the manufacturers for the correct clinical application of the material.

**KEYWORDS:** Composite resin. Composition. Composites.

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## INTRODUCTION

In the early 1960s, the researcher Bowen studied epoxy resins reinforced with load and found that material had negative properties, therefore exhibited low polymerization speed and easy change color, motivating it to combine epoxy and acrylic resins.<sup>1</sup> From this perspective, in the 1970s, began the development of photopolymerizable composites.<sup>2</sup> Through studies and researches, composite resins have become more resistant to wear, stable to color change and longer polymerization reaction time when compared to chemically polymerizable composites.<sup>3,4</sup> However, even with these advances, composites still exhibit low resistance to mechanical wear due to particle size of filler content used.<sup>2</sup> In order to minimize the damages of this characteristic, researchers developed means to reduce size of particles, enabling composites with higher filler content and consequently improvement of mechanical properties.<sup>2</sup> The evolution of these composites allows a wide indication of photopolymerizable composites in clinical practice of several specialties.<sup>5</sup>

Among these indications, highlight direct and indirect restorative procedures, pit and fissure sealants, cores and buildups, inlays, onlays, crowns, provisional restorations, cements for single or multiple tooth prostheses and orthodontic devices, endodontic sealers, and root canal posts.<sup>6</sup> The great clinical applicability of composite materials is possible due to favorable clinical performance (longevity and maintenance of aesthetics), greater preservation of dental tissues and single body formation. This performance is dependent on composition, since qualitative and quantitative constitution of material reflects on the mechanical

properties, biocompatibility, aesthetic quality and adhesion of enamel and dentin.<sup>6,7</sup>

Composite resins are composed of the following phases: organic matrix, inorganic filler and union agent.<sup>5,8</sup> Organic phase consists of dimethacrylates, the most common are dimethacrylate (BisGMA), bisphenol glycidyl dimethacrylate, triethylene glycol dimethacrylate (BisEMA), triethylene glycol dimethacrylate (TEGDMA) and urethane dimethacrylate (UDMA).<sup>9</sup> Polymerization of dimethacrylate by light activation results in a network of polymers whose physicochemical properties depend on degree of conversion and union of monomers.<sup>10</sup>

In order to occur polymerization, the light must activate photoinitiators present in different forms of application of composites. Photoinitiators are responsible for generation of free radicals that will initiate polymerization process and the most found is camphorquinone.<sup>11</sup> However, due to yellowish aspect of camphorquinone, some commercial formulations use other photoinitiators associated with PPD, lucirin TPO and irgacure 819.<sup>6</sup>

In order to increase strength of material in dental compounds are used inorganic filler particles.<sup>11,12</sup> It is commonly use materials such as

quartz, colloidal silica, glass silica containing barium, strontium and zirconia as different types of inorganic filler particles.<sup>13,14</sup> The characteristics of filler particles, in particular the size, will be used to classify composites for inorganic composition.<sup>6</sup> The shape of filler particles varies between irregular or spherical shape and particle size of 0.005  $\mu\text{M}$  to 10  $\mu\text{M}$ .<sup>15</sup> These differences influence roughness, polish, wear resistance, as well as other mechanical properties such as tensile strength and compression.<sup>16-21</sup>

Organic and inorganic phases of composites are responsible for wide clinical applicability of material. However, a chemical bonding agent between these components is required: silane.<sup>22</sup> Silanization must involve the whole surface of inorganic particles to promote improved mechanical properties such as flexural strength and tensile strength, fracture toughness and increase the elastic modulus.<sup>23,24</sup> In addition, presence of silane bonding agent reduces degradation by hydrolysis, avoiding water penetration at filler/matrix interface.<sup>22</sup>

Considering adhesive dentistry and history of composites, photopolymerizable composite has become increasingly indicated and applied clinically. However, for greater longevity and predictability of procedures it is important to know composition of material. This is the first part of 06 relevant topics on composite resins focusing on the last decade: Composition, Properties, Photoactivation, Post-Operative Sensitivity, Color Change and Longevity. Therefore, the purpose of this review is to perform an analysis of literature on constitution of resin composites available and used in Dentistry in the last decade.

## MATERIALS AND METHODS

In order to obtain references, the terms “monomers”, “filler”, “proportion”, “photoinitiator”, “camphorquinone”, “BAPO”, “Lucirin”, “TPO”, “silica”, “Zirconia”, “nano”, “micro”, “hybrid” and “composite resin” were combined. The articles were taken from PubMed platform and after reading title and abstract, 50 articles remained. These were read in full and 30 were used in this review. In addition to scientific articles, the authors surveyed technical profile of manufacturer of 23 composite resins from different manufacturers. There was also an additional search using counter-references and were used articles that fit the study profile. The obtained data were interpreted and discussed. Table 1 contains the results identifying and classifying the composite as to size and type of filler content, monomer composition and percentage of inorganic content in weight and volume.

## RESULTS

Most composite resins are comprised of aromatic or aliphatic diacrylate monomers. The most commonly used dimetracylate monomers are Bis-GMA, UDMA and TEGDMA. In relation to filler content, the most commonly found composition

**Table 1:**

Composite characteristics of composite resins from different manufacturers

PRODUCT NAME	MANUFACTURER	TYPE	ORGANIC MATRIX
Filtek P60	3M ESPE, St. Paul, MN, USA	Hybrid	Bis-GMA, Bis-EMA, UDMA
Filtek Z100	3M ESPE, St. Paul, MN, USA	Microhybrid	Bis-GMA, TEGDMA
Filtek Z250	3M ESPE, St. Paul, MN, USA	Microhybrid	Bis-GMA, Bis-EMA, UDMA
Filtek Z350	3M ESPE, St. Paul, MN, USA	Nanofilled	Bis-GMA, Bis-EMA, UDMA, PEGDMA, TEGMA
Point 4	Kerr, Orange CA, USA	Microhybrid	Bis-GMA, Bis-EMA, TEGDMA
Harmonize	Kerr, Orange CA, USA	Nanohybrid	D.N.F.
Herculite Precis	Kerr, Orange CA, USA	Nanohybrid	Bis-GMA / TEGMA
Beautifil II	Shofu Inc, Kyoto, Japan	Nanohybrid	Bis-GMA TEGMA
Palfique LX5	Tokuyama Dental, Tokyo, Japan	Microhybrid	Bis-GMA, TEGDMA
Ceram-X One	Dentsply, Konstanz, Germany	Nanohybrid	Bis-GMA, TEGDMA, UDMA
Esthet-X HD	Dentsply, Milford, DE, USA	Nanohybrid	Bis-GMA, UDMA, Bis-EMA
Mosaic	Ultradent, USA	Nanohybrid	Bis-GMA, Bis-PEGDMA, TEGDMA
Admira Fusion	VOCO GmbH, Cuchaven, Germany	Nanohybrid Ormocer	Bis-GMA, HEMA, UDMA, TEGDMA
Grandio	VOCO GmbH, Cuchaven, Germany	Nanohybrid	Bis-GMA, UDMA, TEGDMA
Charisma Classic	Heraeus Kulzer, Hanau, Germany	Microhybrid	Bis-GMA, TEGDMA
Charisma Diamond	Heraeus Kulzer, Hanau, Germany	Nanohybrid	TCD-DI-HEA, UDMA, TEGDMA
Clearfil AP-X	Kuraray Medical Inc., Okayama, Japan	Microhybrid	D.N.F.
Clearfil Majesty Posterior	Kuraray Medical Inc., Okayama, Japan	Nanohybrid	Bis-GMA, TEGDMA, UDMA
IPS EmpressDirect	Ivoclar Vivadent, Schaan, Liechtenstein	Nanohybrid	Bis-GMA, UDMA, TCDD
Tetric N-Ceram	Ivoclar Vivadent, Schaan, Liechtenstein	Nanohybrid	Bis-GMA, UDMA
Opallis	FGM Produtos Odontológicos, Joinville, SC, Brazil	Nanofilled	Bis-GMA, Bis-EMA, TEGDMA, UDMA
Vitra APS	FGM Produtos Odontológicos, Joinville, SC, Brazil	Nanofilled	Formula is free of Bis-GMA and Bis-EMA
Luna	SDI, Germany, GmbH Hansestrasse	Nanohybrid	D.N.F.

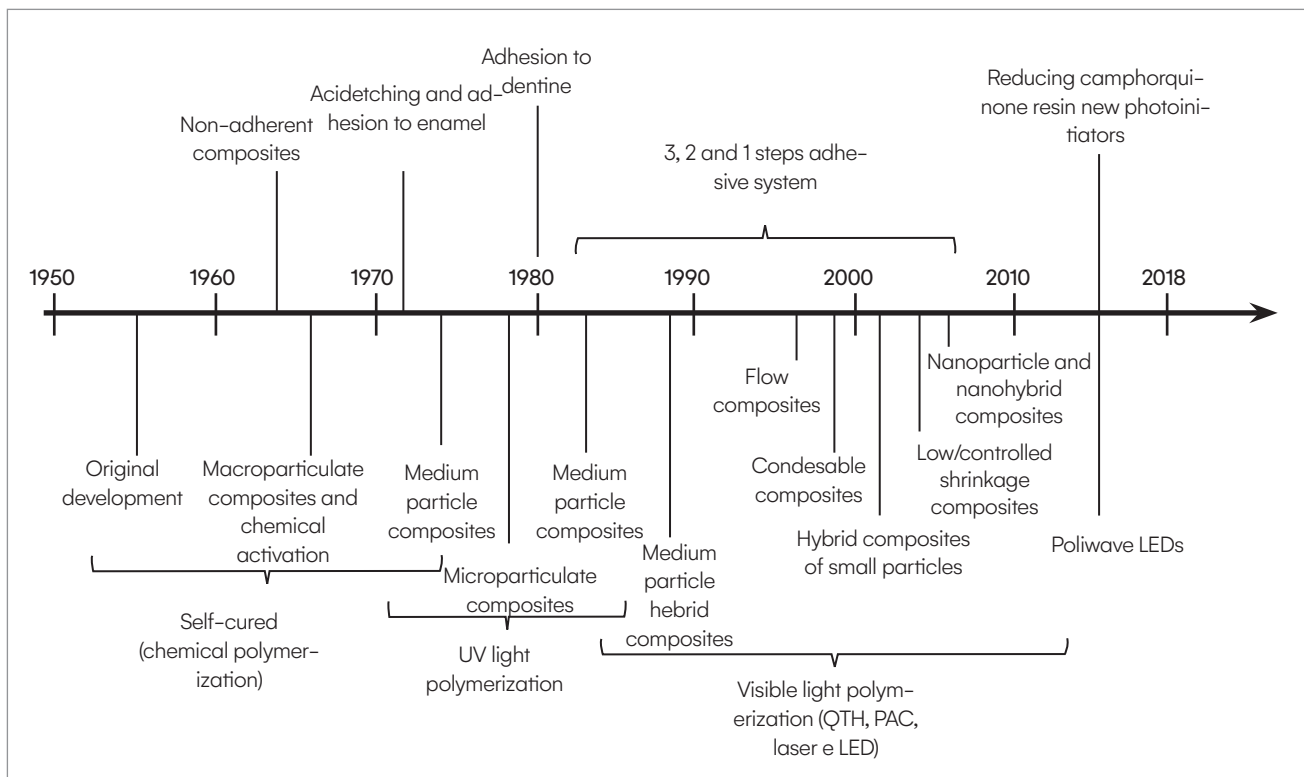
Abbreviations: D.N.F (data not found (technical information not mentioned in the package leaflet. and/or on the manufacturer's website); TCD-DI-HEA (Tricyclodecane-urethane dimethacrylate); CQ (camphorquinone); TPO (trimethylbenzoyldiphenylphosphine oxide); Bis-GMA (bisphenol-A-glycidyl dimethacrylate); UDMA (urethane

FILLER SIZE	INORGANIC FILLER	WEIGHT %	VOLUME%	PHOTOINITIATOR
0,01 – 3,5 µm	Zirconia and silica	83%	61%	D.N.F.
0,01 – 3,5 µm	Zirconia and silica	D.N.F.	66%	D.N.F.
0,01 – 3,5 µm	Zirconia and silica	82%	60%	D.N.F.
Silica 0.02 µm/zirconia 0.004 – 0.011 µm	Nonaggregated silica/zirconia filler. aggregated silica/zirconia cluster	78,5%	63,3%	CQ/amine
0,4 µm	Barium glass and silica	76%	57%	D.N.F.
N.D.E.	D.N.F.	D.N.F.	D.N.F.	D.N.F.
30 – 50 µm / 0,02 – 0,05 µm / (mean 0,4 µm)	Prepolymerized filler. silica nanofiller. hybrid filler (barium glass)	78%	59%	D.N.F.
0,01 – 4,0 µm / (mean 0,8 µm) 0,01 – 0,02 µm	Multifunctional glass filler and S-PRG filler based on fluoroboroaluminosilicate glass	83,3%	68,6%	D.N.F.
0,1 – 0,3 µm (mean 0,2 µm)	Silica-zirconia and composite filler	82%	71%	CQ/amine + Lucirin TPO
1,2 – 1,6 µm	Methacrylate modified polysiloxane. barium-aluminium-borosilicate glass. silica nanofiller	77%	55%	D.N.F.
< 1,0 µm / 0,04 µm	Nano silica particles. barium fluorobosilicate glass	77%	60%	D.N.F.
0,02 µm	Ceramic zirconia-silica glass	D.N.F.	68%dentine 56%enamel	D.N.F.
N.D.E.	D.N.F.	84%	D.N.F.	D.N.F.
1 µm (ceramics) 0,02-0,04 µm (SiO <sub>2</sub> )	Glass ceramics. silicon dioxide (SiO <sub>2</sub> ). iron oxide. titanium dioxide	87%	71,4%	D.N.F.
0,005 – 10 µm	Fluorinated aluminum barium glass. pre-polymerized particles	78%	61%	D.N.F.
0,005 – 20 µm	Glass of barium fluoride and aluminum	81%	64%	D.N.F.
0,1 – 15 µm	Barium glass. silica	86%	70%	D.N.F.
0,02 – 1,5 µm	Aluminum. ceramic glass and barium	92%	82%	D.N.F.
0,04 – 3µm (mean 0,55 µm)	Aluminum and barium fluorosilicate glass. barium glass particles. mixed oxide and copolymers	71,8%	D.N.F.	CQ/amine + Lucirin TPO
3,0 – 0,04 µm	Barium glass. ytterbium trifluoride. mixed oxide and copolymers	80-81%	55-57%	D.N.F.
0,04 – 3,0 µm (mean 0,5 µm)	Barium-aluminum glass. silicate silanides. nanoparticles of silicon dioxide	78,5%- 79,8%	57%-58%	CQ/amine
0,2 µm	Fillers of zirconia. silica	72%-80%	52%-60%	D.N.F.
0,04 – 1,5 µm	D.N.F.	77,5%	61%	D.N.F.

dimethacrylate); TEDGMA (triethyleneglycol dimethacrylate); Bis-EMA (ethoxylated bisphenol-A-dimethacrylate); PEGDMA (polyethylene glycol dimethacrylate); HEMA (2-hydroxyethyl methacrylate); TCDD (dimethanol dimethacrylate tricyclodecane); R.A.P (radical amplified photo-polymerization).

is borosilicate particles, aluminum silicate, lithium aluminum silicate, ytterbium fluoride, barium glass, strontium and zirconia. Camphorquinone

and MPTS is the most found photoinitiator and silane, respectively. Figure 1 presents the chronology of composition evolution of composites.



**Figure 1:**

Chronology of development of dental composites as monomers, filler particles, adhesive system technologies and photopolymerization activation.

## DISCUSSION

### Monomers

Composites based on dimetacrilates have several applications in operative dentistry. Since the introduction in market, research has focused on improving the formulations that use these monomers to increase their clinical care.<sup>9</sup> Among these monomers, Bis-GMA has been the most used monomer in composition. It has a long and rigid chain with double bonds of reactive carbons in extremities. Long length gives lower contraction tension, besides aromatic rings in center being capable of generating greater resistance. Due to high viscosity, Bis-GMA must be associated with other low molecular weight monomers that allow incorporation of initiators, inhibitors and filler particles. This also improves composite esculpture.<sup>15, 25</sup>

TEGDMA is a highly flexible, low molecular weight, low viscosity monomer and its characteristics contribute to high mobility during polymerization, resulting in improved polymerization.<sup>26</sup> However, composites containing large amounts of TEGDMA may contract more and consequently generate higher residual contraction tension.<sup>27</sup>

The most used monomers as diluents for BisGMA are ethylene glycol dimethacrylate (EGDMA), triethylene glycol dimethacrylate (TEGDMA) and urethane dimethacrylate (UDMA). However, use of low molecular weight monomers results in a higher polymerization contraction, decreasing the advantages of use Bis-GMA. In general, the higher use of these monomers increase polymerization contraction and chances of marginal infiltration.<sup>15,25</sup>

### Inorganic filler

Composite resins have in their composition about 30 to 70% by volume and 50% to 80% of their weight in filler particles, which are capable of imparting composite resins an improvement in their chemical and physical properties. This is improvement their resistance to support various conditions found in oral cavity. The higher amount of inorganic filler promotes improvements in compressive strength, tensile strength, modulus of elasticity and toughness. For this to occur the volume of composites should be approximately 70% of inorganic particles<sup>15</sup>.

The addition of filler particles in matrix of composites is able to soften values of shrinkage stress, which are around 1 to 4%. This also give a coefficient of thermal expansion closer to enamel, then decreasing tension between tooth-resin interface.<sup>15</sup> In addition, inorganic filler particles are able to interfere with viscosity, significantly altering clinical characteristics of compound handling. High inorganic filler content is able to decrease water sorption, making composites less susceptible to wear abrasive and staining.<sup>15</sup>

Some particles added to matrix such as Ba, Sr, Zn and YbF<sub>3</sub> confers to composites a radiopacity similar to enamel, which makes it important for diagnosis of secondary caries. Composites can be classified according to size of their particle in microparticulate (0.01 to 0.1 μm), hybrid (0.01 to 10 μm) and nanoparticulate (0.001 to 0.1 μm)<sup>15</sup>. Composites classified as nanohybrids have nanoparticles in composites of hybrid type, these particles can “fill” regions between the larger particles, allowing larger volumetric fractions of filler and reducing polymerization shrinkage.<sup>23</sup> According to some studies, addition of smaller particles provides greater wear resistance, longer polishing longevity and a lower polymerization shrinkage, resulting in less plaque buildup, better color stability and resulting in longer restorations.<sup>15,28</sup>

### **Union agent**

Effective union between inorganic and organic parts through use of silane has demonstrated a decrease in degradation, fracture and improvement in distribution of tension in composite resins.<sup>23</sup> Several types of silanes can be used in formulation of dental composites. Of these, the most studied are the MPTS (methacryloyloxypropyltrimethoxysilane), OTMS (n-octyltrimethoxysilane), UDMS (3-[(1,3(2)-dimethacryloyloxypropyl)-2 (3)-oxycarbonylamido] propyl

triethoxysilane), GPS (γ-glycidoxypropyltrimethoxysilane) and ATES (organosilanes alyltriethoxysilane).<sup>22</sup> However, most of composites use MPTS silane agent, which promotes union between organic and inorganic matrix through copolymerization of covalent and hydrogen bonds.<sup>23,29</sup> Studies have shown that increase in silane concentration (MPTS) led to a decrease in viscosity of experimental composites. Silanized composites have higher flexural strength when compared to non-silanized filler particles.<sup>30</sup> Another factor demonstrated was increased resistance to degradation by hydrolysis of different composites when filler particles were treated with silane.<sup>30</sup> The use in nanoparticulate composites has demonstrated better mechanical and physical properties due to presence of a greater amount of inorganic particles and consequently a larger surface for adhesion of silane molecules.<sup>12,23</sup> In general, treatment of inorganic filler particles with MPTS silane is able to improve flexural strength of composites and resistance to degradation by hydrolysis.<sup>30</sup>



### **Photoinitiator and Inhibitor**

Composites are primarily composed of photoinitiator diketone camphorquinone combined with a reducing agent may be a tertiary amine which is responsible for production of ions radicals upon exposure to visible light in 400–500nm wavelength, initiates polymerization.<sup>31</sup> This type of system has a good acceptance, so it is still widely used. However, camphorquinone presents some minor disadvantages, such as yellowish characteristic<sup>32</sup> and presence of alpha-diketone group, which has an absorption peak in range of ambient light (fluorescent lamps and reflectors). In the latter case, a rapid onset of photopolymerization may occur and cause a short working time.<sup>33</sup> Currently there are other photoinitiators that solve these problems, which has shown a higher degree of conversion when compared to camphorquinone / tertiary amine system.<sup>34, 35</sup> These systems are composed of addition of Lucirin TPO, having characteristics of a less yellowish color, being indicated mainly for composites for whitened and translucent teeth.<sup>36</sup> The absorption spectrum of Lucirin TPO ranges from 380 nm to 425 nm.<sup>37</sup> When the main photoinitiator system of a composite is not camphorquinone, the activation of initiator system has an absorption of light in addition to wavelength of blue LED. However, as some manufacturers do not mention initiator specifications used in their products, it is difficult to predict adequate photoactivation of all types of composite resins.<sup>38–40</sup>

In composite resins, there are still small amounts of butylated hydroxide-luene (approximately 0.01% by weight), which acts to prevent premature polymerization and to prolong life of resin leading to a more stable material.<sup>41</sup> However, addition of this component is also capable of affecting the polymerization of

composites, maintaining slower polymerization and prolonging pre-gel phase. This will promote molecular arrangement of polymer chains, dissipate formed stresses and after total consumption of butylated hydroxytoluene molecules, the reaction will accelerate by promoting increase of polymer chains.<sup>42</sup> In this way, formation of a rigid material and resistant to plastic deformation will occur. Studies have shown that increase in concentration of butylated hydroxytoluene (from 0.05 to 1.0%), is able to decrease stress accumulation rate without causing a significant decrease in final conversion degree.<sup>42</sup> However, studies using high concentrations of camphorquinone and hydroxybutylated (1.5%) demonstrated a high degree of conversion, besides allowing a slow and gradual accumulation of shrinkage tension during polymerization.<sup>43</sup>

### **Optical properties**

Improvements in composite resins not only occurred in mechanical characteristics but also in optics.<sup>44</sup> Among these evolutions, reduction in size of particles that is able to enable better optical properties in nanoparticulate and hybrid compounds.<sup>45</sup> Once formulation of composites influences optical properties.<sup>46</sup> Materials containing different monomer formulations may also exhibit differences in color and translucency.<sup>47</sup> Change in amount of Bis-GMA is a factor that contributes to translucency of composites and may be an alternative for adjusting translucency.<sup>48</sup>

When selecting the color of a composite resin we must bear in mind that light transmission through darker tones is less due to higher opacity compared to lighter tones. Therefore, pay attention to photoactivation and thickness of increments are important in this step.<sup>49</sup> It should also be noted that color selection through pre-fabricated scales should be avoided, because in the same brand and composite color there will be different shades. It may introduce error in process of color selection, so making of customized guides help solve these problems.<sup>50</sup>

### **CONCLUSION**

It is possible to observe that a wide variation in amount of compounds used in composite resins are capable of altering mechanical and physical properties. Therefore, it is necessary for manufacturers to make components present in composites clearly available for correct clinical application.

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