Graphene-based Nanostructures for Future Dental Composites

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Editor's note: This review paper provides a systematic overview of the latest advancements in the development of next-generation dental composites utilizing graphene, graphene-based nanostructures, and biomaterials. Allahbakhsh indicates that nanotoxicity is a significant barrier to the widespread use of graphene-based nanostructures in dental composites. He believes that addressing this issue could pave the way for new developments in future dental composites.

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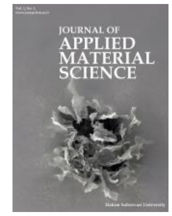

Review

Graphene-based Nanostructures for Future Dental Composites

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Abstract

Dental composites are becoming more important as an alternative to traditional dental materials. However, their low mechanical properties have limited their widespread use. Recently, there has been a lot of research interest in using graphene-based nanostructures to improve the mechanical properties of dental composites. The challenge now is to ensure that these future dental composites are biocompatible. This review paper provides an overview of the latest research on developing graphene-based dental composites, covering their structure, mechanical properties, and biocompatibility. It first gives a brief overview of dental composites and their structures, then introduces and compares different types of graphene-based nanostructures. Finally, the paper presents recent advances in developing graphene-based dental composites. The overview confirms that the mechanical properties of dental composites can be significantly improved by incorporating graphene-based nanosheets. However, further studies on other types of graphene-based nanostructures, specifically on the biocompatibility of these future dental composites, seem necessary.

Keywords: Graphene, Nanostructures, Dental composites, Biomaterials.

1. Introduction

Over the past few years, there has been a remarkable increase in research on the use of graphene-based nanostructures in dental restorations [1]. Graphene and its derivatives are being explored as promising materials for creating dental adhesives [2], substrates and scaffolds for dental pulp stem cells [3, 4], teeth whitening [5], dental implants [6, 7], dentin erosion protective coatings [8], and dental composites [9, 10]. The extensive potential applications of these materials stem from their impressive properties and their customizable nature [11]. The high surface area of graphene allows for easy modification of this material by adding different functionalities to its basal plane and edges [12]. Furthermore, 3D graphene-based nanostructures can be produced through the one-pot self-assembly of graphene derivatives [13]. Hence, graphene-based nanostructures could pave the way for the development of future dental materials, particularly dental composites.

Graphene-based nanostructures can be divided into three main categories [14]: (i) graphene-based nanosheets, which include graphene, graphene oxide (GO), reduced graphene oxide (rGO), and functionalized GO or rGO nanosheets, (ii) graphene-

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based porous nanostructures, such as rGO and GO hydrogels, aerogels, and foams, and (iii) graphene-based composites. The latter type is widely used in the design of dental composites. In these composites, graphene-based nanostructures are dispersed in the structure of a polymeric resin used in dental composites [15]. Moreover, graphene-based porous nanostructures provide large specific surface areas and highly porous structures with very low densities, which can be beneficial for the development of lightweight dental composites [16].

Different classes of graphene-based nanostructures have already been used in the development of biomaterials and biosystems. However, nanotoxicity is the major limit for any biological applications of most graphene-based nanostructures [17]. Nanotoxicity in graphene-based nanostructures is closely related to the type and concentration of these materials [18]. Consequently, understanding the mechanisms involved in the nanotoxicity of graphene-based nanostructures can provide a roadmap to develop next-gen dental composites based on advanced nanostructures such as graphene-based nanostructures.

In this review paper, a systematic overview of the recent advances in the development of graphene-based dental composites is provided. The first section introduces dental composites, followed by an exploration of graphene-based nanostructures applicable to the design of dental composites. Finally, the recent advances in the development of high-performance graphene-based dental composites are covered.

2. Dental composites

The replacement of dental amalgams with dental composites has been increasingly accelerated in recent decades due to aesthetic and health reasons [19]. However, commercially available resin-based dental composites still have some significant shortcomings that require further research. One of the main drawbacks of dental composites is their lower mechanical properties compared to dental amalgams. The modulus of commercially available composites is about 30-50% lower than dentine [20]. Additionally, the average lifetime of dental composites is in the range of 3-5 years, which is much lower than that of amalgams (7-10 years). Issues such as hydrolysis of the filler due to water absorption and the release of chemical compounds over time are also significant concerns with current dental composite technologies [20]. Therefore, there is a clear need for more reliable dental composites with more advanced matrix and filler systems.

Dental composites have some key advantages, such as a tooth-like appearance, resistance to the oral environment, easy handling, and the ability to cure with light. However, they also have some drawbacks, including shrinkage during the curing process, the risk of secondary cavities, high thermal expansion, and low wear resistance [21]. Also, the leaching of uncured monomers due to an uneven curing process can be a significant concern in practical applications, as these monomers can cause cytotoxic effects in the surrounding gum tissues [22].

The main components of a dental composite formulation are (i) an organic matrix, usually based on 2,2-Bis[p-(20-hydroxy-30-methacryloxypropoxy)phenylene]propane (Bis-GMA) or 1,6-bis(methacryloxy-2-ethoxycarbonylamino) -2,4,4-trimethylhexane (UDMA) and the co-monomer triethylene glycol dimethacrylate(TEGDMA) to control viscosity, (ii) a reinforcing filler, such as clay, quartz, or fused silica, and (iii) a coupling agent, such as γ-methacryloxy propyltrimethoxysilane, to enhance bonding between the filler and resin matrix.

Recent studies suggest using nanomaterials instead of traditional reinforcing fillers to enhance the mechanical properties of dental composites [23]. Various types of nanomaterials such as nano clay [23], nano SiO2 [24], alumina [25], and different carbon nanomaterials [5, 26, 27] have been utilized in the development of dental composites. Among these materials, graphene-based nanostructures show great promise due to their superior mechanical properties and are considered for the development of next-gen dental composites [5, 28].

3. Graphene-based nanostructures

Graphene-based nanostructures have been a major focus in nanotechnology over the past decade. The unique structure of graphene nanosheets gives them exceptional properties, including a large surface area of 2630 m2/g, Young's modulus of approximately 1.0 TPa, and a breaking strength of around 40 N/m [29]. Moreover, the large surface area of graphene can be easily modified with different functionalities to achieve a wide range of surface chemistries with various properties. This tunable surface can be used to create high-performance resin-based nanocomposites with a high degree of compatibility between nanosheets and the polymeric matrix [30, 31]. More importantly, this customizable functionality can be utilized to impart important characteristics to resin-based composites, such as antibacterial properties [32], biocompatibility [28], and low toxicity [33]. This is a key reason for the significant research interest in graphene and its derivatives for biomedical applications [34, 35].

The unique surface functionality of GO can be utilized to create high-performance porous nanostructures with a large specific surface area and diverse surface chemistries [36]. These nanoporous platforms offer increased surface areas at low densities, resulting in enhanced mechanical properties and reduced resin shrinkages due to strong matrix-filler interactions [20]. Moreover, these structures can be easily converted to bioactive materials by impregnating their porous structure with different bioactive agents [20]. However, there is limited studies on the application of graphene-based porous nanostructures in dental materials, and only a few on graphene in fabricating dental composites.

4. Graphene-based dental composites

Both graphene oxide (GO) and nitrogen-doped graphene (N-graphene) show potential for use in dental composite applications [37]. However, reduced graphene oxide (rGO) may not be suitable for this application. In comparison to rGO nanosheets, GO nanosheets have low cytotoxic effects and cause oxidative stress without damaging cell membranes [37]. Nitrogen-doped graphene nanosheets also have lower cytotoxic effects than rGO nanosheets. However, some antioxidant activity was observed for N-doped graphene nanosheets, especially at high doses [37].

GO nanosheets showed no effect on the cell cytoskeleton at concentrations ranging from 4-20 μg/mL [37]. In contrast, rGO nanosheets disrupted the microtubule network at a wide range of doses (4-40 μg/mL). Furthermore, condensation and loss of adhesion were reported at high doses of N-doped graphene nanosheets (40 mg/mL). Additionally, GO nanosheets demonstrate good biocompatibility in nanocomposite form, making them ideal candidates for the development of dental composites (Figure 1).

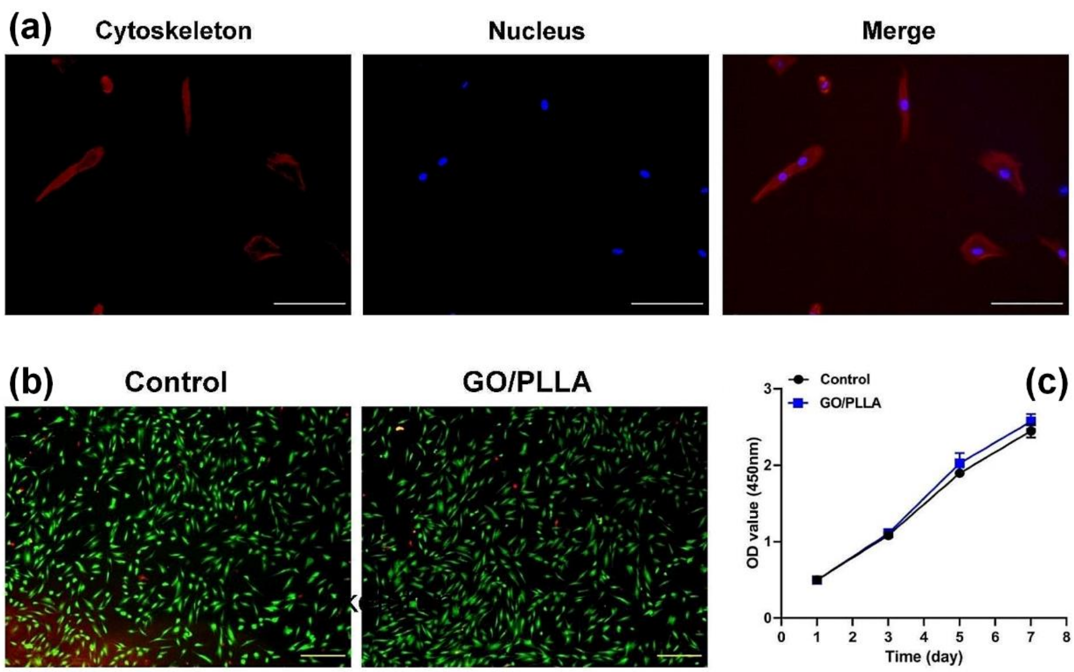

Figure 1. (a) The cytoskeleton staining results of dental pulp stem cells on composite scaffolds based on GO and poly-L-lactic acid (PLLA) with the cytoskeleton stained in red and the nucleus stained in blue, (b) fluorescence microscopic images of GO/PLLA scaffolds, (c) cell viability study of the GO/PLLA scaffold (scale bars in a and b panels show 200 and 500 μm, respectively) [4].

As discussed earlier, graphene-based nanostructures can also be employed in fabricating dental adhesives [1]. Micro-cavities form between the healthy tissue and the dental composite due to the composite shrinkage can cause dental restoration failure, as bacteria can access dental tissues through these cavities [38]. Consequently, dental composites should be bonded to the hard tissues of teeth to make non-invasive caries removal achievable. Moreover, since biofilms play a major role in periodontal diseases, anti-biofilm dental adhesive can be a perfect strategy to overcome these issues. Anti-biofilm dental adhesives can be fabricated by embedding antibacterial agents in the adhesive material [39].

Graphene and its derivatives have been considered widely as antimicrobial materials and substrates and therefore, these nanostructures have a high potential to be employed in fabricating anti-biofilm adhesives. Three main mechanisms have been suggested for such a performance of these nanostructures. (1) These 2D structures can wrap the cells and induce mechanical stress, which leads to the prevention of nutrient uptake. (2) The sharp edges of these nanosheets can act as nano-knifes and disrupt the biofilm. (3) The oxidative stress production, which prevents the biofilm formation.

In a comparison between functionalized and unfunctionalized graphene nanosheets, it was found that oxygen-containing functional groups on the basal plane and edges of GO nanosheets can enhance the adhesion of bacteria to the nanosheet because surface structural defects increase biofilm adhesion to a surface [40]. However, while oxygen-containing functional groups can improve the compatibility between nanosheets and the polymeric matrix, these functionalities can also affect the cytotoxicity of nanosheets. Akhavan and Ghaderi [40] suggested that the higher antibacterial activity of the reduced form of nanosheets could be attributed to the improved charge transfer between the bacteria and the sharper edges of reduced nanosheets through nanosheet-bacteria interactions. This aligns closely with the findings of Bregnocchi and colleagues [41], who studied the antimicrobial and anti-biofilm performance (against *S. mutans*) of Bis-GMA-based dental adhesive composites in the presence of graphene nanosheets (Figure 2).

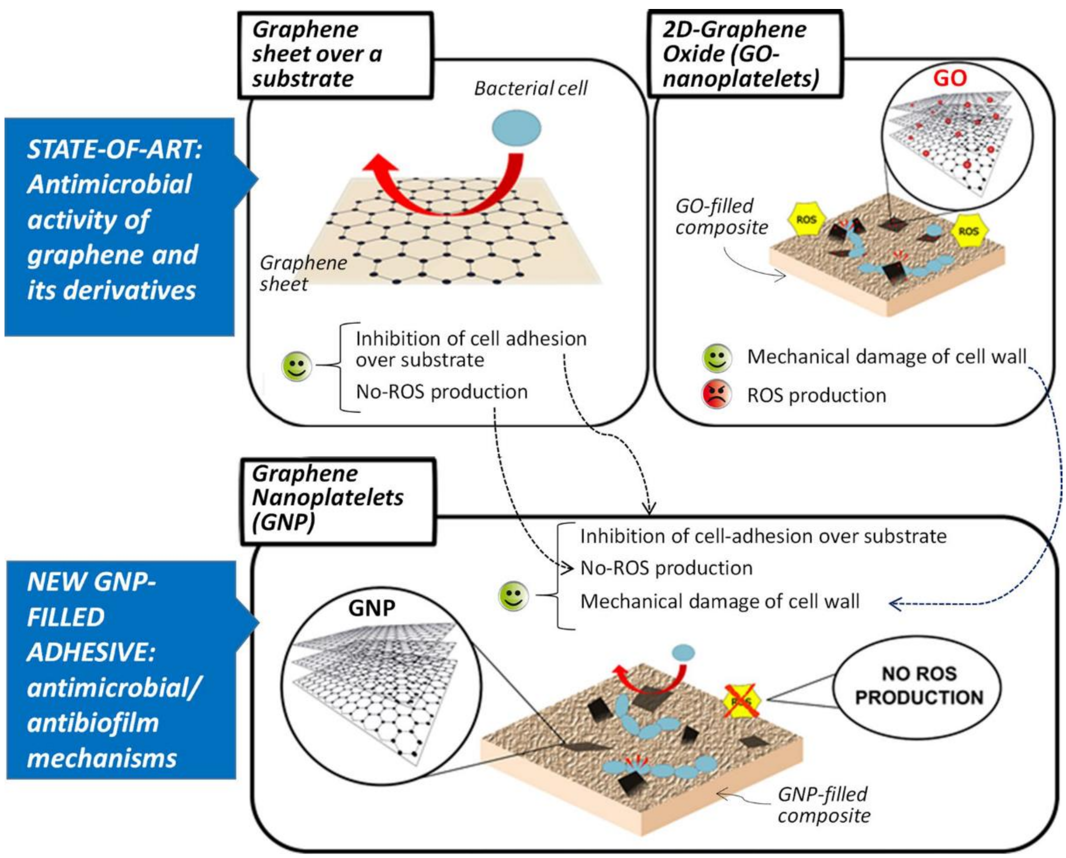

Figure 2. A schematic presentation for the anti-biofilm and antimicrobial characteristics of graphene, GO, and rGO nanosheets [41].

Bregnocchi *et al.* [41] utilized 0.2%wt graphene nanosheets, which were prepared by thermally expanding graphite, to fabricate nanocomposite adhesive systems with anti-biofilm properties. The nanocomposite adhesive showed almost no difference in μ-TBS compared to a commercial adhesive. Using 0.2%wt graphene nanosheets led to a more than 50% reduction in biofilm formation (Figure 3). However, it was suggested to control the shear rate applied to the nanocomposite adhesive, as low shear rate regimes resulted in processing difficulties. The antibacterial properties of graphene-based dental composites are reported in Table 1.

Graphene-based nanosheets, including GO and rGO, can be further modified with functional groups such as grafting with polymeric agents like BisGMA-UDMA to improve compatibility with the resin matrix [45]. Such a grafting can extremely improve the compatibility between graphene-based nanosheets and the resin matrix. Additionally, silane surface modification has been found to be a promising technique for enhancing the compatibility of fillers and the matrix in dental composites [46, 47]. GO nanosheets can be surface-modified with silane modifiers to improve the compatibility of nanosheets and the polymeric matrix [48]. However, it is important to consider the

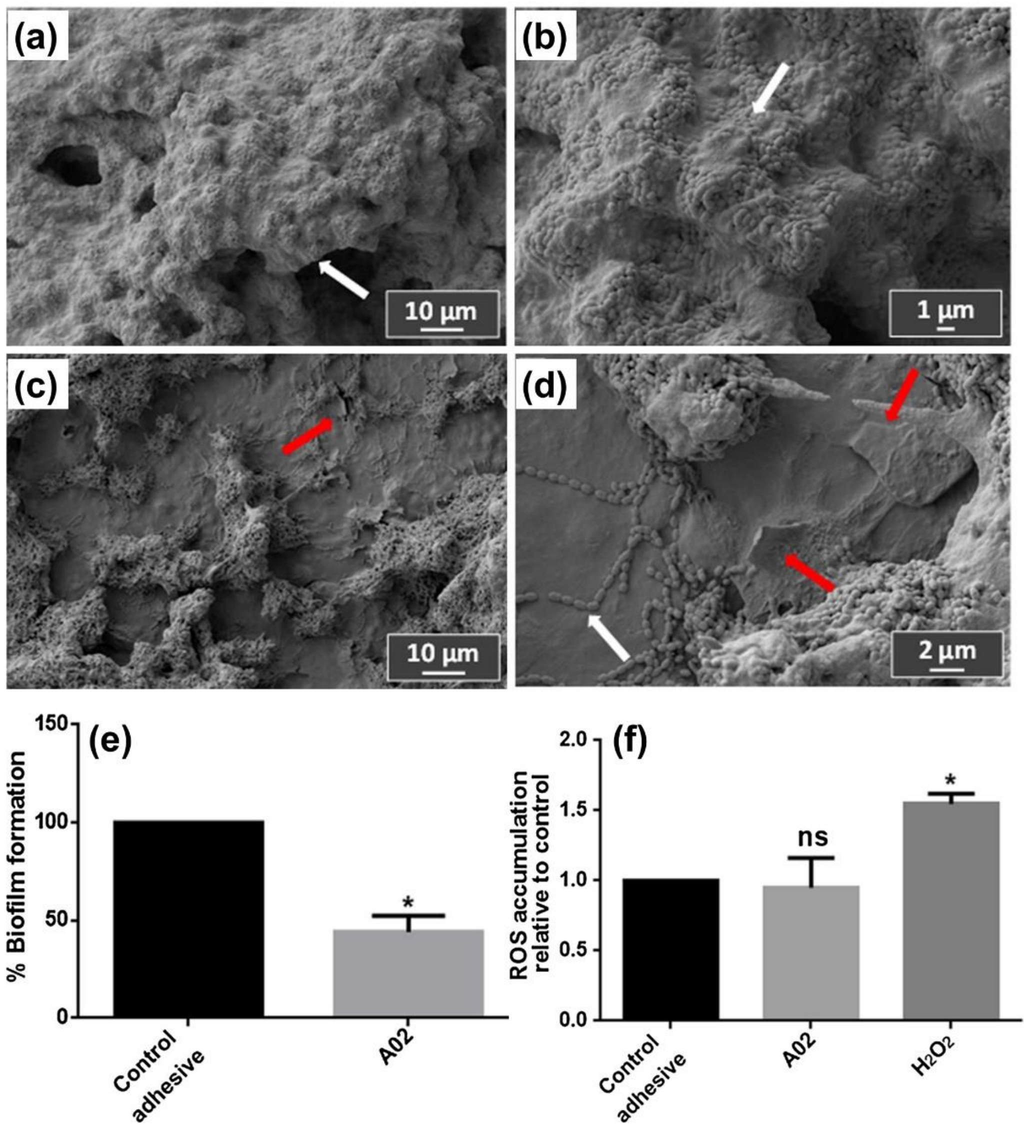

Figure 3. (a, b) Scanning electron microscopy (SEM) images of *S. mutans* biofilm growth on the teeth coated with the control adhesive and (c, d) a dental nanocomposite adhesive with 0.2wt% graphene nanosheets in the structure (A02), (e) biofilm formation percentage and (f) ROS accumulation of the A02 adhesive [41].

Table 1. Antibacterial properties of different graphene-based nanostructures in dental composites

Resin	Nanomaterial	Concentration (wt.%)	antibacterial activity	Ref.
BisGMA/TEGDMA	GO-SiO2	1.8	*S. aureus*	[42]
BisGMA/HEMA	Graphene	0.2	*S. mutans*	[41]
PMMA	GO	2	*Candida albicans*	[43]
Glass ionomer cements	Fluorinated graphene	4	*S. aureus* and *S. mutans*	[44]
BisGMA/TEGDMA	GO	3	*S. mutans*	[9]

cytotoxicity of silane-based monomers when using them for surface modification of graphene-based nanosheets for dental composite applications [49].

The strong compatibility between nanosheets and the resin matrix not only affects the shrinkage of the dental composite but also its mechanical properties. Sun et al. [44] used a fluorinated graphene nanosheet system to enhance the mechanical properties, wear-resistance, and antibacterial characteristics of conventional glass ionomer cements (GICs) at low loading contents (0.5-4 wt.%). A hydrothermal synthesis process was utilized to synthesize fluorinated graphene powder from GO nanosheets using hydrofluoric acid and nitric acid. Results showed that employing only 2 wt.% of fluorinated graphene nanosheets led to more than 60% enhancement in microhardness and 59% enhancement in compressive strength of the dental composites. The study also reported a significant increase in inhibitory rates against *S. mutans* and *S. aureus* (up to 85.3% and 88.1% with 4 wt.% of fluorinated graphene, respectively) in the fluorinated graphene-incorporated dental composites. Additionally, the results revealed that fluorinated graphene nanosheets not only improve the mechanical properties but also enhance the tribological properties of dental composites [44].

Wear resistance is another important characteristic of dental composites in their clinical applications. Occlusion, chewing, and tooth brushing can cause wear of dental composites [50]. Previous research has primarily focused on improving the wear resistance of dental composites by enhancing resin crosslinking degree, filler size, and morphology [50, 51]. In addition, the bonding between the reinforcing agent and the polymeric resin (the filler/resin interface) also significantly affects the wear resistance of dental composites [51].

Apart from the discussed mechanical and anti-microbial properties, graphene-based nanostructures can significantly enhance the wear resistance of dental composites. Graphene nanosheets, whether in single- or few-layer forms, are known for their exceptional wear resistance, even under harsh sliding conditions [52]. Furthermore, incorporating graphene nanosheets into a polymeric matrix can significantly reduce the wear rate of the polymer [53]. This characteristic can significantly enhance the practicality of dental composites reinforced with this type of reinforcement.

As previously mentioned, the limited mechanical properties of commercially available dental composites are a major constraint to their widespread use. The incorporation of graphene-based nanostructures has the potential to improve this key aspect of dental composites, even at very low concentrations. In a study by Malik et al. [54], graphene nanosheets at a very low content (0.2 wt.%) were utilized to enhance the mechanical properties of a polyacrylic resin-based dental composite. While the study did not report the dispersion state of the nanosheets, the results indicated a significant improvement, with a more than 22% increase in compressive modulus and a 27% enhancement in compressive strength of the dental composite when enhanced with 0.2 wt.% graphene nanosheets [54].

Bacali et al. [55] proposed using a hybrid nanostructure filler composed of silver nanoparticles and graphene nanosheets to improve the mechanical properties of poly(methyl methacrylate) (PMMA) dental composites (Figure 4). They were able to achieve a perfect dispersion and distribution of the hybrid Ag-graphene nanofiller system in the polymeric matrix. While the modulus of the systems showed only a minor increase of less than 10%, there was a meaningful improvement in compressive strength [55]. It is possible to infer from their results that the suggested hybrid Ag-graphene nanosystem may have a more significant impact on enhancing PMMA-based dental materials if interactions between the nanosystem and the matrix are observed. Additionally, a higher ratio of graphene to silver in the hybrid nanosystem, proposed as the reinforcing phase in this study, could lead to better mechanical enhancements.

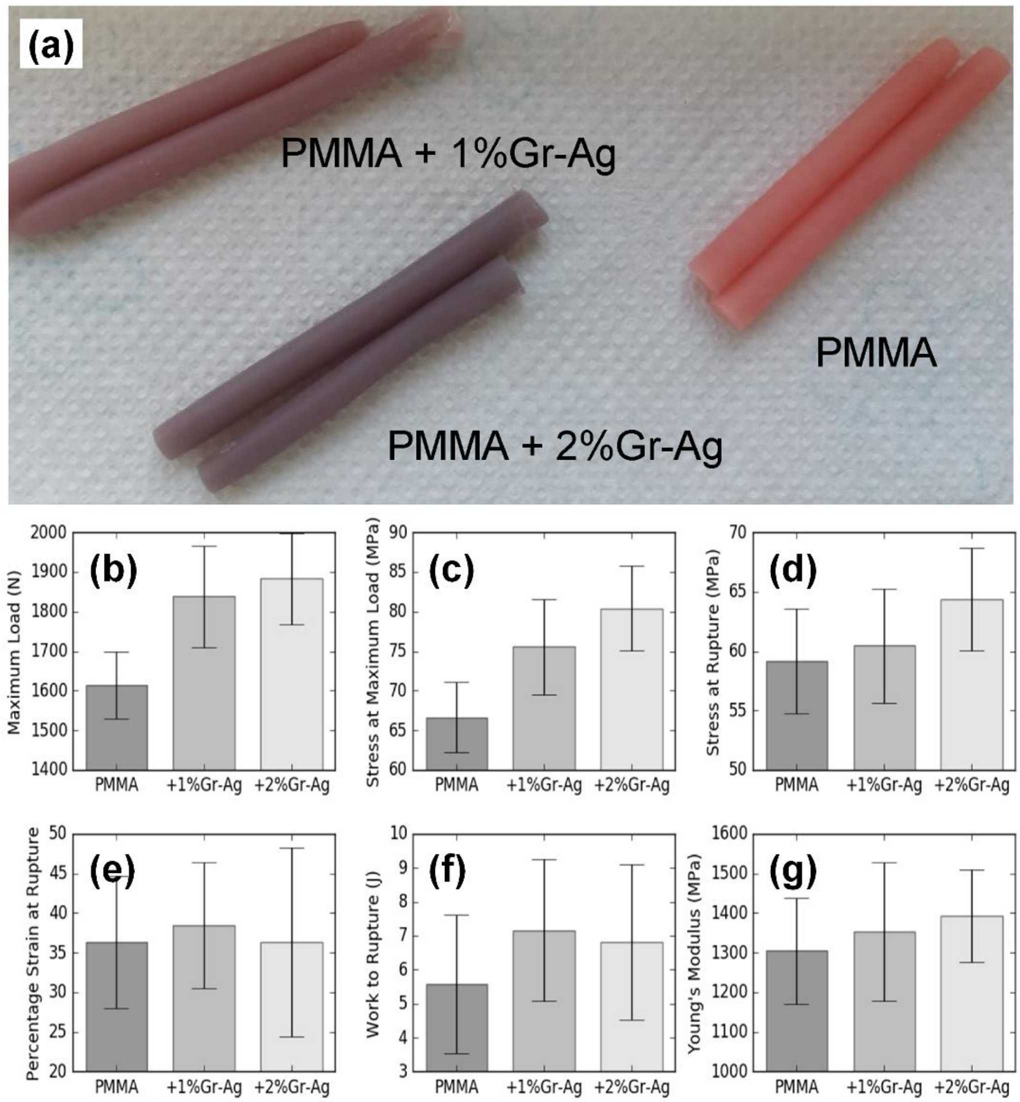

Figure 4. (a) a photograph showing the appearance of samples after curing; (b) maximum load, (c) stress at maximum load, (d) rupture stress, (e) rupture strain, (f) rupture work, and (g) Young's modulus of PMMA/graphene-Ag dental composites [55].

Moldovan et al. [56] investigated the use of GO-based filler systems in the development of dental composites. Specifically, they looked at GO-hydroxyapatite-SiO_2 (GS) and GO-hydroxyapatite-ZrO_2 (GZ) compared to a hydroxyapatite-Ag (CC) filler system. These composites were based on the Bis-GMA/TEGDMA matrix systems. The researchers used γ-methacryloxypropyltrimethoxysilane (A-174) to enhance the compatibility of the polymeric resin and nanofiller systems. They focused on evaluating the color stability of the dental composites when exposed to coffee and red wine, as well as assessing their antibacterial activity (Figure 5).

The color stability of dental composites was enhanced by using GO nanosheets. In comparison to the CC filler system, which exhibited the least stability, the GS filler system showed the greatest improvement in color stability of the dental composite, followed by the dental composite filled with the GZ filler system. Additionally, according to atomic force microscopy (AFM) studies, the dental composite filled with the GZ filler system demonstrated the best resistance to surface roughness and erosion, followed by the GS filler system [56].

The antibacterial properties of dental composites filled with different filler systems against *Staphylococcus*

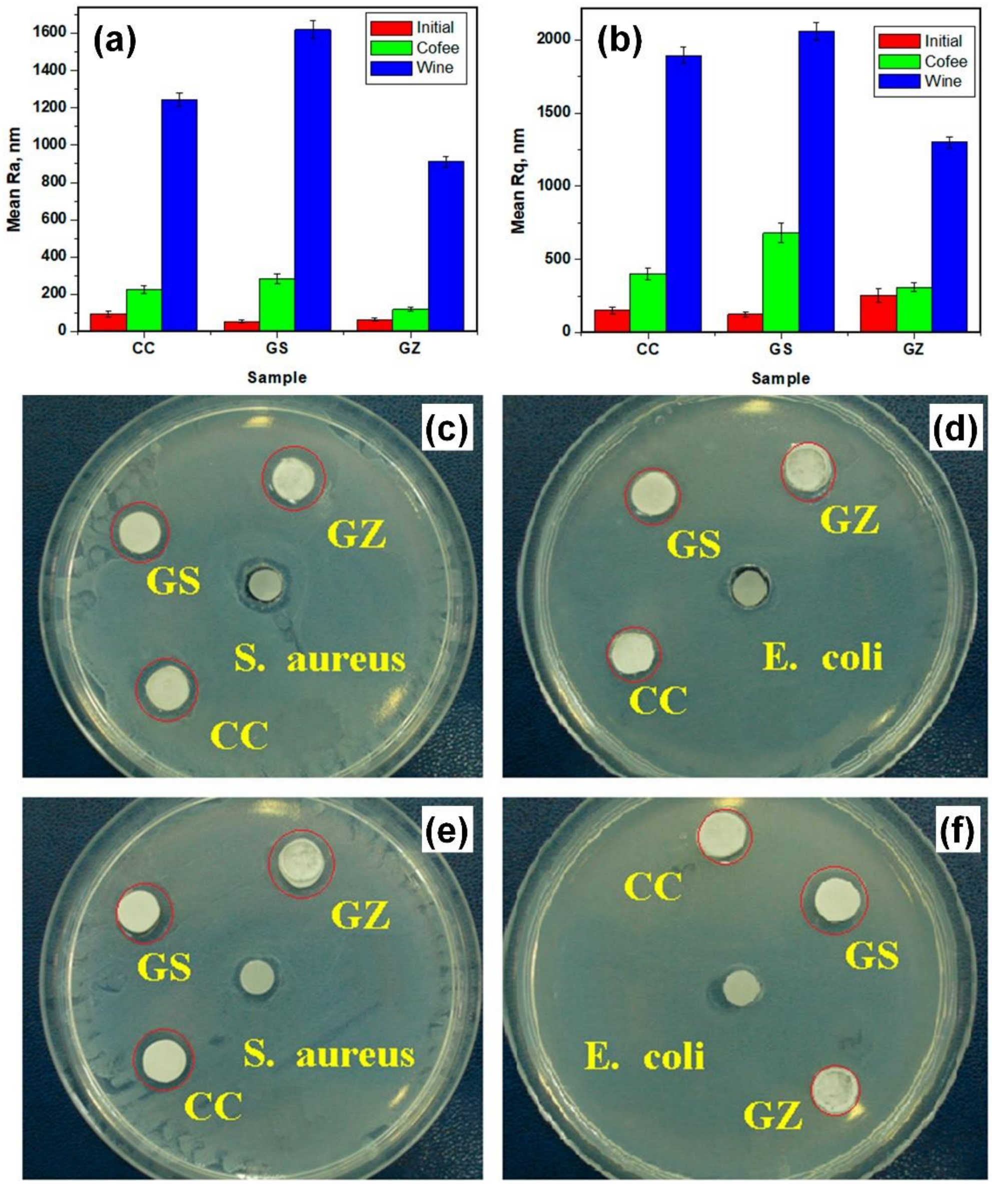

Figure 5. (a) Average roughness (R_a) and (b) root mean square roughness (R_q) of dental composites filled with GO-hydroxyapatite-SiO_2 (GS), GO-hydroxyapatite-ZrO_2 (GZ), and hydroxyapatite-Ag (CC) filler systems. Additionally, it includes disk diffusion (c, d) and well diffusion (e, f) antibacterial activity assays of dental composites [56].

aureus (*S. aureus*) and *Escherichia coli* (*E. coli*) were influenced by the presence of GO nanosheets in the filler systems. The dental composite filled with the GS filler system exhibited the strongest antibacterial activity against both bacteria strains, as shown by the size of the bacteria growth inhibition zones in Figure 5. Additionally, the dental composite filled with the GZ filler system showed moderate antibacterial activity compared to the GS and CC filler systems. The authors proposed that by using various oxides (SiO_2 or ZrO_2) as additional fillers in the system, the antibacterial activity of the dental composites filled with GO nanosheets could be further enhanced [56].

As previously mentioned, the main challenge in using graphene-based nanostructures in dental composites is their biocompatibility [57-59]. Several *in vitro* and *in vivo* studies have focused on the biocompatibility of various types of graphene-based nanostructures in dental applications [60-62]. However, most biocompatibility studies on graphene-based nanostructures found in the literature are related to bone regeneration due to the novelty of the subject [63-65].

Experimental studies on dental composites containing 0.3% rGO/ZrO2 and 0.3% TiO2-Ag-GO fillers in a Bis-GMA/UDMA/TEGDMA matrix showed that there was no significant toxicity or inflammation (Figure 6). However, the results indicated some oxidative stress in groups filled with GO-loaded dental composites. The presence of fibrous reaction near the test material suggests a healing response of body tissues around implants or foreign materials [66]. However, the complete picture in this study suggests that the implantation of the graphene-loaded dental composite is well-tolerated and is associated with a normal healing response, including inflammation and bone formation [66].

5. Conclusions

This review paper provided an in-depth overview of the latest research on the development of graphene-based dental composites, covering their structures, mechanical properties, and biocompatibility. The overview confirmed that incorporating graphene-based nanosheets can significantly improve the mechanical

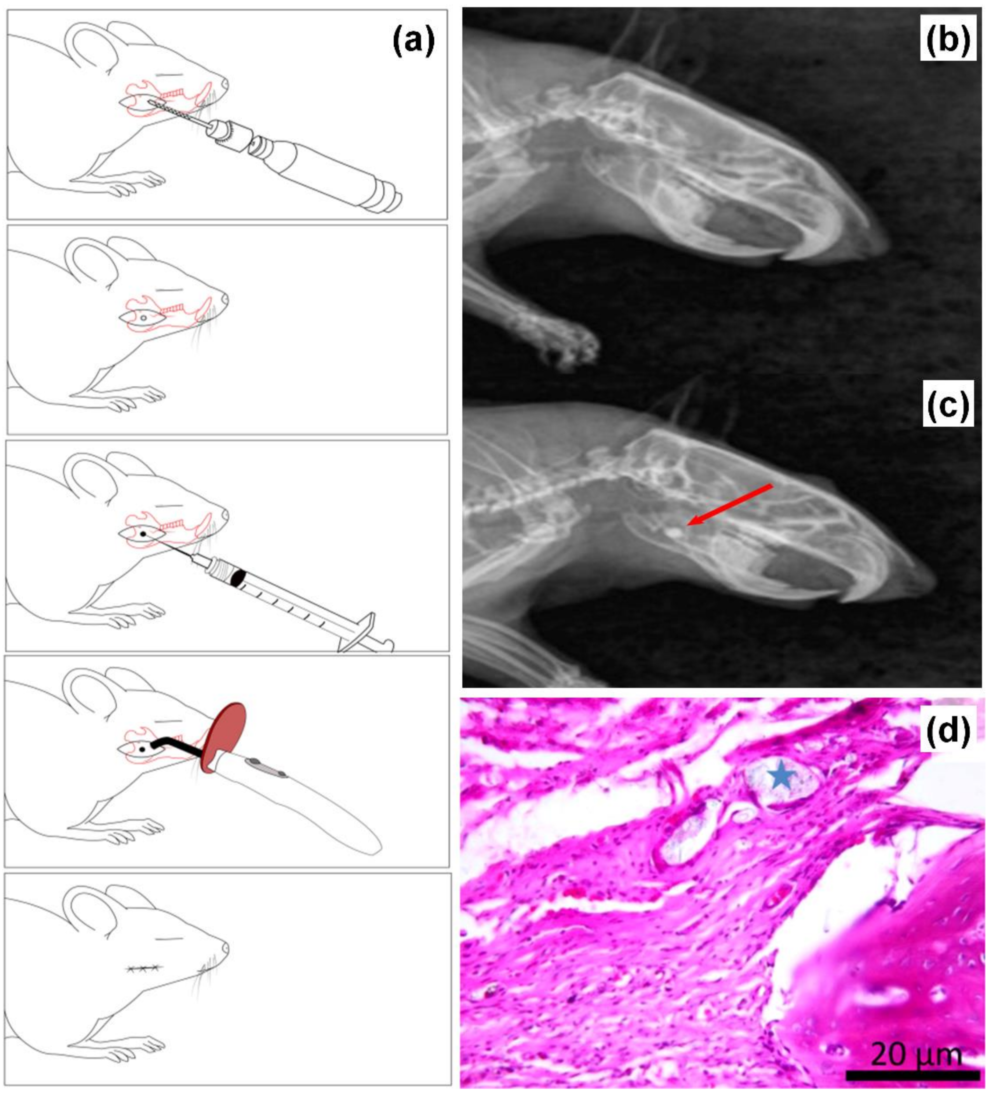

Figure 6. (a) The protocol followed for the in vivo experimental studies on graphene dental composites, the X-ray images of (b) control and (c) group filled with the graphene dental composite, and (d) fibrous reaction near the test graphene dental composite [66].

properties of dental composites. Additionally, the discussions suggested that the biocompatibility of such dental composites has been proposed in the literature. However, the literature had limited content on graphene-based nanostructures and their types, making it difficult to reach a clear conclusion on the biocompatibility of graphene-based dental composites. Therefore, further exploration into other types of graphene-based nanostructures, particularly concerning the biocompatibility of these potential dental composites, seems necessary.

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Conflict of Interest

The author declares no conflict of interest.

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