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Graphene loaded into dental polymers as reinforcement of mechanical properties: A systematic review

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Graphene compounds are incorporated into polymers in order to improve their mechanical properties and in dentistry this modification favors the clinical performance of these materials. The aim of this review was to evaluate graphene compounds, their concentrations, and their effect on mechanical properties as flexural, tensile, and compressive strength and hardness of polymethylmethacrylate (PMMA) and polyether-ether-ketone (PEEK) for dental application. The search was carried out in two steps in PubMed/Medline, Embase, Scopus, and Web of Science databases. The eligibility criteria included studies that incorporated pure graphene compounds into dental polymers and evaluated their mechanical properties. Were found 4984 results, of which 11 articles were included in this review. Graphene compounds: graphene oxide (GO), reduced graphene oxide (rGO), and graphene nanoplatelets (GNP) were incorporated into PMMA and PEEK, in concentrations ranging from 0.1 to 10 wt%. Concentrations lower than 0.75 wt% of GO in PMMA and 1 wt% of GNP in PEEK resulted in increased flexural, tensile, compression strength, and hardness of these polymers. It was concluded that the incorporation of graphene compounds in low concentrations increases dental polymers' mechanical properties.

Contents

1. Introduction

Graphene in its 2D structure, composed of only one layer of atoms, was first reported by Novoselov et al., 2004 and enabled innovations in materials [1,2] due to its biocompatibility, flexibility, and resistance. [3] Graphene compounds are obtained from their oxidation (graphene oxide) or reduction (reduced graphene oxide), and applied in science, technology, biomedicine, and dentistry, through metal functionalization, organic ligands, and polymer matrix in order to improve your properties. [3–5].

Graphene oxide (GO) is obtained by oxidizing graphite, with hydrophobic and hydrophilic parts of the molecule, [6,7] and oxygen functional groups that facilitate the chemical association of graphene with other compounds. [8] GO is water soluble, has a high contact surface area, electrical and optical properties, and chemical reactivity, which allows its multifunctionality and wide technological applicability. [9] The reduced graphene oxide (rGO) synthesized by the reduction of GO, starts to present free radicals that decrease the presence of oxygen, which makes it more stable due to

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Graphene composites	Presence of oxygen(O)	Solubility	Surface area	Electrical conductivity
GO	●●●●●	●●●●●	●●●●●	●●●●●
rGO	●●●○○	●●●○○	●●●○○	●●●●●
GNP	○○○○○	○○○○○	●●●●●	●●●●●

Fig. 1. Physicochemical properties of graphene compounds. Legend: The schematic Fig. 1 represents the inherent characteristics of graphene oxide (GO), reduced graphene oxide (rGO) and graphene nanoplatelets (GNP). It is understood that the greater the amount of filled circles, the greater dominance of such characteristics, i.e., the presence of oxygen (O) and the solubility of the compounds are in dominant amounts in GO, followed by rGO and absent in GNP. As for surface area, the dominant amount in GO and GNP, followed by rGO. For the electrical conductivity, all compounds present similar characteristic.

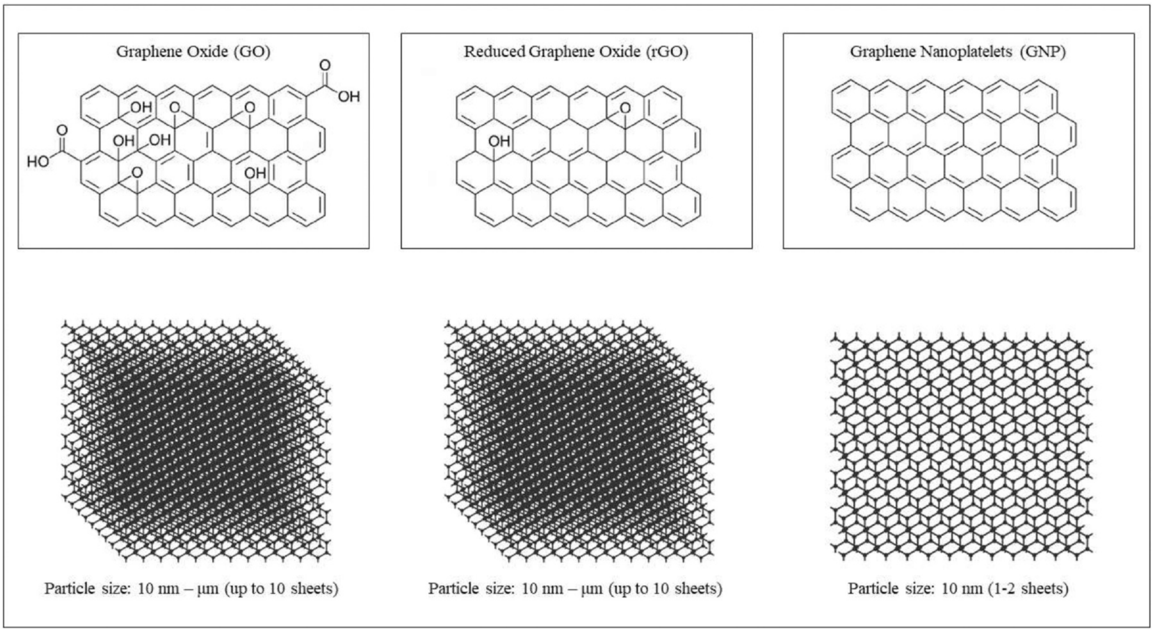

Fig. 2. Differences between graphene compounds.

the greater amount of carbon-carbon bonding, in addition to high electrical conductivity. [7,10] The reduction of oxygenated functional groups, however, can result in a decrease in the surface area potential and solubility of the reduced compound. [2] Graphene nanoplatelets (GNP) are obtained by exfoliation of graphite, have high electrical conductivity and consist of one to two layers of the exfoliated compound, containing only carbonic bond, with lateral dimension in the nanometer range and thin thickness. [11] Because they do not have the presence of oxygen, they are not soluble in water, but present a large surface area given by the dispersion of nanoparticles (Fig. 1). [12,13] The particle size of the compounds can influence their functionalization, because when they are larger they tend to agglomerate more easily. [14] The size of GO and rGO particles varies depending on the production method and synthesis conditions, with dimensions ranging from nanometer to micrometer. [2,15] On the other hand, GNP have nanometric dimensions, with thicknesses ranging from 0.34 to 100 nm and lateral diameters from 5 to 500 nm (Fig. 2). [13,16].

Graphene compounds, when added to implant materials, such as titanium [17] and zirconia, [18,19] favor osseointegration [17–19] and reduce microbial adhesion, [17] and in dental polymers, provide antimicrobial efficacy [20] and increase mechanical properties [11,21] such as wear and fracture resistances. [12,20,22–24] Polymers with a dental application must be biomechanically resistant to masticatory forces and parafunctional habits that occur in the oral cavity. [25] These forces are commonly evaluated by flexural, tensile, compression, and hardness resistance tests. [22].

Polymers such as Polyether-ether-ketone (PEEK) and Polymethylmethacrylate (PMMA) are synthetic materials with controllable mechanical, physical and chemical properties, which make them ideal for medical and dental applications. [26] The PEEK family polyaryletherketone (PAEK), presents advantages over the polymers already studied, with future perspectives to improve the quality and satisfaction of dental treatments for being stable at low temperatures and above 300 °C, which makes it resistant to structural damage. [27] It was first applied in dentistry to make implants, [28] due to its high resistance to fatigue, wear and ability to withstand mechanical stress and is currently used for fixed partial dentures [29] and removable partial dentures, from the making of clasp [30] to infrastructure. [31] The material is also color-characterizable, which favors clinical esthetics. [32] The PMMA is used for the fabrication of prosthetic devices, occlusal and orthodontic appliances, due to its favorable physicochemical and mechanical properties for use in the oral cavity, in addition to its cost-effectiveness, easy handling, acceptable esthetics and stability. [33–37].

Studies that incorporated graphene compounds into dental polymers to positively alter their mechanical properties [11,12,20–24,38–41] observed that, depending on the concentration and method of incorporation, these properties are negatively affected. [11,21,38,41] Thus, due to the versatility and graphene potential application in dentistry, this review evaluated which graphene compounds incorporated into dental polymers provide the increase in mechanical properties.

Table 1
The Population, Intervention, Comparison, Outcome, and Study Design (PICOS) strategy for this systematic review.

PICOS	Description
Population	Polymers for dental application
Intervention	Graphene compounds incorporation
Comparisons	Polymers without intervention
Outcome	Mechanical properties evaluation
Study	In vitro experimental studies

2. Material and methods

Based on the PICOS strategy, the review question was "Does the incorporation of graphene compounds in dental polymers improve the mechanical properties of these materials?" (Table 1). This review was performed according to the preferred reporting items for systematic reviews and meta-analyses (PRISMA) [42] and registered in Open Science Framework (osf.io/ensz8).

Eligibility criteria included articles that incorporated pure graphene compounds into dental polymers and evaluated their mechanical properties. Articles that addressed: a) graphene associated with other compounds, b) polymer coated with graphene compounds, c) polymers without dental application, d) animal studies, clinical trials, systematic reviews, book chapters, short communications, conference abstracts, case reports, and personal opinions were excluded.

The databases used for the searches were Pubmed/Medline, Web of Science, Embase, and Scopus. An initial search was performed in August 2022 using the terms ((polymer OR "dental polymer") AND (incorporation OR addition OR modified OR inclusion) AND (graphene OR "graphene oxide" OR nanographene OR "nanographene oxide") AND ("mechanical properties" OR hardness OR "flexural strength" OR "compressive strength" OR "diametrical compression")), applying filters "article" and "article in press" in Web of Science and Scopus databases. A complementary search was performed in the same databases in September 2022 using the terms ((methacrylate OR polymethylmethacrylate OR PMMA OR PEEK OR "denture base") AND (incorporation OR addition OR modified OR inclusion) AND (graphene OR "graphene oxide" OR nanographene) AND ("mechanical properties")). The reference list of the included studies was analyzed, in addition to experts in the subject, in order to include more studies.

The studies were initially selected by two independent authors (B.D.S and A.B.V.T), reading the title and abstract in Rayyan software. The articles chosen for full-text reading were included or excluded according to eligibility criteria. In cases of disagreement, a third author was consulted (A.C.R). The data were extracted to a table addressing the following topics: polymer, graphene compound, concentration, incorporation method, mechanical properties, and outcome.

Risk of bias evaluation was performed using the Joanna Briggs Institute (JBI) tool, adapted for this systematic review, in which 2 of the 9 questions were excluded for not being suitable for in vitro studies. Questions were classified as high or low risk, or unclear, according to the tool's instructions.

3. Results

Fig. 3 shows the study selection results. In the initial search, 4490 results were found, of which 712 were duplicates, and after evaluating the title and abstract in 3778 articles, 4 were selected for full reading: 2 were included in this review, and 2 were excluded (one for not being applicable to dentistry and the other for associating graphene with another compound). In the complementary search, 494 results were found, of which 179 were duplicated, 315 articles were evaluated by title and abstract, and 7 were selected for reading in full: 4 were included and 3 were excluded for not showing dental

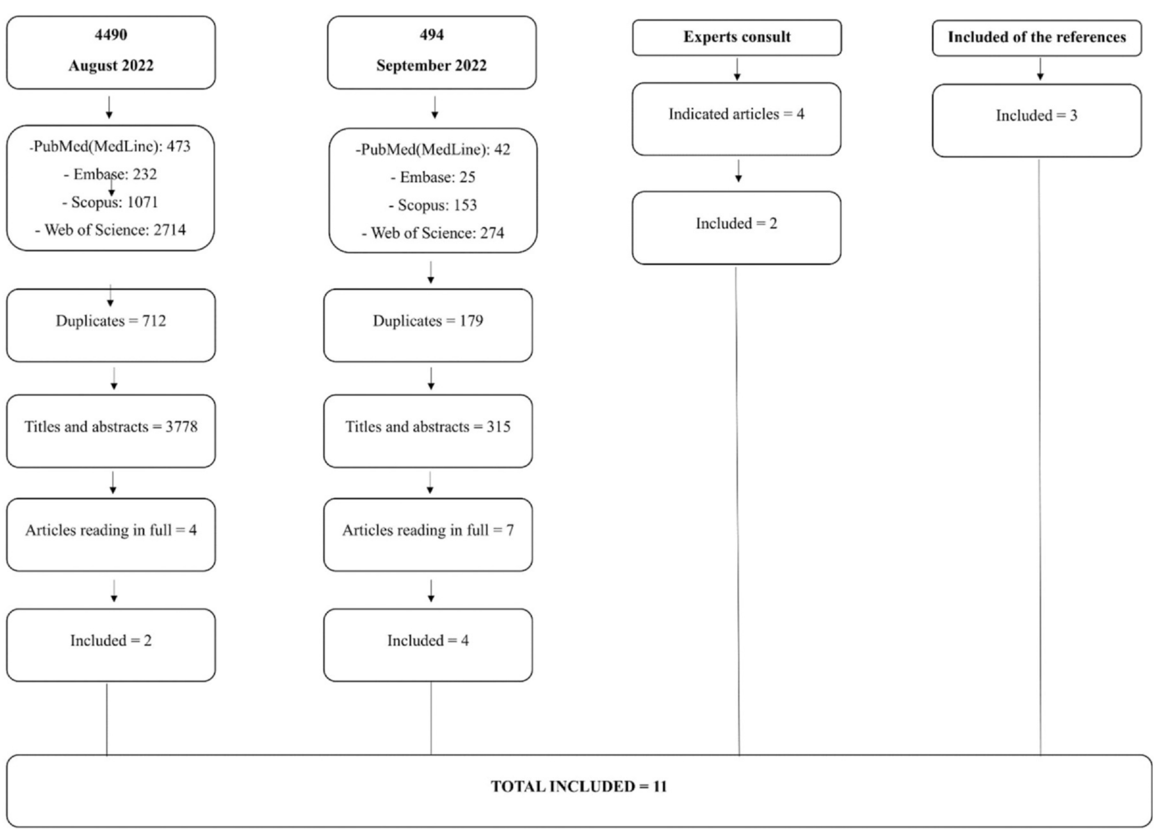

Fig. 3. Study workflow.

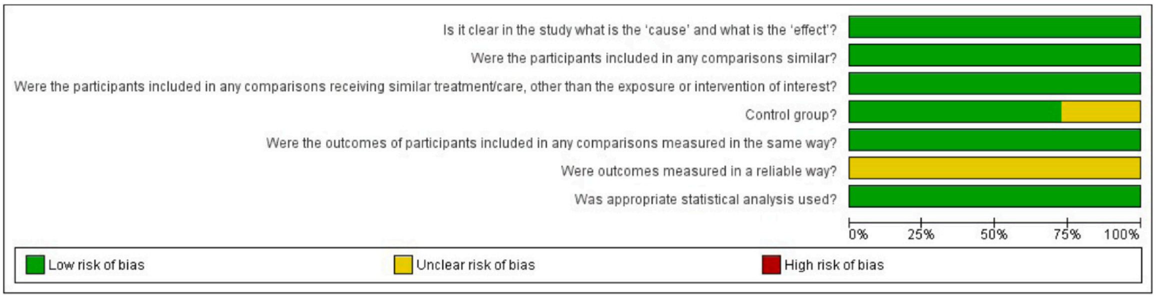

Fig. 4. Risk of bias general analysis of the included studies.

application. An expert indicated 4 articles that were read in full and 2 were included (the other 2 were excluded for associating graphene with another compound). When analyzing the reference list, 3 more articles were included, totaling 11 articles included in this review.

The risk of bias results is shown in Figs. 4 and 5. Most of the studies showed a low risk of bias. Agarwalla, Malhotra, Rosa (2019), Ciocan et al. (2021), and Çakmak et al. (2022) were not clear regarding the control group description and the concentrations incorporated. None of the included studies were clear about the criteria "Were outcomes measured in a reliable way?" as the authors did not report whether the outcome was assessed by a single researcher.

Due to data heterogeneity, a descriptive analysis of the results was performed without meta-analysis. Table 2 shows detailed information regarding the included studies. The studies included in this review evaluated polymers used in dentistry as Polymethylmethacrylate (PMMA) [20–24,38–41] and Poly-ether-ether-ketone (PEEK) [11,12] incorporated with graphene oxide (GO), [20–24,39–41] reduced graphene oxide (rGO) [38] and graphene nanoplatelets (GNP) [11,12] in concentrations ranging from 0.1 to 10 wt%.

Mechanical properties evaluated on the modified materials were hardness, [20,39–41] flexural, [11,12,20–23,40] tensile, [12,38] and compression strength. [12,24].

Ghosh, Shetty (2020) observed a decrease in the flexural strength of PMMA with the incorporation of 0.5 wt% of GO, unlike Lee et al. (2018), who observed an increase in the flexural strength of PMMA incorporated with 0.5 wt% GO. Di Carlo et al. (2020) and Çakmak et al. (2022) also reported an increase in CAD-CAM PMMA- modified with GO flexural strength. Jiang et al. (2021) evaluated PEEK-modified with GNP mechanical Properties and observed an increase in flexural, tensile, and compression strength when 1 wt% was incorporated. In the study by Tripathi et al. (2013), values above 1 wt% of rGO negatively affected PMMA tensile strength. As for Levenez et al. (2021), PMMA incorporated with 0.1, 0.5, and 0.75 wt% GO resulted in an increase in compressive strength. Only in Lee et al. (2018) study, did there a significant increase in PMMA-incorporated with GO hardness, in disagreement with studies by Agarwalla, Malhotra, Rosa (2019) and Khan et al. (2022), who did not show significant changes in PMMA-incorporated with GO hardness; and Ciocan et al., (2021), in which a reduction in CAD-CAM PMMA-modified with GO hardness was observed.

4. Discussion

This review evaluated the effects of the GO, rGO, and GNP incorporation into PMMA and PEEK for dental application, and found that concentrations of 0.1–0.75 wt% GO increased the PMMA compression strength, [24] and concentrations up to 0.5 wt% improved the PMMA hardness and flexural strength. [20] In PEEK, the addition of 1 wt% of GNP positively changed the polymer's flexural, tensile, and compressive strength. [12] The mechanical properties improvement is desired once increase the material longevity in clinical practice, by resisting masticatory forces and parafunctional habits, which prevents fractures or deformations.

Regarding the type of graphene compound used, GO stood out for its performance when incorporated into PMMA. The increase in PMMA mechanical properties with the GO incorporation [20,22,23] is due to functional oxygen groups that favor the chemical bond of graphene with the polymeric matrix. [8] In contrast, in Ghosh, Shetty (2020) study, GO-0.5 wt% incorporated into PMMA negatively affected flexural strength, which according to the authors was due to the compound agglomerations in the resin. The particle aggregation affects the chemical bond between the GO molecule and polymeric matrix, which results in pore formation and failure susceptibility. [21] One way to improve the GO dispersion is to add it to the polymer through ultrasonication since the vibration allows better homogenization of the particles in the solution. [43] When not performed, the compounds are not uniformly distributed over the entire surface of the modified polymer, which interferes with the reticulation process of the polymeric matrix and negatively influences the mechanical performance. [20].

Graphene compounds were incorporated into commercially purchased PMMA CAD-CAM blocks. [22,23,40,41] Despite the incorporated concentration not being informed by the manufacturer, GO addition improved the flexural strength, [22,23] and negatively affected the PMMA hardness. [41] The technology of the compound introduced directly into the CAD-CAM block, when compared to other manual incorporation methods, makes the GO dispersibility more homogeneous throughout the PMMA, which consequently reduces the agglomeration of the particles. However, according to Agarwalla, Malhotra, Rosa (2019), GO addition in CAD-CAM PMMA block does not promote a significant improvement in the mechanical properties evaluated.

Biomaterials, when used in high concentrations as reinforcements for polymeric structures, easily agglomerate and change the material's performance since they form stress concentration points, which favors fracture and device deformation. [12,40] The incorporation of GO high concentrations (1 and 2 wt%) caused a reduction in the PMMA flexural strength and hardness, forming agglomerates that resulted in high viscosity of the mixture. [20] The authors reported that the GO initial dispersion in water was homogeneous, however, when incorporated into the PMMA liquid and later into the powder, the GO particles did not disperse properly. [20] To overcome this challenge, the study recommends the GO particles silanization or incorporation of carboxylic groups. [20].

The rGO when incorporated into PMMA in concentrations greater than 1 wt%, negatively affects tensile forces, [38] despite reports in the literature that it presents a more favorable molecule to establish carbon bonds with the polymer, due to the lower presence of oxygen compared to GO. [7,10] There was also a reduction in PMMA performance with the addition of concentrations greater than 1 wt% of rGO due to particle agglomeration, which results in polymer fragility [38] and compromises its clinical durability.

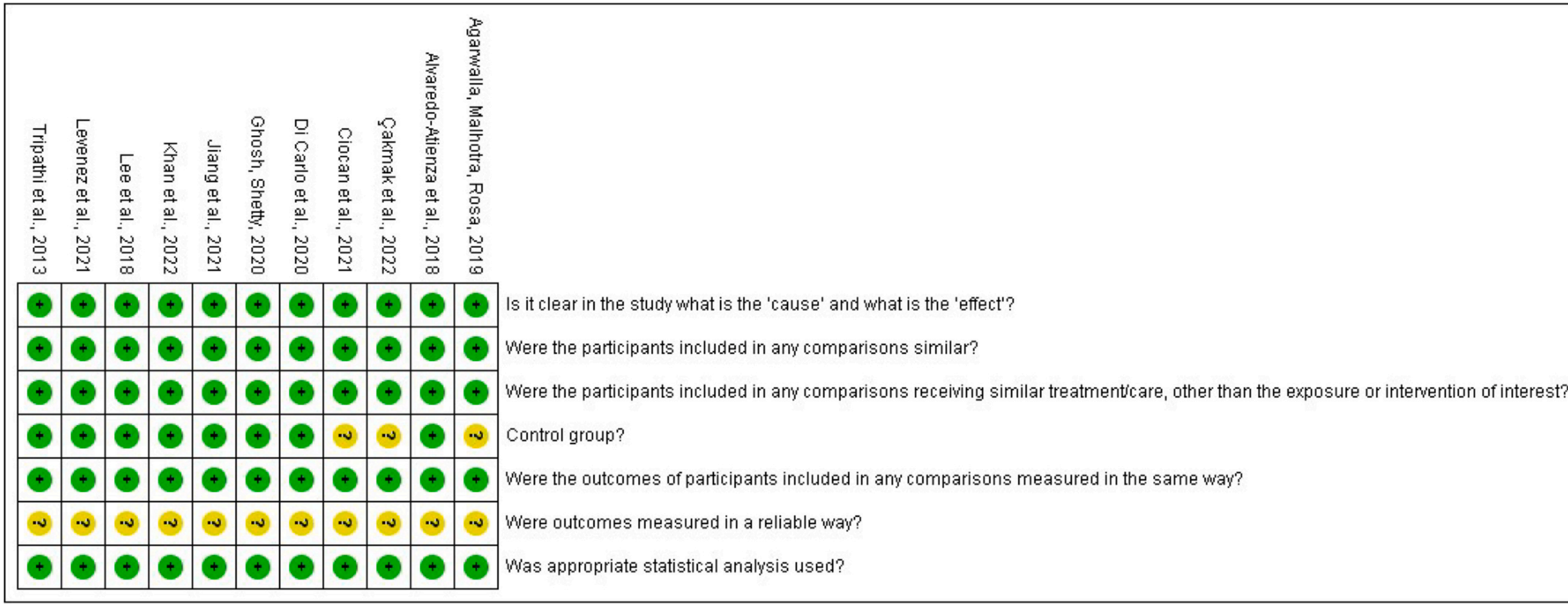

Fig. 5. Analysis of the risk of bias per study.

Table 2
Summary of included studies.

Author and year	Polymer	Graphene compound	Graphene concentration	Incorporation method	Mechanical properties evaluated	Conclusion
Agarwalla, Malhotra, Rosa (2019)	Polymethylmethacrylate (PMMA)	Graphene oxide (GO)	Not informed	Commercial CAD-CAM resin block	Hardness and flexural strength	GO incorporation into PMMA did not influence the evaluated properties.
Alvaredo-Atienza et al. (2018)	Poly-ether-ether-ketone (PEEK)	Graphene nanoplatelets (GNP)	0.5, 1, 5, and 10 wt%	Injection molding	Flexural strength	The concentrations of 0.5%, 1%, and 5% of GNP did not influence the flexural strength of PEEK, and 10% of GNP caused a flexural strength decrease.
Çakmak et al. (2022)	PMMA	GO	Not informed	Commercial CAD-CAM resin block	Flexural strength	GO incorporation increased the PMMA flexural strength.
Ciocan et al. (2021)	PMMA	GO	Not informed	Commercial CAD-CAM resin block	Hardness	GO incorporation decreased the PMMA hardness.
Di Carlo et al. (2020)	PMMA	GO	Not informed	Commercial CAD-CAM resin block	Flexural strength	GO incorporation increased the PMMA flexural strength.
Ghosh, Shetty (2020)	PMMA	GO	0.5 wt%	Incorporated into the liquid	Flexural strength	GO incorporation (0.5 wt%) decreased the PMMA flexural strength.
Jiang et al. (2021)	PEEK	GNP	0.1, 0.5, 1, and 5 wt%	Injection molding	Flexural, tensile, and compression strength	The incorporation of 1% GNP into PEEK provided higher flexural, tensile, and compression strength.
Khan et al. (2022)	Soft denture liner PMMA based	GO	0.1, 0.3, and 0.6 wt%	Incorporated into the liquid	Hardness	GO-incorporated concentrations did not influence denture liner hardness.
Lee et al. (2018)	PMMA	GO	0.25, 0.5, 1, and 2 wt%	Incorporated into the liquid	Hardness and flexural strength	GO-concentrations ≥ 0.5 wt% increased the PMMA hardness and flexural strength.
Levenez et al. (2021)	Bone cement PMMA based	GO	0.1, 0.5, and 0.75 wt%	Incorporated into the liquid	Compression strength	GO incorporation increased the bone cement compression strength.
Tripathi et al. (2013)	PMMA	Reduced graphene oxide (rGO)	0.1, 0.5, 1, 1.5, and 2 wt%	Incorporated into the liquid	Tensile strength	rGO-concentrations ≥ 1 wt% decrease PMMA tensile strength. Lower concentrations caused no changes.

Graphene synthesized in nanoplatelets form has a structure that favors the polymer's reinforcement due to its smaller number of layers, which allows better dispersibility and insertion of the compound between the polymer chains, thus increasing the rigidity of the material. [21] Result observed by Jiang et al. (2021), in which the incorporation of 1 wt% of GNP into PEEK showed a 10% increase in flexural, tensile, and compressive strength values when compared to pure PEEK. Alvaredo-Atienza et al. (2018) reported that in low concentrations, such as 0.5 wt%, the fracture toughness of the GNP group was 150% higher compared to the control group, reducing the fracture risk. On the other hand, the authors observed a decrease in flexural values when high concentrations of GNP (10 wt%) were added to PEEK. [11] The increase in concentration leads to a decrease in the fracture toughness value due to the presence of agglomerates not homogeneously dispersed in the PEEK matrix, which generates tension points in the material, structural defects that impair its clinical performance. [12].

The hardness of a polymer is desirable for it to be resistant to wear and deformations suffered in the oral cavity, especially when it is in contact with antagonistic materials. However, unlike the other properties, the addition of graphene compounds as a reinforcing filler to polymers has little effect on improving hardness. [7,40,41] Lee et al. (2018) observed that the incorporation of GO-0.5 wt% changed the PMMA hardness, but this change was not significant for clinical practice due to the lack of chemical bonding between the polymeric chains and GO.

Given this review findings, it can be inferred that the incorporation of graphene compounds, such as graphene oxide (GO) and graphene nanoplatelets (GNP), in low concentrations promoted improvements in mechanical properties; and in higher concentrations, agglomerate which impairs the mechanical performance of dental polymers. Incorporation methods such as ultrasonication, silanization, and association of carboxylic groups can improve the dispersibility of these particles and consequently their properties.

5. Conclusion

Graphene compounds at low concentrations such as 0.25, 0.5, 0.75 wt% of GO, and 1 wt% of GNP improve the mechanical properties of hardness, flexural, tensile, and compression strength of dental polymers and, in high concentrations, negatively affect the performance of these materials.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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