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Critical review of the article "Effect of Low Hydroxyapatite Loading Fraction on the Mechanical and Tribological Characteristics of Poly (Methyl Methacrylate) Nanocomposites for Dentures"

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Fouly, A., Mahmoud Ibrahim, A. M., Sherif, El-Sayed, M., FathEl-Bab, A. M.R., and Badran, A.H. (2021). Effect of Low Hydroxyapatite Loading Fraction on the Mechanical and Tribological Characteristics of Poly (Methyl Methacrylate) Nanocomposites for Dentures. *Polymers, 13(6), 857, 1-17. DOI: 10.3390/polym13060857*

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The study of new materials for biomedical application is still in the focus of scientific researchers because materials that satisfy completely biological needs have not been found yet. The recent investigation of Ahmed Fouly *et al.,* (2021) addresses the analysis of a nanocomposite broadly employed as denture base material: the poly (methyl methacrylate) (PMMA) reinforced with Hydroxyapatite (HA). The relevance of the research lies in the need of enhancing the mechanical and tribological composite properties to decrease material failures, disturbing of patients, as well as cost and duration of treatments. In this scenario, the authors evaluated PMMA/HA nanocomposites with different low loading fractions of HA and related the effect of those concentrations on the change in the specified properties.

In this work, the authors highlight the importance of PMMA as a biomaterial as well as its main limitations. They also reference some reinforce materials used to improve the mechanical properties of PMMA. With this base, authors propose the use of low HA loads (0, 0.2, 0.4, 0.6, and 0.8 wt.%); since, as they mentioned, it improves mechanical characteristics (surface hardness and toughness) of composites and does not produce negative reactions in the environment of the mouth. For this purpose, they prepare the nanocomposites with the traditional method for producing dentures, which encompasses: weighting of the dry MMA and HA nanopowders; mechanically stirring for achieving a homogenous solid mixture; adding liquid monomer, manual stirring at 28 °C and 55% of RH, molding of a cylindrical die through compression, and removing from the mold, according to the PMMA producer recommendations. For the characterization of the materials, the authors measured the density and void volume fraction of the PMMA/HA nanocomposites and determined their chemical composition using XRD analysis. A slight increase in density, when the concentration of reinforcing material increases, was reported. Moreover, thanks to the compression molding part of the production method and the low concentrations of HA employed by the authors, acceptable voids volume percentage (lesser than 3% with the highest load fraction) were obtained. By analyzing the XRD patterns of PMMA, HA, and nanocomposites, the authors suggested that composites have an amorphous nature.

On the other hand, the authors evaluated the shore hardness, elasticity modulus, compressive yield strength, relative toughness, and relative ductility as the main mechanical characteristics for their application as denture base material. Their results showed that HA increases the hardness of the PMMA achieving a maximum hardness increase of 9% with the highest concentration of HA. The mechanical properties that were most favored, with the addition of low ratios of HA, were Young's modulus and compressive yield strength; they revealed a tendency to increase with the rise of HA content, reaching peaks of up to 70.8% and 29.96%, respectively. The relative ductility and fracture toughness of the PMMA/HA nanocomposites exhibited similar tendencies reaching up to a 9% increase in ductility and 8% in toughness at 0.8% wt. of HA. The authors emphasize that these percentages of improvement have not been seen with other higher concentrations of HA, which can instead cause negative results, due to the agglomeration and deterioration of the material properties.

Suitable friction and wear resistance are very important characteristics of biomaterials for dental applications since friction can occur between the materials and different teeth in the mouth accelerating natural wear. To evaluate these characteristics, the authors performed dry sliding tribological assessments by the pin-on-disk reciprocating method (ASTM G 99-95) with a stroke of 50 mm, at a constant sliding speed of 0.4 m/s, under several normal loads (3, 6, 9, and 12 N), at a temperature of 27 °C and relative humidity of 60%. PMMA/HA composites (cylinders of 8 mm in diameter and 25 mm in length) were tested against rectangular disks of stainless steel and PMMA with 0.025 and 0.018 µm in surface roughness, respectively. Before these tests, the stainless steel and PMMA counterparts were prepared with acetone and dried with a heat gun to remove any impurity, whereas composites were washed ultrasonically and dried. Their results showed a tendency in the friction coefficient to decrease as the HA concentration increases, but to increase as the load increases with both counterparts. They observed maximum friction reductions of 20.7% against stainless steel (PMMA with 0.8% wt. of HA at 3N), and up to 25.5% against PMMA (PMMA with 0.8% wt. of HA at 9N). Similarly, analyzing the effect of the HA concentration on the wear response, an improvement in wear reduction was observed when incorporating HA nanoparticles, reducing weight loss when augmenting HA concentration but increasing with the rise of normal load. Authors attributed the observed tribological behavior of PMMA/HA nanocomposites to the enhancement of their load-carrying capacity due to the low HA contents. They validated their designation by analyzing the contact stress generated in the tribological contact by modeling in finite element the reciprocating frictional test. A tendency on the maximum equivalent stress and the wear layer thickness to decrease when the HA loading fraction increases was also observed; the highest reductions were observed in the PMMA/HA nanocomposite with 0.8% HA being 15% and about 9%, respectively. By SEM micrographs authors observed an important improvement in the related wear mechanisms on the surface of the nanocomposites, since the fatigue wear surface damage was notably decreasing with the increasing of the HA load fraction.

Finally, the investigation of Ahmed Fouly *et al*. shows extensive work in the characterization of PMMA/HA nanocomposites with low concentrations of HA as a reinforcing material. Nevertheless, there are still some issues to attend to enhance this work. In the tribological evaluation, although authors stated that they were conducting these tests to simulate the real conditions of PMMA, when used as a dental restorative material; it is important to note that they do not consider the *in situ* conditions of the oral cavity since saliva (a very important biological fluid for the maintenance of homeostasis, consisting of 99% water, and a variety of electrolytes, proteins, and immunoglobulins) can affect the mechanical and tribological properties of PMMA/HA composites in a real application. For this reason, the most successful examination could have been different, using tribological wet conditions with water or some simulated physiological fluid. Additionally, they do not contemplate the temperature factor that significantly affects the response to friction and wear of materials. In this case, a better estimation could be made at the temperature of the oral cavity, which is close to 37 °C, instead of 27°C. Furthermore, the surface preparation for the PMMA disks employed in the tribological investigation could be amended to avoid surface damage that alters the material significantly since it is well known that acetone degrades PMMA. Besides, the use of a heat gun could also generate thermal degradation of PMMA because it would generate temperatures between 100-500 °C, while the degradation temperature for PMMA is 92-126 °C.

Additionally , to obtain the best results of any biomaterial in clinical applications, the required standards for biological properties, during the characterization and testing phase, must be complied with; however, they were not studied in this work. For that reason, in the future, authors could also evaluate the biocompatibility of the PMMA nanocomposites with low HA loading fractions with different tests such as: in vitro (Cytotoxicity test, Membrane permeability test, Agar overlay method, Dentin barrier tests); in vivo (Mucous membrane irritation test, Skin sensitization, Implantation tests, testing on animal models); and usage (Dental pulp irritation tests, Periapical tissue damage, and Endodontic usage, Dental implantation in bone). The cytotoxicity could be also evaluated during the initial examination of the material by cytotoxicity tests. These are conducted to measure the cellular reaction by the material or its eluates, and to assess the cell viability, cell growth or function, enzyme activity, membrane integrity, and formation of reactive oxygen species before and after exposure of cells to the material. This could ensure that the low HA load fractions in PMMA composite allow good bioactivity (for example by an in vivo test by immersion of the nanocomposites into simulated body fluid and assessing the formation of HA on its surface), osseointegration (by in vitro assessments of the osteogenic differentiation through measurements of the activity of alkaline phosphatase; this result could also be obtained by in vivo tests through implantation of the material within the bone of an animal model followed by micro-CT measurements to evaluate the bone volume density), osteoinduction, and none foreign body reactions.

All of the above emphasizes the importance of a multidisciplinary study that contemplates both, mechanical/tribological and biological properties to decrease the gap between the new biomaterials and their applications in real conditions.

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Last Publications:

- Campos-Sanabria, V., Hernández-Sierra, M.T., Bravo-Sánchez, M.G., Aguilera-Camacho, L.D., García-Miranda, J.S., y Moreno, K.J. (2018). Tribological and mechanical characterization of PMMA/HAp nanocomposites obtained by free-radical polymerization. *MRS Advances, 3*, 3763-3768. DOI: 10.1557/adv.2018.622
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