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To evaluate and compare the mechanical properties and wetting ability of heat polymerized polymethyl methacrylate resin after reinforcement with halloysite nanotubes and Titania nanoparticles

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Abstract

To evaluate and compare the mechanical properties and wetting ability of heat polymerized polymethyl methacrylate resin after reinforcement with halloysite nanotubes and titania nanoparticles. A conventional heat-cured resin (DPI heat cure material) will be used as the matrix component while halloysite (group A) and titania (group B) nanoparticles as a reinforcing agent. Group C specimens will be fabricated with trevalon (Trevalon – HI). The specimens will be stored in distilled water overnight to simulate oral environment before performing the tests for determining the flexural strength (FS) and contact angle (CA). The contact angle data states that Halloysite reinforced PMMA is more hydrophilic. UTM analysis states that Flexure strength of trevalon highest amongst all the three group. Resin reinforced with halloysite had higher contact angle hysteresis hence greater retentive value and wetting ability while travelon had higher flexure strength.

Keywords: halloysite nanotubes, titanium nanoparticle, Trevalon, flexure strength, contact angle

Introduction

The numerous advantages of polymethyl methacrylate (PMMA) make it the most dominant polymer used as denture base material. The ease of processing, low cost, light weight, stability in the oral cavity and aesthetic properties are few advantages of this material. However, it has poor surface properties and weak mechanical properties including impact and flexural strengths. Therefore, to accomplish the expected outcome, resins should be reinforced using different materials to enhance their properties.

In this study two variants of nanoparticles namely, halloysite nanotube (HNT) and Titania/Titanium dioxide nanoparticles (TiO₂NP) will be reinforced into PMMA to produce hybrid resins. The diameters of HNTs are approximately in tens of nanometers, length ~200 nm to 1-2 μm. Hence it can be easily processed and can be dispersed uniformly into the resin matrix. The literature investigated the effect of TiO₂NP on some properties of PMMA but a comprehensive study on the overall performance of PMMA nanocomposite was not observed. Properties of TiO₂NP has proved to have antimicrobial properties. Moreover, both the materials are cost effective, biocompatible, chemically stable, free of toxicity, resistant to corrosion with high strength. Therefore, this study aims to investigate the effect of incorporation of HNTs and TiO₂NP into PMMA resin there after evaluating the wetting ability and mechanical property in terms of flexural strength, young's modulus of the hybrid resin

Materials and Method

Sample_grouping;

The study was carried out on 24 specimens (PMMA blocks). The specimens were categorized into 3 groups (n = 8) coded A to C. Group C was the control group (unmodified high strength acrylic resin specimens) fabricated using trevalon (Trevalon – HI). The specimens of the remaining two groups (A and B) was modified with addition of halloysite nanotubes and titania nanoparticles respectively.

Group A: PMMA (DPI heat cure material) reinforced with halloysite nanotubes. Group B: PMMA (DPI heat cure material) reinforced with titania nano particles. Group C: Trelalon (Trelalon – HI)

Sample preparation

Modelling wax patterns sized 65 mm × 10 mm × 10 mm

(figure 1a) were prepared using silicon moulds. The patterns were invested (figure 1b) and later on dewaxed to obtain plaster moulds (figure 2) of the desired size. The acrylic specimens were then prepared by packing the unmodified and modified acrylic resin into the moulds present in denture flasks.

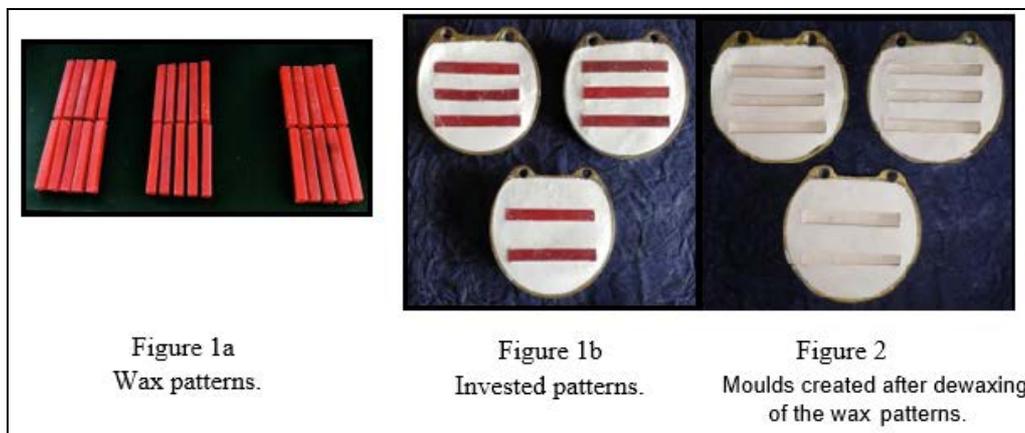


Fig 3: (a) Halloysite nanotubes 0.3 wt%. 60gms of PMMA (weight of the container 1.98gms.) Titania nanoparticles 1 wt%.



Fig 4: Nanoparticles being reinforced into PMMA by hand mixing.

For preparation of group A and B specimens, conventional heat-cured resin was used as matrix component, HNTs and TiO₂ nanoparticles as the reinforcing agent. The nanoparticles and PMMA was thoroughly mixed using a mortar and pestle for initial mixing and blending, followed by hand tumbling in a porcelain jar (figure 3 and 4) until an even colour was obtained. The resin powder was then mixed with monomer in a ratio of 2:1 by volume in a mixing jar with a tightly fitting lid.

The specimens were stored in normal water for a week to simulate oral environment before performing tests for determination of the flexural strength (FS) and contact angle (CA). The flexural strength was measured using a three-point bending test in a universal testing machine at a crosshead speed of 5 mm/min. Specimens were loaded until fracture occurred. From the stress-strain curve, the flexural strength and Young's modulus was calculated.

Wetting ability or the contact angle of the resin blocks will be assessed by evaluation of the contact angle hysteresis obtained by calculating the advancing and receding angle made by a drop of distilled water on the surface of the specimen.

Flexure strength measurement

Specimens were tested by 3-point bend test on Lloyd universal testing machine (Model multitest 10, Mecmesin, CENSE) at a cross head speed of 5 mm/min

For the 3-point bend test, a fixture was fabricated as shown in figure 5. The maximum force (F) necessary to produce fracture of the specimen was recorded in Newton (N). The flexural strength Q was calculated in (MPa) for all specimens using Equation.

where "F" is the maximum/fracture force in Newton (N), "l" is the distance between the two supporting points in (mm); "B" is the specimen width in (mm) and "H" is the specimen height subjected to bending in (mm).

$$Q = \frac{3FI}{2BH^2}$$



Fig 5: Utm analysis

Contact angle

Dynamic contact angle analysis was carried out to measure the advancing and receding contact angles made by the liquid on the specimens.

The fluid/media, in this study- distilled water, to be tested was dispensed by a syringe onto the specimen (Figure 6).

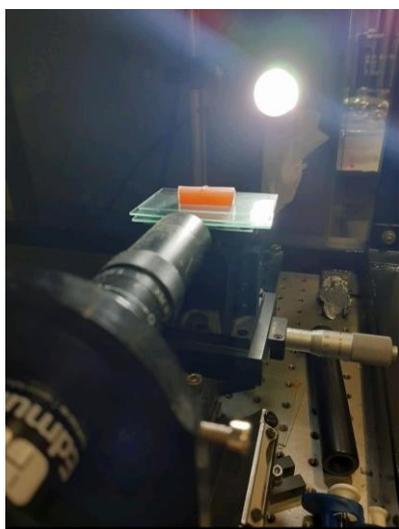


Fig 6: contact angle analysis.

The system allows for a standardized volume of fluid to be used on the specimen surface while measuring the advancing and receding contact angles. The system uses a high-speed camera to record changes of the drop contour which has been dispensed on to the specimen surface. The system's program determines the advancing and receding contact angles. (figure 7 and 8)

The advancing contact angles for the liquid used in this study were generally independent of time in the range of 1 to 10 minutes. Readings, therefore, were made 1 to 2 minutes after placement of the drop on the surface.

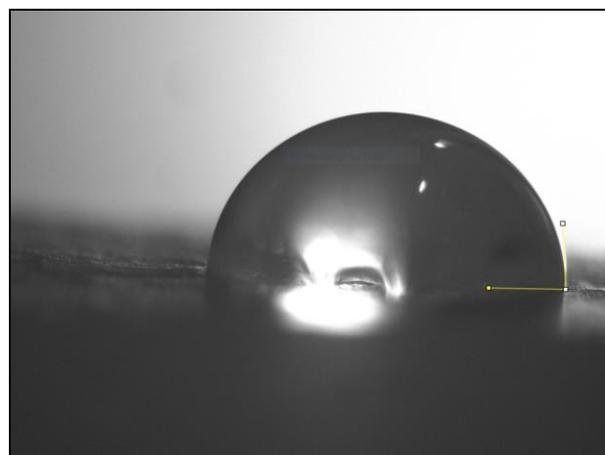


Fig 7: Advancing contact angle being recorded on group A (halloysite) specimen.

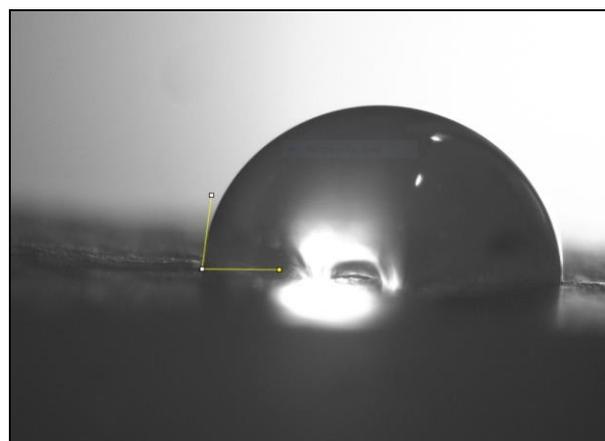


Fig 8: Receding contact angle being recorded on group A (halloysite) specimen.

Discussion

A common and inevitable problem faced while using acrylic denture base is its fracture. Therefore, various trials were done to bring about an improvement in the mechanical properties of PMMA. It can be achieved in the following three ways:

1. Replacing PMMA with an alternative material.
2. Chemically modifying it.
3. Reinforcing the PMMA with other materials like fibres or metals [24, 25].

This investigation principally aimed to assess possible changes in the mechanical properties (the flexural strength and contact angle) of two types of PMMA namely high impact acrylic resin (trevalon) and conventional PMMA modified by infusion of two types of nanoparticles (TiO₂ nanoparticles and halloysite nanotubes).

In nanotechnology different nano materials are used like carbon nanotubes, nanofluids, nanoparticles, nanoemulsions, nanocapsules. the results of these nano materials are not considered safe for humans and for the environment as well due to their toxic effects. Halloysite nanotubules (HNTs) are naturally occurring environmental friendly nanotubules and are cost effective. HNTs have high mechanical strength and modulus. Due to these properties halloysites are considered ideal for various applications. They are also used in the fabrication of high quality white-ware ceramics. Due to easy dispersability in polymer matrix, abundant availability and biocompatibility, HNTs are also used in different dental

materials.¹² On the other hand, TiO₂ nanoparticles are increasingly used owing to the impressive features as nontoxicity, chemical inactivity, low cost, high refractive index, antibacterial effect, corrosion resistant and high micro hardness. Furthermore, literature has also showed that nanoscale TiO₂ reinforcement agents bring new optical, electrical, physiochemical properties attained at very low TiO₂ content, which makes polymer-TiO₂ nanocomposites a promising new class of materials. It can be anticipated that it will be commercially beneficial for widespread fields^[22, 23]. Moreover, TiO₂ nanoparticles have been used as additives to biomaterials in order to induce antimicrobial properties. Antimicrobial activities of TiO₂ against *Candida albicans*, *Staphylococcus aureus*, *Pseudomonas aeruginosa*, *Escherichia coli*, *Lactobacillus acidophilus*, etc. have been proved by recent studies^[1].

To the best knowledge of the authors, the most suitable concentration for the addition of different nanometals to the acrylic resin that can lead to the best properties is doubtful until now. Addition of nanoparticles beyond a proven threshold makes it behave like a plasticizer thus causing slippage of molecules and compromising the mechanical properties of the resin.

Literature has proved that concentrations above 5% have led to massive changes in the colour of acrylic which decreases its aesthetic value and patient acceptance^[18].

Therefore, the two concentrations: 0.3% by weight of halloysite nanotube and 1% by weight of titania nanoparticles were selected^[11, 20].

Adhesion, defined as the attraction of unlike molecules, is one of the fundamental forces involved in denture retention. The wettability of a liquid to a solid surface plays an important role in determining adhesion^[25]

The wettability of a liquid to a solid surface can be studied by

measuring the contact angles formed between them. However, the fundamental requirement suggested for denture retention is contact angle hysteresis. Contact angle hysteresis (CAH) can be defined as the difference of the advancing (A) and the receding (R) contact angle for a contact line moving in an opposite direction at the same velocity^[28].

CAH is one of the most important and classic elements of wetting of liquid droplets in systems from centimeter to micrometer scales. Higher the contact angle hysteresis, greater is the retention.

It is perhaps most intuitively understood by looking at a droplet resting on a vertical substrate (picture)^[27].

The hysteresis of the contact angle ($\square A - \square R$) for pure liquids is mainly caused by surface flaws which could be geometric flaws/ surface roughness/the heterogeneous chemical composition of the surface. In case of polymers, the presence of liquid in contact with a solid may provoke the reorientation of surface groups, leading to contact angle hysteresis. In addition, contact angle hysteresis of polymer surfaces can be induced by the mobility and reorientation of surface polymeric chains^[16, 29].

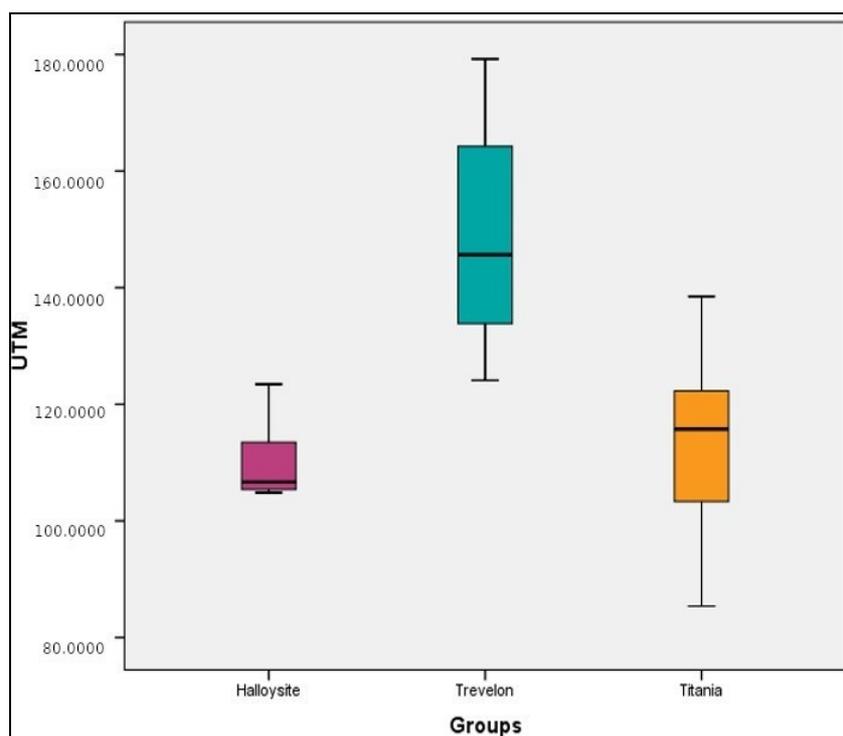
Result

Data was subjected to normalcy test (Shapiro-wilk test). Data showed non normal distribution. Hence non-parametric tests (Kruskal-wallis with post hoc Mann-whitney) were applied. Kruskal-wallis test showed statistically significant difference between groups of types of acrylic resin. The group C (trevalon) showed significantly highest mean flexural strength followed by group B (titania). The lowest flexure strength was seen in group A (halloysite). There were significant differences ($P < 0.05$) between studied groups.

Flexural strength measured for all specimens groups and values are listed in Table 1:

Table 1: Comparison of Utm Among the Groups Using Kruskal Wallis Test.

Group	Minimum	Maximum	Median	P value
Halloysite	107.86	123.4	106.6	0.001*
Travelon	124.11	179.2	145.6	
Titania	83.7	138.4	118.7	



Contact angle

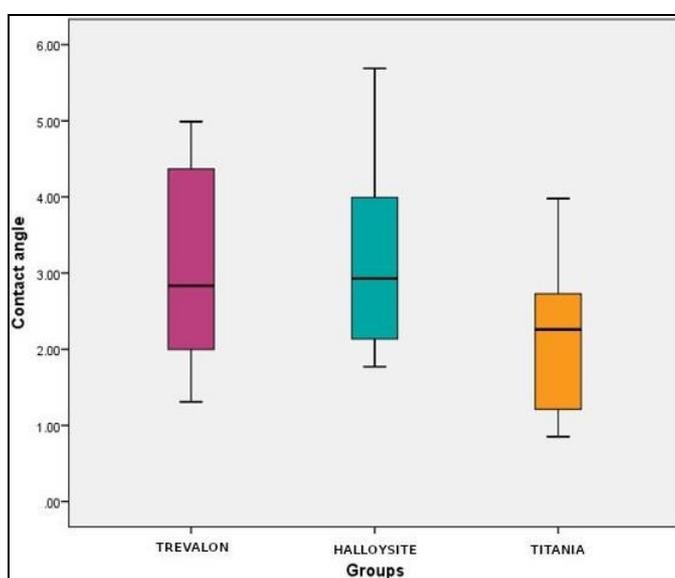
The data were statistically analyzed using Kruskal-wallis test. The results of the analysis are presented in tables 2 and graphically depicted.

The contact angle data (Table 2) clearly indicate that the heat-polymerized samples reinforced with HNTs are more hydrophilic (CAH- 2.92) than the samples made of trevalon (CAH- 2.83) and reinforced with TIO₂ nanoparticles (CAH- 2.25)

Highest hysteresis values were calculated for group A (halloysite) followed by group C (trevalon). Low-advancing and receding contact angles were demonstrated on group B (titania) denture base material.

Table 1: Comparison of Contact Angle (1a-1b) Among the Groups Using Kruskal Wallis Test.

Group	Minimum	Maximum	Median	IQR	P value
Trevalon	1.31	4.99	2.83	2.67	0.225
Halloysite	1.77	5.69	2.92	2.2	
Titania	0.85	3.98	2.25	1.82	



Conclusion

Within the limitations of this study, it could be concluded that: Flexure strength of group C, trevalon was demonstrated to be the highest amongst all the three groups hence provided better fracture resistance followed by group B (titania) and lastly group A (halloysite)

Group A, halloysite nanotube reinforced PMMA has better wetting ability and could possibly provide the best retention of the three denture base materials tested as it had the highest hysteresis value. This was followed by group C (trevalon) and lastly group B (titania).

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