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INVESTIGATION SOME MECHANICAL PROPERTIES OF PMMA COMPOSITE-REINFORCED CERAMIC POWDERS

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Abstract- Many researchers believe that the problem of adding reinforcing materials to a base material such as polymers to improve its properties still exists and requires further studies to make it possess the specifications of an advanced formula for dentures that have mechanical properties that are flexible in the ability to withstand plastic deformation and have sufficient strength and durability (fracture resistance). Or the brittle fracture when a crack occurs in it as a result of thermal conductivity, in addition to the stability of dimensions without a permanent change in its dimensions or surfaces when working, insoluble in liquids and oral saliva, color stability, does not cause the oral environment any taste or smell, stable in color and chemical solution in The oral environment, its biological materials are available in the environment so that it is inexpensive and with a good shelf life, all these specifications and others have achieved The interest of researchers, as a lot of research and studies have been prepared in this field, and the current study is an extension of those studies that preceded it in time. It is an experimental attempt in which PMMA was used and reinforced with nanomaterials to improve its mechanical attributes for the applications of the base of prosthetic dentures. The study aimed to know the effect of enhancing PMMA polymeric material with Al_2O_3, TiO_2 in improving its mechanical properties. To achieve the goal of the research:

1. The mechanical properties were determined in order (tensile, impact, hardness, creep, fatigue).
2. The polymer (acrylic resin) of PMMA type was selected as the base material for the kit, with weights (98%, 96%, 94%, 92%).
3. Powders (Al_2O_3, TiO_2) and a mixture of them (Al_2O_3 + TiO_2) were used as reinforcing materials. With fixed weights (2%, 4%, 6%, 8%), the mixing process was carried out according to the mixing law that determined the weights. Tests of mechanical properties were carried out according to the specific instructions for each test.

To analyze the results, analysis of variance, arithmetic averages, and lattice tests for homogeneity of samples were used. The following results were reached:

1- The homogeneity of the three samples prepared with the properties (tensile, impact, hardness, fatigue), as the addition of reinforcement materials have improved the resistance of the composite materials to stresses and fracture and has an appropriate hardness that resists bending and ease of formation and operation.

2- A statistically significant difference between the samples prepared with creep characteristic, and the sample prepared from the mixture (Al_2O_3 + TiO_2) got the best with this characteristic.

Keywords: PMMA, Acrylic Resin, Composite, Reinforcement, Plastic.

1. INTRODUCTION

Despite the advantages of dentures, there are defects in their use related to infections of the gums and the oral cavity, as it is not a permanent treatment. The life span of them is five to seven years, during which the shape of the jaw changes with the passage of years as a result of the receding bone, which makes it necessary to change the denture, about that the researchers' concerns Scientists have prompted them to think of alternative materials that tend towards natural materials such as (natural powders, pistachio shells, bamboo, coconut husks, corn husks, rice husks... etc.), and some of them used (polyethylene, glass fibers, TiO_2 SiO, ZrO_2, aramid, carbon hydroxyapatite, etc.) It has been used as a reinforcement material that extends life and changes many of the mechanical and physical attributes of the base material due to its structural advantages, as well as its low cost and high density [1, 2] .

The discrepancy in the results of research on dentures legalization, the researcher believes that the problem of using reinforcing materials and its impact on the properties of the compensatory structures of the basic materials for dentures still exists and requires more studies, and this current study is an experimental attempt that is related to the studies and research that preceded it in time that used a PMMA-based material and strengthened it with nanomaterials to boost its mechanical characteristics for the applications of the base of prosthetic dentures, and soon. The need to conduct the current study is summarized in the following points:

1) The importance of obtaining mechanical specifications and properties for the development of dentures that have mechanical properties, better biocompatibility, and flexibility in the ability to withstand plastic deformation of sufficient strength and durability (fracture resistance) or brittle fracture when there is a crack in them as a result of thermal conductivity, dimensional stability without change Permanent in its dimensions or surfaces when working, insoluble in oral liquids, color stability, does not cause the oral environment any taste or smell, stable in color and chemical solution in the oral environment, its biological materials are available in the environment so that it is inexpensive with a good shelf life, all these specifications And others have attracted the attention of researchers, as they have prepared a lot of research and studies in this field, [3, 4] as the study of both:

W.S. Chow, et al., (2008), E. Ellakwa, et al., (2008), I.N. Safi, et al, (2011), A. Hanan, et al., (2013), M.M. Mansour, et al., (2013) Khalarf (2013, 2016), B.S. Jasim, and I.J. Ismail, (2014), I.J. Ismaeel, et al., (2015), G.V. Kumar, et al, (2016), H.S. Fadhil, et al. (2017), W. Hussein, F.S. Hashim, (2017), Q.A. Hamad, (2018), Abdul Rahman, Hadil Jabbar (2018), Nada Naser Kadhim, (2019), Abdul Monem, Zainab Moaead (2020) [5-18].

These studies varied in studying the mechanical properties of polymeric composite materials that were prepared for their research, whether from a complete prosthesis or as basic materials for dentures. The present study is an extension of these studies to develop a PMMA-based nanocomposite material by enhancing its mechanical properties for applications of the base of prosthetic dentures.

2) Due to the urgent need to develop the health sector to serve the health of patients and the speed of their recovery, specialists from fields other than medicine needed to intervene to design and legalize. The vital devices and materials that are used to replace or replace lost and worn-out devices or tissues resulting from accidents or aging in the elderly. Therefore, engineers can be familiar with medical sciences from the anatomy and physiology of the human body and other medical sciences to understand the working mechanism of each system in it and to harness their knowledge and engineering specialization to develop these devices, Hence the need for an engineer who is partially acquainted with all these specialties on the one hand, and who can deal with doctors on the other hand, bearing in mind that he is not a substitute for any of them. Concerning dentistry, several studies have reported the improvement of the basic materials for PMMA dentures using additives such as fibers, fillers, and hybrid enforcement nanofibers. However, the majority of research was restricted to in vitro analysis without consideration of bioactivity or clinical consequences. According to the review's findings, PMMA can have some of its qualities enhanced by adding nanoparticles and a hybrid reinforcing system, but there is no perfect foundation material for dentures.

3) The current study is a scientific addition to the scientific and medical office that benefits primary and postgraduate students and specialists in the field of dentistry.

4) The method of rationing and the results of the current study may benefit dental specialists and dentists in their work.

2. SEARCH OBJECTIVE

The current research aims to study the effect of enhancing PMMA polymeric material with Al_2O_3, TiO_2 in improving its mechanical properties. To fulfill the goal of this research, it was needed to specify the mechanical properties such as (tensile, impact, hardness, creep, and fatigue).

3. PRACTICAL PROCEDURES

3.1. Base Material

The utilized polymer was PMMA as the base material for dentures. Acrylic resins have been widely used as a base material for dentures since 1937 [19] and this substance served as the foundation for around 98 percent of denture bases [20]. This material demonstrates a variety of favorable handling characteristics, including cheap cost, lightweight, simplicity of handling, ease of polishing, outstanding aesthetics, absence of toxicity, and biocompatibility in the oral cavity [21, 22].

Additionally, denture substances should not be utilized for extended periods while crawling under chewing loads [23]. Additionally, these materials must be strong and flexible enough to endure typical chewing pressures. As a crucial quality, the material's resistance to abrupt shocks brought on by impact pressures shouldn't degrade or alter the composition of the material in the aqueous environment of the mouth. To retain the teeth in a condition of occlusion while chewing and to lessen the unequal loading of mucus under the dentures, the polymer needs to be hard enough to prevent the creation of madness brought on by solvents in meals, beverages, or medications [4]. PMMA acrylic resins are usually available in powder or liquid form [24], Table 1 details its composition.

Table 1. Constitution of denture base substances (heat treatment)

Powder	Polymer	PMMA
	Initiator	Benzoyl peroxide (BPO) (0.5)
	Pigments	Salts of cadmium or iron or organic dyes
Liquid	Monomer	MMA
	Crosslinking agent	Ethylene glycol dimethacrylate (EGDMA) (10)
	Inhibitor	Hydroquinone (> 1%)

The main constituent of the powder is PMMA granules with up to 100 μm diameters. The methyl methacrylate (MMA) monomer, which contains an initiator, may be used to create these granules through suspension polymerization, in which the monomer is suspended as a droplet in water. The peroxide is then broken down at a higher temperature, which also causes MMA to polymerize into PMMA granules that dry to a free-flowing powder at room temperature. Crosslinking particles have also been used to minimize the chance of the denture developing tiny surface fractures after drying. The shelf life of the liquid component is increased by adding an inhibitor (hydroquinone) [24].

Consistent with the initiation method, which has a great influence on the attributes of the materials produced during manufacturing, the condition of the processing method affects the mechanical and physical attributes of the denture [21].

3.2. Reinforcing Materials

A composite substance is a substance involving two or more physical and/or chemical different phases, that are appropriately organized or dispensed. Usually, the synthesis material contains properties that are not represented by any of its constituents in separation from others. In general, Al-Mousawi et al. (2013) mentioned that the composite substance comprises two components [25]:

a) Matrix substance: this phase is continuous. It could be a polymer, ceramic, or metal matrix. In comparison to a metal-ceramic matrix, the polymer matrix has superior mechanical and thermal characteristics and may be reinforced with a much greater proportion of fibers. It is also simpler to make and less expensive. In the current study, thermosetting polymethyl methacrylate (PMMA) resin is used as a matrix material for the base of dentures produced by Spofa Dental. Where pure samples of PMMA were prepared with different weights (98, 96, 94, 92%).

b) Reinforcement substance: The reinforcement phase is the name given to the dispersed phase. There are many different types of reinforcing materials that may be used, including continuous fibers, short fibers, filaments, and particles, among others. Fibers, mineral fibers, ceramic fibers, and particles are all types of reinforcement used with organic fibers like carbon and Kevlar. Studies and research are continuing in this field to find suitable ways to improve mechanical properties and obtain the desired properties in actual applications. The search for natural materials such as reinforcement fillers is not only inexpensive but also able to reduce environmental pollution from the biodegradable property [26].

In the current study, two materials (Al_2O_3 and TiO_2) and a mixture of them (Al_2O_3 + TiO_2) were used. For the preparation of samples of industrial reinforcement materials (alumina and (TiO_2) and a mixture of the two materials. With fixed weights (2, 4, 6, 8%) as the mixing process was carried out according to the mixing law that determined the weights after repeating the process several times by adding the liquid substance and then the powders to the above samples to obtain Required particle size These industrial powders have a particle size in the order of 50 (µm, mesh white) for alumina (MW97.90) for titanium where the particles were added to the acrylic powder to produce composite samples.

4. MECHANICAL TESTS

4.1. Tension Test

It is an important engineering test, and it is used to determine the (stress-strain) curve for each composite sample, which in turn is used to evaluate tensile attributes like modulus of elasticity, elongation ratio at the breaking point, and tensile strength. The experiment was conducted at laboratory temperature utilizing a universal tensioning machine (LARYEE) available in the Materials Engineering Department at the University of Technology, with a (5kN) load capacity and a speed of loading of (5 mm/min). The tensile load was slowly applied until the fracture was reached. Samples are designed according to international standards (ASTM D638-03).

4.2. Impact Test (Shock)

In this test, Izod type of XJU series pendulum shock testing machine was used (Izod / Charpy), where the prepared samples were fixed at one end and held by a vertically protruding beam. The impact velocity of the pendulum used in this test was (5.5 J) and (3.5 m / s), respectively, and the impact test is performed at laboratory temperature without causing a slit on samples.

4.3. Hardness Test

The prepared samples (Al_2O_3+ TiO_2, Al_2O_3, TiO_2) were subjected to a hardness test of the type ((Shore (D)) suitable for measuring the hardness of acrylic resins. The vehicle was designed according to (ASTM, D2240) standard, with an average of five readings to obtain a high percentage Accuracy Load applied (50 Newtons) and depression time equals (15 seconds) The hardness value of each sample is recorded by calculating the average of five readings, as each sample was simultaneously tested in five different positions to obtain higher accuracy of the hardness values.

The sample is very important, as it should be very soft, especially in the test area. The hardness measurement is affected by many factors, including thickness, diameter, and the distance from the edge of the specimen, as it should not be less than 12 micrometers). These factors were taken into consideration when measuring the hardness value. The hardness value and indentation depth of this test were measured with a scale (0 to 100) on the scale's hardness number.

4.4. Creep Test

The measurement process is conducted by exposing the composite samples to constant tensile stress at the laboratory temperature. Then we measure the resulting strain in a period of (48) hours by taking four readings for each weight and finding the average to be one average for each weight and follow this procedure on all samples and under the same conditions in terms of stress time Its value, temperature, and sample dimensions.

4.5. Fatigue Test

The smoothing process was carried out for the composite samples using smoothing paper to get rid of surface defects and impurities adhered to them. All samples were designed with a height of (5 mm) and a surface area of (54 mm^2) for identical stress values. The samples were perforated before the examination with a hole of (1mm) diameter at a 45° angle. All the composite samples were numbered according to the type of material and the mixing ratio.

5. RESULTS AND DISCUSSION

To achieve the goal, it is necessary to verify the significance of the differences between the composite materials among themselves for revealing the specific mechanical attributes of the three composite samples so that we can know any composition and specifications of better properties that can be relied upon in the legalization of dentures. The following is an explanation of that.

5.1. Statistical Significance of Mechanical Properties

Descriptive statistics (mean, variance, standard error, minimum, and maximum) and one-way ANOVA (F-test) [27] were used to verify the statistical significance between the three composite subjects. Table 2 indicates the symmetry of the effect of adding reinforced nanomaterials (Al_2O_3 and TiO_2) and a mixture of (Al_2O_3 + TiO_2) to PMMA polymer on the tensile mechanical properties, and the effect, hardness, and fatigue, as there were no statistically significant differences between them, as the calculated t-value, respectively, amounted to (2.95, 2.97, 0.13, 2.41), which are lower percentages compared to the tabular value (4.26) at the level of statistical significance (0.05 = α) and confirms This is due to the homogeneity of the samples prepared with these characteristics, despite the difference in the averages obtained by those mechanical properties. While in the Creep feature, there were statistically significant differences between them, as the calculated t-value reached (14.26) compared with the tabular value and at the same level of statistical significance.

The curves of (the tensile, impact, hardness, creeping, and fatigue) were drawn for the three prepared composite substances that were enhanced with various particle sizes and various weight fractions of fine powders (Al_2O_3 and TiO_2 and a mixture of (Al_2O_3 + TiO_2) shown in Figures 1-5 where the linear part indicates Initially from these curves to the elastic behavior of the three samples, then changing them to a non-linear region where the samples are plastically deformed, And the plastic deformation continued until the occurrence of fractures, the largest of them all at a mixing ratio (4%). The behavior of the mechanical properties curves depends on some factors, including the physical and mechanical properties of the composite components, in addition to the strength of the interface between the matrix and the reinforcement material.

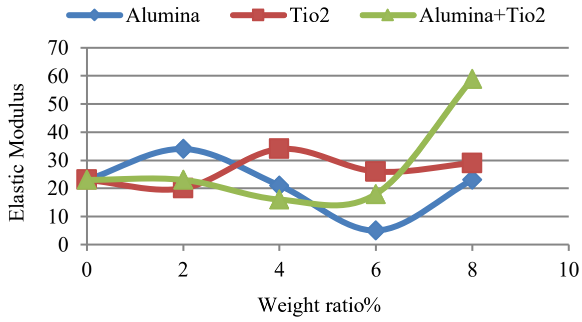


Figure 1. Elastic modulus of PMMA composite material

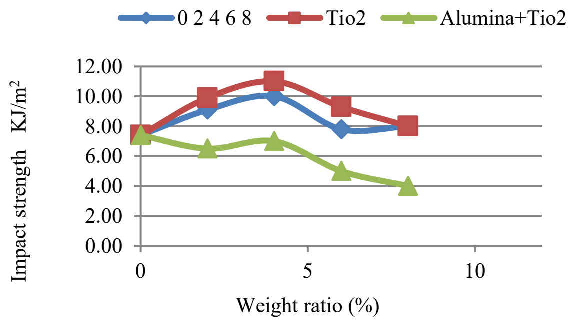


Figure 2. Impact strength of PMMA composite materials

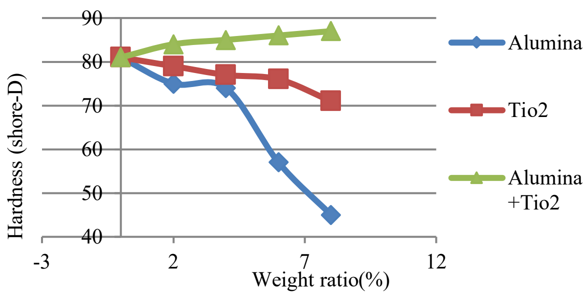


Figure 3. Hardness (Shore-D) of PMMA composite materials

Table 2. Statistical significance of mechanical properties of polymer reinforcement materials PMMA

Feature	Source of Variance	Sum of Squares SS	Mean Squares MS	$F=\frac{s_B^2}{s^2 w}$	F_{table}	α=0.05
T ensile	Between Group	SSB=7238.667	S^2_B= 2412.889	2.41	4.26	Not statistically significant
	Within Group	SSW =8996.167	S^2_w= 999.57			
Impact	Between Group	SSB=708.8	S^2_B= 236.26	2. 95	4.26	Not statistically significant
	Within Group	SSW ==719.95	S^2_w=79.99			
Hardness	Between Group	SSB=62226.17	S^2_B=20742	2.97	4.26	Not statistically significant
	Within Group	SSW=62890.67	S^2_w=6987			
Creep	Between Group	SSB=35.90	S^2_B= 12.17	14.2	4.26	Statistically significant
	Within Group	SSW=36.5	S^2_w= 0.86			
fatigue	Between Group	SSB=1.54×10^{12}	S^2_B=5.13×10^{11}	0.13	4.26	Not statistically significant
	Within Group	SSW==3.56×10^{13}	S^2_w=8.8×10^1			

5.2. Interpretation and Discussion of the Results

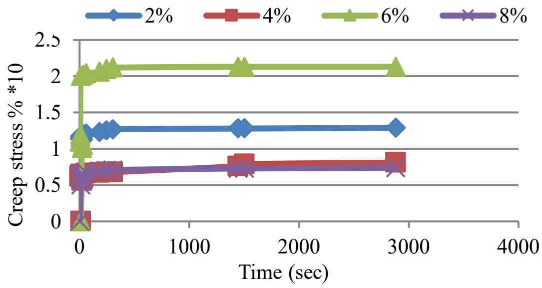
• First: the symmetry of the effect of adding reinforced nanomaterials (Al_2O_3 and TiO_2) and a mixture of (Al_2O_3+ TiO_2) of PMMA polymer in mechanical properties (tensile, impact, hardness, and fatigue) as a result of forming crystal structures with similar coordination and bonds as well as exposure to similar conditions of temperature, impact time and loads.

• Second: a statistically significant difference between the samples prepared with the creep characteristic. The reasons for this difference, in the researcher's belief, are due to the following:

1. The temperature of the laboratory may be a variable factor affecting the results of the examination, as the examination was conducted in the summer and the general weather was hot and the laboratory temperature was relatively high than moderate.

2. The exposure time is relatively long, which caused a slow and gradual deformation until the prepared samples crawl with the applied load force, which is less subject to a similar load on everyone, which made all samples unable to perform the job, and this was not achieved by special tests (tensile, impact, hardness and fatigue).

3. The deformation occurred suddenly as a result of the pressure exerted by the load at a specific time and for the same fractional weight (4%).



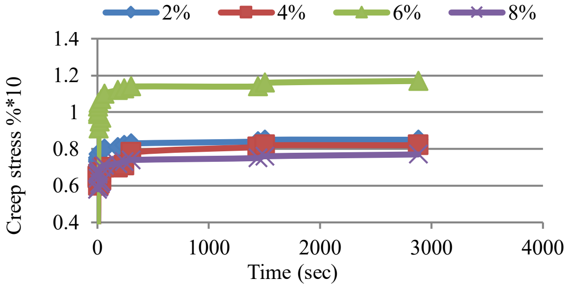
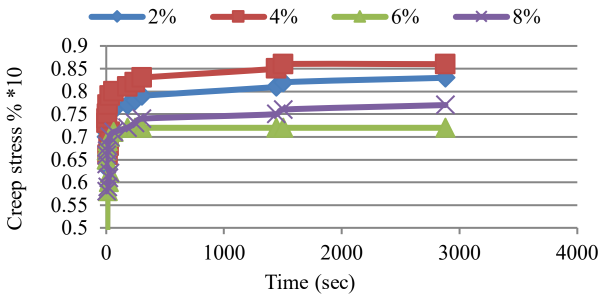


Figure 4. Creep curves for composite samples



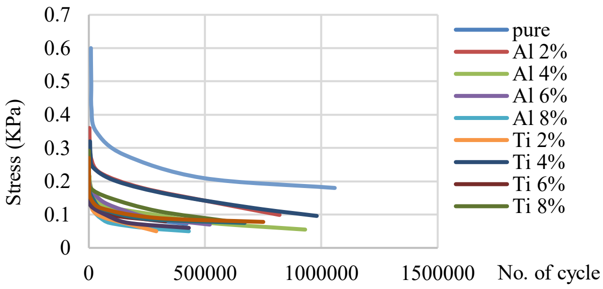


Figure 5. Fatigue curves for the composite samples

6. CONCLUSIONS

In light of the results of the study, we conclude the following:

1) The effect of adding reinforced nanomaterials (Al_2O_3 and TiO_2) and a mixture of (Al_2O_3 + TiO_2) to PMMA polymer is similar in improving mechanical properties tensile, impact, hardness, and fatigue, as it was found that the prepared samples had the same behavior in the mentioned properties.

2) The difference between the three prepared samples in their effect on creep characteristics, and the sample reinforced with a mixture of (Al_2O_3 + TiO_2) had better creep and fracture resistance.

3) The effect of adding reinforced nanomaterials (Al_2O_3 and TiO_2) and a mixture of (Al_2O_3 + TiO_2) to PMMA polymer is similar in improving mechanical properties tensile, impact, hardness and fatigue, as it was found that the prepared samples have the same behavior in the mentioned properties compared to each other.

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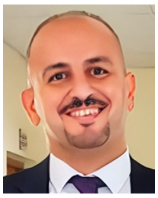

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Scientific Publications: 4 Papers, 1 Book, 2 Projects, 2 Theses