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Mechanical Characteristics of (TiO₂-ZnO)/PMMA Nanocomposites for Dentures

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ABSTRACT

Mechanical properties (impact strength, compressive strength and flexural strength) have been studied for (Polymethyl-methacrylate (PMMA)) polymer reinforced with nano (TiO_2+ZnO) (0 v%, 1 v%, 2 v%, 3 v%, 4 v% and 5 v%). Ultrasonic dispersion technique was used to prepare the nanocomposites samples, follow with cold casting technique using Teflon molds standard conditions. Charpy impact instrument was used to measure the impact strength, the compressive strength of each sample which was tested in newton units using digital compression tester (TinusOlsen: H50KT, England), while flexural strength can be found from mathematical formulas which depend on the impact strength, compressive strength and flexural strength. Results show that values increase in 1 v%, then decrease alternately by (2 v%, 3 v%, 4 v%, and 5 v%), while 0 v% is the less value of volume fraction of fillers. Young modules values increase alternately by the volume fraction of fillers until 2 v%, while if it is higher and lower than 2 v% it decreases the modulus of elasticity.

Keywords: Polymethyl-methacrylate (PMMA), Polymer nano-composites, Impact strength, Compressive strength, Flexural strength, Young modules, Dentures

INTRODUCTION

Many modern technologies require materials with unusual combinations of properties that cannot be met by conventional metal, ceramics, alloys, and polymeric materials [1]. The most extensively used material for the fabrication of dentures is PMMA acrylic resin, as it possesses a combination of favorable characteristics such as light weight, fabrication is inexpensive, lack of toxicity, stability in the oral environment and appropriate aesthetic and color matching ability [2]. However although it does not ideal in every aspect; and has several drawbacks that need to be addressed including low impact resistance, low thermal conductivity which compromises the patient assessment of taste and palatability [3]. Many attempts have been made to overcome these defects and improve the performance of PMMA denture matrix material either by modifying the structure of PMMA or by copolymerization with rubber or reinforcement by incorporation of different forms and types of fillers like metallic wire, fibers and the use of metallic oxides [4-7]. Great attention is directed towards the use of nano-sized fillers with the development of nanotechnology and nano-phased materials to reinforce the denture base resins thus producing a polymer nano-composite with improved physical and mechanical properties as compared to those filled with micro-scale particles [8]. Generally, the term composite is utilized to materials that are created mechanically by bonding two or more different materials together. The resulting materials have characteristics that are different from the characteristics of the component in isolation [9], the use of multiple nano-fillers rather than single additive develops a high performance composite which cannot be achieved by using single filler [10]. Polymer nano-composites have achieved great interest as high-performance structural materials this is due to their dimensional stability with high strength to stiffness ratio [11]. Liu, et al., found that TMSPM had remarkable effectiveness on the mechanical properties of the composites due to the improvement of interfacial adhesion between filler and matrix with better dispersion of the modificated particles in the matrix.

Related Works

Schwartzand Bahadure studies the incorporating of particles into polymers (PMMA) with improved hardness and storage modulus (stiffness), which in turn result in improved wear resistance [12]. Khalid, et al., found that using meth acrylic acid as a coupling agent in the synthesis of TiO₂-PMMA nano-composite; the resulted nano-composite exhibited improved elastic properties and has potential applications in dental composite and bone cement [13].

Sodagar, et al., found that on the addition of nano-sized fillers to reinforce the denture base resins thus producing a polymer nano-composite with improved mechanical and physical properties as compared to those filled with microscale particles. On the other hand, the use of certain nano-particles can add anti-microbial activity to dental materials [14]. Asar, et al., found that there was a significant increase in impact strength of heat polymerized acrylic resin after the addition of a mixture of 1% ZrO₂ and 1% TiO₂ surface modified micro particles.

In our research article, a surface hardness was positively affected by the addition of [Zn+TiO,] nano-particles for all the treatment percentage, surface roughness was positively affected by the addition of [Zn-TiO₂] nano-particles for all volume fraction, and the addition of [Zn+TiO₂] nano-particles to PMMA for teeth base resin positively affected the compressive strength of the material, improve the impact strength, transverse strength maximum Young's modulus of specimen's.

Theoretical Consideration

One of the most important properties of dentures materials is their ability to withstand the various mechanical forces applied on the material during its use such as a restoration, impression, model, tools, and investment material, polymers differing from one another by their chemical structure and properties are usually mixed together, either homogeneously or heterogeneously. The mechanical properties of the heterogeneous mixture are worse than those of individual polymers, whereas the mechanical properties of the homogeneous mixture are sensible. Using nanoparticles significantly improved the mechanical, properties as well as the surface smoothness of nano-composites [15,16]. Impact strength is the ability of the material to resist the break of sudden impact, which was calculated from equation 1.

$$IS = \frac{u}{A} (kJ/m^2)$$
(1)

where I.S=Impact strength

u=Energy of fracture in KJ

 $A=b \times d=cross-section area in m^2$

- b: The width of the specimen
- d: The thickness of the specimen

Compression strength: It is the force per unit area produced in the body in response to any applied force, which tends to compress (shorten) the material; it is accompanied by compressive strain. Compressive strength is, therefore, a useful property for the comparison of dentures resin composites. The compressive strength is the most important mechanical property of restorative materials. Factors affecting on compressive strength are filler (size, volume fraction, morphology, type; resin matrix chemistry or structure; polymerization reaction and particle/matrix interfacial adhesion) [17]. When a structure is subjected to compression note, the failure of the body may occur as a result of complex stress formations in the body. Compression strength is calculated from the slope of the stress against the deflection curve by the following formula:

$$E = \frac{F / A}{\Delta L / L}$$

E: Young's modules GPa

 $\frac{\Delta L}{L}$: Stress (Newton/cm²)

60

(2)

(4)

 $\underline{\Delta L}$: Strain (cm)

Flexural strength is defined as the maximum stress in the outermost reinforced, which is calculated by the following formula [18].

Transverse strength:

$$Kc = \frac{3PL}{2bd^2} \tag{3}$$

where b: The width of the specimen

P: The peak load

L: The span length

d: The thickness of the specimen

We can also calculate the flexural strength using Young modules and impact strength by the following formula:

$$Kc = \sqrt{IS.E}$$

where Kc: The flexural strength

IS: The impact strength

Hardness is the resistance of the material to deformation caused by abrasion or indentation forces for the surface. Hardness measurements have been successfully used as an indirect method of evaluating polymerization depth of resin-based. The Vickers hardness test technique consists of indenting the test material with a diamond indenter, within the sort of a pyramid with a square base associate degree at an angle of 136° between opposite faces subjected to a test force of between 1 gf and 100 kgf. The complete load is often applied for 10-15 seconds. The 2 diagonals of the indentation are left within the surface of the fabric when removal of the load was measured employing a microscope and their average calculated. The Vickers hardness is the quotient obtained by dividing the kgf load by the square millimeter space of indentation.

$$HV = 1.854 \frac{F}{d^2} \tag{5}$$

MATERIALS AND METHODS

Nano-composites are prepared by dispersing nano $[TiO_2 \text{ and } ZnO]$ kinetically by ultrasonication. To achieve a better state of dispersion first the nano-particles were treated with alcoholic medium (ethanol or acetone) for the de-agglomeration of the particle bundles. The treated particles are then added to the pure resin and solicited for 2 hours at room temperature as shown in Figure 1.



Figure 1 Show the equipment used for surface modification of nanoparticles A: prob sonication apparatus; B: magnatic stirrer

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Then the mixture was cured under vacuum at (363K) for 10 hours followed by hardener addition by using simultaneous magnetic stirring (100 rpm), for an hour to homogenization. The prepared samples were treated at (353K) for 6 hours in the oven to remove the moisture contents of the samples. The samples were placed between 2 metal plates under pressure to reduce porosity forming during hardening, before mechanical measurements, the surfaces of the specimens were mechanically polished to minimize the influence of surface flaws, mainly the porosity to organize the nano composite samples, molds were made of Teflon. The mold was made smudgy by wax before the mixture was poured into the mold during homogeneity. Impact strength test was conducted following the procedure recommended by the specification ASTM D790 with charpy impact testing device. The sample was supported horizontally at its both ends and was struck by a free swinging pendulum which was released from a fixed height in the middle. A pendulum of 2 joules testing capacity was used. The scale reading gives the impact energy absorbed to fracture the specimen in joules when it was stroked by a sudden blow. The charpy impact strength of unnotched specimen was calculated in KJ/m² by applying the relationship, Figures 2 and 3 show the impact strength equipment and specimens before and after testing.



Figure 2 Impact testing device (Charpy type)



Figure 3 Impact test samples: A: before testing B: After testing

Compressive strength was measured using a multi-purpose hydraulic piston to measure the maximum load on the test model universal tensile testing machine system having a capacity of 50 KN (Tinius Olsen, U.K., model HKT 50 KN). Compression strength test was carried out according to ASTM D 659-85 Digital device measuring maximum force. Testing was conducted for compressive strength of samples prepared from PMMA material before adding

reinforcement materials nano-particles of titanium dioxide and zinc oxide together, and other samples after the addition of nano-particles mentioned materials of different proportions of volume friction 0%, 1%, 2%, 3%, 4%, 5% (and get the curve) stress-strain and by knowing the slope of these curves. It obtained elastic modulus by equation 2, Figures 4 and 5 show the compressive strength tester and test samples A. before testing B. after testing strength compressive.



Figure 4 Compressive strength tester



Figure 5 Compressive strength samples, A. before testing B. after testing

Flexural strength test was carried out according to the specification ASTM- D790. All specimens were tested for flexural strength with a 3-point bending test. Each specimen beam was horizontally positioned on 2 fixed rollers (each 3.2 mm in diameter), the distance between the rollers was 50 mm, the loading head ended with a roller 3.2 mm in diameter, tests was performed for the specimens at room temperature using a universal testing machine (Instron® model 1122, Instron Corp, Canton, MASS) at a crosshead speed of 1 mm/min centrally loaded and deflected (midway between the supports), until fracture occurred data (stress) were recorded and the flexural strength was calculated using the equations 3 and 4 [19]. Finally, Figures 6 and 7 shows specimen was under load for the transverse strength test and, Vickers hardness test was performed by using a Vickers diamond indenter (digital micro hardness tester-HVS 1000), is a valid tool for evaluating the hardness of the polymers, with a load of 0.4403 N. The load was applied for time of 20 seconds.



Figure 6 Specimen was under load for the transverse strength test



Figure 7 Specimen groups A: before B: after testing with the 3-point bending test (flexural strength)

Three hardness readings were taken for each specimen at different locations of the test specimens. In order to eliminate possible segregation effect, after each indentation, the operator of the test machine read the lengths of the diagonals immediately. With a minimal (as short as 10 seconds) period of time elapsed between making and reading the indentations. It is assumed that, due to the short time elapsed between makings and reading the indentation, the visco elastic recovery of the diagonals after indentation is minimal. The operator measured the diagonals, and the equipment automatically converted these measurements to Vickers hardness numbers VHN (MPa) with a scale of 1 digit to the right of the decimal point in a number. Three indentations are made on each sample and the mean value is calculated by using equation 5. Figures 8 and 9 show Vickers hardness tester and hardness test samples.



Figure 8 Vickers hardness test



Figure 9 Hardness test samples

Table 1 showing tests type's schematic and dimensions, and standard methods of samples.

Table 1 Tests types' schematic and dimensions, and standard methods of samples

Test	Dimensions (mm)	Standards
Compressive test	D=20 mm diameter; Z=20 mm thickness	ASTM D-790
Impact, flexural test	X=100 mm length; Y=10 mm width; Z=5 mm thickness	ASTM D-790
Vickers hardness test	D=20 mm; Z=3 mm	ASTM E-92-82

RESULTS AND DISCUSSION

The resistance to impact is one of the determining properties of materials, mean values of the impact strength test results are presented in Table 2 and Figure 10. It clarifies the ability of all specimens to absorb a higher quantity of energy in comparing with specimen No.1 (pure PMMA) and show increase in the value of impact strength that is due to the addition of nano (TiO_2+ZnO)%, specimen No. 5 possess the highest impact strength which affects the stress distribution in the sample in comparison with specimens No. (1, 2, 3, 4, 6) this is due to the increase in cross-linking for material.

Fable 2 Impact strength, co	mpressive strength,	Young's modulus,	flexural strength	test results hardness
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Sample Code	No. of Sample	Impact Strength (KJ/m ²)	Compressive Strength (Mpa)	Young's modulus (GPa)	flexural strength (MPa)	Hardness (MPa)
Pure PMMA	1	7.347920	82.90	0.32100	51.900	9.3
1% TiO ₂ /ZnO	2	8.063789	128.20	0.44300	56.780	12.4
2% TiO ₂ /ZnO	3	5.319652	111.80	0.58000	54.123	14.6
3% TiO ₂ /ZnO	4	4.720000	96.67	0.26200	34.090	17.9
4% TiO ₂ /ZnO	5	9.284462	82.90	0.52089	71.535	22.5
5% TiO ₂ /ZnO	6	3.459310	85.70	0.41690	37.980	30.4



Figure 10 Impact strength of specimens with volume fraction (TiO₂+ZnO)%

The highest mean value of impact strength is 9.824462 KJ/m² related to the group of (4% TiO₂/ZnO) nano-powder, then the impact strength tends to decrease with the increase of (TiO₂+ZnO) %, the lowest mean value is 3.45931 KJ/ m². Impact strength is a measure of the energy absorbed by the material before fracture. There was a tendency for the impact strength to decrease when the (TiO,+ZnO)% content was increased. High aspect ratio fillers lead to large stresses within the polymer, near the filler edges [20]. Stress concentrations also occur at regions where nano additives have contact with another area of decreased interfacial bonding between the nano-additives and the matrix caused by clustered nano additives and void spaces act as stress concentration points in the polymer matrix [21]. The presence of such defects within the polymer matrix allows for a significant reduction in the total energy absorbed by the material due to an impact [22] Kamil, et al., stated that treatment of PMMA with loose silanated randomly oriented glass fibers 2 mm, 4 mm in length and in concentrations of 1% and 2% resulted in a significant increase in impact strength which may disagree with the results of our study [23]. Also Dogan, et al., findings disagree with the results of the current study; since a concrete increase in impact strength was obtained when different fibers (E-glass, nylon, rayon, and polyester) of different lengths were added in the concentration of (3, 4, 5)% to the poly (methyl methacrylate) resin [24,25]. Such differences between the above mentioned studies and the present study may be attributed to the difference in configuration of the functional fillers regarding dimensions, geometry, aspect ratio and orientation of the fillers within the thickness of the polymermatrix. Mean values, for compressive strength, are shown in Table 1 and Figure 11 which illustrates the compressive strength of different types by adding different volume fraction of nano (TiO₂+ZnO)% to PMMA (matrix).





It is clearly shown that adding nano (TiO₂+ZnO) material with different specimens prepared in this study percentage increased the compressive strength of the nano composite. The highest increase was obtained by adding 1% of (TiO₂+ZnO) nano powder to the PMMA (matrix). Table 2 shows the mean difference of compressive strength between the neat specimens with addition of (TiO₂+ZnO] nano powder in different volume fraction (0%, 1%, 2%, 3%, 4%, 5%). Specimen with 4% addition of (TiO₂+ZnO) showed a sharp decrease in compressive strength with an increase in nano powder, it's like a neat blend specimen which indicates to the variation of stress-strain for all the specimens, we found that specimen No. 2 had a better compressive strength with others specimens because the (TiO₂+ZnO) nano powder distribution was isotropic in 3-dimention with PMMA which givess it a good compression behavior. Other specimens did so in comparison with specimen No. 1 (standard) and nano composites have a higher compressive strength than that made of poly (methyl methacrylate) alone, this is due to the reasons mentioned above. Modulus of elasticity were also evaluated and the results are shown in Table 2, it can be seen that addition of 2% of nano (TiO₂+ZnO)% with concentration higher and lower than 2% decreased the modulus of elasticity of the material as shown in Figure 3. In this study, the modulus of elasticity was measured from tension (stress-strain) curve.

Elasticity was determined as shown in Figure 12, and Table 2 shows the variation of modulus elasticity (E) with the addition of bonding nano additives, specimen No. 3 possess the highest E-value in comparison with other specimens, it is clear that the bonding nano additives decreased the movement of polymeric chains and increased the density of

cross-linkage, resulting in a more rigid material and lower strain values and consequently to a higher E-values, but the other specimens show a lower bending modulus because they became more brittle [26].



Figure 12 Young's modulus and (TiO,+ZnO)%

As the content of nano $(TiO_2+ZnO)\%$ was increased, voids may be seen around the surfaces of the nano fillers which result from the polymerization shrinkage of the matrix; this issue is in agreement with Vallittu, et al., with voids between the matrix and the nano (TiO_2+ZnO) , the load-bearing capacity of the reinforced composite may decrease. Mean values, of the transverse strength test result, are present in Table 2. Figure 13 represents the mean values of the transverse strength results with the volume fraction of $(TiO_2+ZnO)\%$. The (4% $TiO_2+ZnO)$ acrylic samples demonstrated 38% increase in transverse strength with a mean value of 71.53 Mpa, while the (3% $TiO_2+ZnO)$ samples showed 34.208% decrease in transverse strength with a mean value 34.08.9 Mpa and because the modulus of elasticity of nano composite (sample No. 5) is high, most of the stresses are received by it without deformation [27].





Obviously, PMMA with nano additives (TiO₂+ZnO) possesses an elastic modulus greater than that of the PMMA resin; the incorporation of nano [TiO₂/ZnO] along with its geometry; noticeably, alters the elastic modulus of the (TiO,+ZnO) acrylic resin composite samples. The incorporation of nano (TiO,+ZnO) in concentrations of 4% allows greater stiffness, higher modulus (providing greater resistance to the applied load) and maintain a reasonable amount of elasticity and deflection (which is assumed to be lesser than that of the un reinforced samples) for the reinforced acrylic resin samples before fracture, the variance in samples with higher nano (TiO₂+ZnO) content was rather disturbing, as the nano (2% TiO₂+ZnO) concentration was increased to exceed 82.692% the modulus of elasticity (stiffness) of the composite samples was assumed to increase also; but, this increase is thought to be on the expense of the elasticity and deflection (capability to deflect within the elastic range) of the material, rendering the material stiffer, more brittle and prone to fracture, the nano (TiO_2 +ZnO) reinforcement used in the acrylic resin test specimens enhanced the transverse strength of the PMMA for both the (1%, 2%, and 4%), with the 4% nano (TiO₂+ZnO) content revealed the most favorable effect. However, when nano (TiO₂+ZnO) concentration of 4% was used; a clear, considerable transverse strength improvement of 38% was found in comparison with the strength of the pure samples. Contrarily, the increase in the concentration of nano (TiO2+ZnO) reinforcement (3%, and 5%) of the composite mass actually weakened the resin, and a decrease of 34.2% and 26.83% respectively. (TiO₂+ZnO) surfaces with the monomer may result in voids, the voids on the poorly impregnated nano (TiO,+ZnO) are oxygen reserves. As the content of nano additives was increased, voids may be seen around the surfaces of the nano additives, which result from the polymerization shrinkage of the matrix. With voids between the matrix and the nano additives, the load-bearing capacity of the reinforced composite may decrease. Also, the fact that the nano additives are free to orient themselves randomly within the thickness of the specimen means that they do not contribute significantly to improve the strength. However, further study needs to be conducted with a larger sample size indicated that the incorporation of small percentages of loose shortcuts (TiO₂+ZnO)% enhanced the fracture resistance of the PMMA tests specimens. The best (TiO₂+ZnO)% content was found to be between 1%, 2% and 4% of the total mass of the acrylic resin composite; the flexural strength increased by 9.5% on adding 1% best (TiO₂+ZnO) 4.48% on adding 2% and 36.08% on adding 4%. There appeared to be a specific limit of glass fiber concentration, above which, the level of enhancement was reduced. Substantially, high concentrations of fibers actually reversed the effect and weakened the resin significantly The results obtained by Stipho, et al., are valuable and are in agreement with the findings of this study; since there was a significant decrease in transverse strength for 5%, revealing that higher nano additives percentages acted on the contrary and lowered the fracture [28,29]. The results of Al Momen, et al., agree with this study since the addition of 5% randomly oriented loose nano additives resulted in a significant decrease in transverse strength reinforcement with randomly oriented effective if the stresses can be fillers in the loose form is only transferred from the polymer matrix (continuous phase) to the filler particles (dispersed phase) [25,30]. It has been shown by Vallittu, et al., that optimal adhesion between the polymer matrix and the fillers is essential for successful reinforcement (TiO₂+ZnO) used as reinforcement in the present study [31,32]. The higher content of nano (TiO2+ZnO) was used as reinforcement in the present study. The higher content of nano (TiO₂+ZnO) may tend to clump together when mixed with the monomer [33]; clumped nano (TiO₂+ZnO) masses may act as inclusion (foreign) bodies, each actually cause a micro fracture that breaks up the homogenous matrix of the PMMA instead of reinforcing it [34]. Void spaces within the clumped masses may act in a similar manner to break the PMMA matrix [29]. Poor impregnations (wetting) of the nano, the mean values of the test specimens are illustrated in Figure 5. The mean values of the surface hardness test are detailed in Table 1. The pure PMMA acrylic resin specimens (0% (TiO,+ZnO)) exhibited a mean surface hardness of (9.2); when all the mean values of the test samples were compared; there was an obvious trend of surface hardness increase along with the increase in the volume fraction of nano additives (TiO2+ZnO). Hardness may be defined as the resistance to permanent surface indentation or penetration, and it is indicative of the ease of finishing of a structure and its resistance to in-service scratching [35]. In an attempt to explain these results, one must imitate the micro-structure of the reinforced PMMA resin specimens, these specimens are planar structures (having thickness much lower than their other 2-dimensions i.e. length and width) and during the fabrication of these specimens using the compressionmolding technique a considerable amount of these nano particles might align parallel to the specimen's principal plane especially as the nano particles approaches the surface, and as these nano particles are high aspect ratio fillers, they offer greater opportunity of overlapped surfaces; and since they possess an elastic modulus greater than that of the denture base resin so they are stiffer and deform less than the acrylic matrix. In the result, much more resistance was provided against the penetrating indenter as more nano fillers were incorporated into the acrylic resin specimens. This finding agrees with that of who indicated a significant increase in surface hardness when 5% of nano (TiO₂+ZnO) was added to the PMMA matrix [36]. Therefore, the increase in hardness observed in the present study suggest that monomer to polymer was taking place as shown in Figure 14.



Influence of volume fraction Of PMMA/TiO2-ZnO on hardness

Figure 14 Surface hardness with volume fraction of (TiO₂+ZnO)%

Finaly the SEM photographs of selected combinations of filled and unfilled nano (TiO_2+ZnO) samples were subjected to a mechanical test that was studied for 0 v%, 1 v%, 2 v%, 3 v%, 4 v%, and 5 v% nano. A difference is observed because of variation in the amount of the additive and this is due to agglomeration winning in the polymer matrix as in the Figures 15-18.



Figure 15 SEM image of pure PMMA



Figure 16 SEM image 1% (TiO₂+ZnO)



Figure 17 SEM image 2% (TiO₂+ZnO)



Figure 18 SEM image 5% (TiO,+ZnO)

CONCLUSIONS

The following conclusions could be drawn:

- It has been found that the addition of nano additives in different volume fraction of (TiO₂+ZnO), 1%, 2%, 3%, 4%, 5%, to the poly (methyl methacrylate) which is used in a denture base produce improvement and reinforcement to the resin material of (PMMA) in the mechanical properties which were studied
- On the basis of the results, it significantly improves the impact strength at 1% and 4%, transverse strength at 1%, 2%, and 4%. Young's modulus of specimens at 2%, 4% and 1%, 5% respectively
- The addition of (TiO₂+ZnO) nano particles to PMMA denture base resin adversely affected the compressive strength of the material for (1%, 2%, 3%) concentrations. The focus was 1% of the highest value for the compressive strength
- Surface hardness was positively affected by the addition of (TiO₂ +ZnO) nano articles for all the treatment percentages, this indicates that the addition of (TiO₂+ZnO) does not have an effect on the color of the acrylic resin material which also falls within the desired range for the remaining additives
- The use of (TiO₂+ZnO) nano particles as a reinforcing filler in low concentrations proved to be a simple laboratory technique requiring no sophisticated equipment's or procedures. It is a convenient method to develop products with desirable properties. Also, this work is expected to introduce a new class of polymer nano composite that might find mechanical applications and dentures

DECLARATIONS

Conflict of Interest

The manuscript has no influence on the conflict of interest from all authors regarding the publication of the article.

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