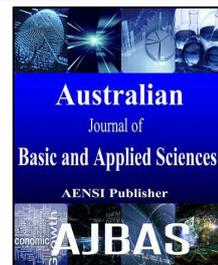




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Investigation on the Physical Properties of Denture Base Resin Filled with Nano-Barium Titanate

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ABSTRACT

Background: Poly(methyl methacrylate) (PMMA) is the material of choice for denture base construction because of its many good qualities. However, PMMA suffers from polymerization shrinkage and low strength, and its slow water absorption causes serious warpage and dimensional change in the material. Objective: This study aimed to evaluate the effect of nano-barium titanate (NBT) inclusion on the water absorption, polymerization shrinkage, and surface hardness of PMMA denture base. Results: The PMMA composites showed lower water absorption and polymerization shrinkage than pure PMMA. The density of the PMMA composites was increased by 5%, and a marked improvement in surface hardness was observed on the filled samples. Conclusion: NBT inclusion considerably reduced the water absorption and polymerization shrinkage, as well as improved the surface hardness and density, of PMMA. Such enhancements could promote the longevity of the composites.

INTRODUCTION

Dental polymer composites expansively utilized as dental fillings have gained a more significant attention than denture base composites (Elshereksi *et al.*, 2014a). Despite improvements in their qualities, restorative composite resins still suffer from mechanical strength deficiencies, lack of radiopacity, and polymerization shrinkage (PS). Thus, advanced research has focused on evaluating and improving composite resins to fabricate materials with a high strength and low PS. Previous studies have reported that a processing method may be employed to reduce distortion in denture base fabrication (Kobayashi *et al.*, 2004).

Dental composites undergo PS because of the conversion of intermolecular van-der-Waals forces to single covalent bonds during polymerization (Miao *et al.*, 2012). Shrinkage of 21% occurs when the monomer polymerizes. A mixture of a powdered polymer (pre-polymerized PMMA particles) and a monomer is used to minimize this shrinkage. The dissolution of the polymer in the monomer results in the formation of a plastic dough. The powder/liquid (P/L) ratio of 3:1 reduces the PS to 7% because a portion of the polymerization occurs when the denture reaches a solid state, which inhibits shrinkage (Kwon *et al.*, 2014). The development of volumetric contraction by resin composites is complicated by the effects of several factors, including polymerization degree, monomer functionality and size, and filler type, amount, and morphology (Boaro *et al.*, 2013). However, shrinkage leads to tensile forces within the material at the filler–matrix interface or at the interface of dental composites (Satterthwaite *et al.*, 2012). Even minor clefts increase water absorption into the

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interface of the two phases of composites and induce corrosion effects, which reduce the strength of the materials (Soanca *et al.*, 2011). The deleterious clinical effects of shrinkage–stress are due to the alteration of the final fit of the denture. Although the positive effect of filler impregnation on shrinkage reduction is well established (Razak and Harrison 1997, Sudheer and Manjunath 2011, Wang *et al.*, 2015), others reported that increasing the filler content is insufficient to reduce the overall shrinkage of composites (Boaro *et al.* 2013). Acrylic dentures in an aqueous oral environment absorb water, which lead to the breakdown of the polymer and the debonding of the filler from the matrix substance, ultimately reducing the longevity of composite restorations (Yoshida *et al.*, 2016).

Hardness is an important physical property of acrylic resins. This property enables these materials to be used for manufacturing denture bases that resist forces, such as those arising from occlusion and mechanical denture cleansing, thereby increasing the longevity of dental prostheses (Farina *et al.*, 2012). Hardness is the resistance of a material to plastic deformation typically measured under an indentation load. The indentation is imaged, and then the indentation area is measured and calculated to determine the hardness value. Hardness values have been used to predict the wear resistance of many restorative materials. In general, a material with a high surface hardness is wear resistant (Abed *et al.*, 2015). However, a correlation between surface hardness and wear resistance remains to be established (Suwannaroop *et al.*, 2011).

Nanomaterials with a small particle size and a high specific surface area show unique physical and mechanical properties (Kango *et al.*, 2013). Filler content not only directly determines the mechanical properties of composite resins but also allows for a reduction in monomer content. It improves the handling properties and influences the wear resistance, opalescence, radiopacity, intrinsic surface roughness, and polishability of resins (Ibarra *et al.*, 2015). Resins composed of this type of particle improve the surface finish and longevity of the material (Mousavinasab 2011). In this study, nano-barium titanate (NBT) is used as a dental filler because of its favorable mechanical properties and excellent biocompatibility (Ciofani *et al.*, 2013). To date, no study has been conducted on the dental applications of NBT. Therefore, this study aimed to evaluate the effect of NBT inclusion on the hardness, water absorption, and PS of PMMA denture base composites.

MATERIALS AND METHODS

Materials:

The solid components consisted of PMMA (molecular weight: 996,000 GPC) and 0.5% benzoyl peroxide (BPO). The liquid component comprised methyl methacrylate stabilized with 0.0025% hydroquinone and the cross linking agent 10% ethylene glycol dimethacrylate (EGDMA). NBT supplied in powder form constituted the filler. These materials were purchased from Sigma–Aldrich (USA). The titanate coupling agent KR 12 or isopropyl tri[di(octyl) phosphato] titanate (Figure 1) was supplied by Kenrich Petrochemicals (Bayonne, USA).

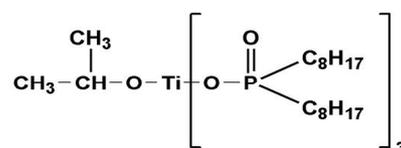


Fig. 1: Chemical formula of KR 12; or Isopropyl tri[di(octyl) phosphato] titanate.

Methods:

Surface modification of NBT:

5 g of NBT was added to 100 mL of acetone followed by KR 12 under stirring for 45 min. The mixture was maintained under sonication for 30 min at room temperature for deagglomeration. The slurry was stirred for 30 min and then the temperature of the mixture was increased up to 80 °C to remove the solvent. Finally, the modified NBT was dried for 24 h in a vacuum oven at 120 °C.

Sample preparation:

Various ratios (1, 3, 5, 7, and 9 wt.%) of the treated filler were used. Control samples comprised pure PMMA. Modified NBT was added to the liquid part (MMA monomer and 10% EGDMA) under continuous stirring to realize complete wetting of the filler surface and then sonicated for 20 min. The mixture was transferred to the stirrer before adding the solid components (PMMA and 0.5% BPO) and mixing by hand. The P/L solution in a 2.5:1 ratio was mixed until the dough stage was reached. The mixture was kneaded and packed into the prepared molds. Trial closure was performed at a low pressure, and excess materials were removed. The molds were clamped and pressed under 14 MPa at room temperature for 25 min to allow proper penetration of the monomer into the polymer, even flow of the material, and outward flow of the excess material. The molds were immersed in a water bath at room temperature in a thermostatically controlled water bath. The temperature

was slowly raised to 75 ± 1 °C for 90 min and then elevated to the boiling point for 30 min. The molds were allowed to bench cool before opening. The cured samples were carefully removed from the molds, the excess flush was eliminated, and the specimens were polished with a fine sand paper. The adopted procedures are consistent with the prescribed standard method for preparing a conventional denture base in the dental laboratory.

Field-emission scanning electron microscopy (FESEM):

The morphology of the NBT in powder form was studied using FESEM (Supra 55VP). Samples were prepared through dispersion, and one drop was pipetted onto a carbon support film on a 3 mm copper grid. The acceleration voltage used was 5 kV. The image files were processed on more than 500 particles.

Determination of Composites Density:

The density of the test specimens was determined in accordance with the ASTM D 792 water-displacement method (Method A). A densitometer (model MS204S/01) was used to measure the weight of the samples in air and in distilled water. The density of the specimens was calculated using the following equation:

$$\rho = W_1 / (W_1 - W_2) \quad (1)$$

where W_1 and W_2 are sample weights in air and water, respectively. The density of NBT as quoted by manufacturers is 6.08 g/cm^3 .

Water Absorption Test:

The water absorption test was conducted in accordance with ISO 1567-2000 specifications. The specimens were cut to 1–1.2 g using a band saw and then polished with sand paper grades p1200 and p2000 to improve their surface finish and remove cutting marks. Five specimens were prepared for each material. The specimens were dried in a vacuum oven at 37 ± 1 °C for 24 h and then stored in a desiccator containing silica gel for 24 h prior to immersion in distilled water. The specimens were weighed using an analytical balance with an error reading of 0.0001 g. Afterward, the samples were immersed in distilled water at 37 ± 1 °C and weighed after 1, 15, 30, 45, and 60 days. The change in weight percentages was calculated using the following formula:

$$\text{Change in weight (\%)} = \frac{W_1 - W_0}{W_0} \times 100 \quad (2)$$

where W_0 and W_1 being the weight of the samples before and after immersion respectively

Polymerization shrinkage (PS):

PS was calculated from the densities measured in accordance with Archimedes' principle. Uncured specimens were removed from the mold and weighed in air and in distilled water with an electronic balance accurate to ± 0.0001 g. Afterward, the same procedures were repeated with heat-cured specimens in a water bath, and the density of each specimen before and after curing was calculated using Eq. (1). Finally, the PS of each specimen was calculated using the following formula (Wang *et al.* 2015):

$$\text{PS (\%)} = \frac{\rho_{\text{cured}} - \rho_{\text{uncured}}}{\rho_{\text{cured}}} \times 100 \quad (3)$$

where ρ_{uncured} = the density of the uncured specimen (g/cm^3), ρ_{cured} = the density of the cured specimen (g/cm^3).

Surface Hardness Test (VHN):

The hardness test was applied in accordance with the ASTM E 384-89 standard. A calibrated Vickers hardness Tester (Shimadzu DUH-W201S) was used to force a diamond indenter of 0.3 Kgf for 15 s into the polished surface of the sample and to measure the diagonal length optically. The hardness test was performed on several samples for all formulations of composites, and the average of five readings at different points was obtained for each formulation of the composite samples. The Vickers hardness was computed using Eq. (4), and the prediction of hardness was theoretically calculated via the rule of mixture (ROM) in Eq. (5):

$$HV = 1.8544P/d^2 \quad (4)$$

where P = load, kgf, and d = mean two diagonals of indentation d_1 and d_2 , mm,.

$$H_c = H_m V_m + H_f V_f \quad (5)$$

where subscripts c, m, and f refer to the composite, matrix, and filler, respectively; H stands for hardness; and V denotes volume fraction.

RESULTS AND DISCUSSION

Dispersed phase characteristic:

With the development of polymeric dental composites, several works have focused on the optimization of filler types, compositions, and loading. This optimization has led to the enhancement of the physical–mechanical properties of fillers (Wang *et al.* 2015). Particle size is an important factor influencing the properties

of the resultant composite. Small filler sizes contribute toward good surface polishing and, consequently, high gloss. Size reduction allows for close particle packing, which facilitates the fabrication of resin composites with a high filler volume fraction and good aesthetic properties without compromising their mechanical and wear characteristics (Masouras *et al.*, 2008).

Nanocomposites reportedly possess improved mechanical, physical, and optical characteristics (Sideridou *et al.*, 2011). A decrease in filler particle size with a constant filler volume fraction enlarges the surface area of the dispersed phase and then increases the probable constraint upon the matrix phase. Thus, stress is inversely proportional to filler particle size for this factor. Filler particles are constrained by the matrix and undergo translation and rotational movement, thereby relaxing stress within the material (Satterthwaite *et al.* 2012). However, the same study presented a controversial finding, that is, composites with small filler particles exhibit a high shrinkage because of the effect of particle shape.

In the present study, NBT was used as a reinforcing filler to improve the properties of PMMA. The physical structure of the filler is presented in Figure 2. Microscopy is the only method in which the sizes of individual particles are directly observed and measured (Akbari *et al.*, 2011). The FESEM image of the filler in powder form was obtained at 80,000 \times magnification. The NBT filler is composed of a fine powder that contains spherical particles. These particles promote good mechanical adhesion between the filler's particles and the matrix. Satterthwaite *et al.* (2012) described that spherical filler particles display better property enhancement and lower shrinkage–stress values than irregular filler particles. Spherical particles also provide uniform distribution, which maintains dimensional stability. In addition, the capability of the dispersed phase to move within the matrix and relax stress increases as the sphericity of the dispersed phase increases. Another study reported that the presence of spherical filler particles affects the microfracture mechanisms of dental resin composites and increases the bending strength and fracture toughness at a high rate for elastic modulus (Mousavinasab 2011).

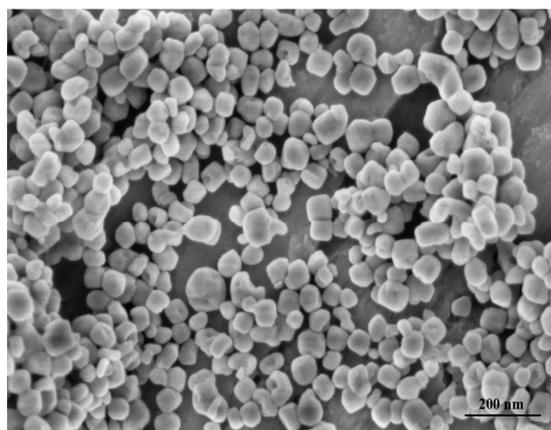


Fig. 2: FESEM image of the NBT nanoparticles.

Data on particle size were obtained after counting more than 500 particles distributed on approximately 18 FESEM images. Data on particle size and size distribution can be represented in either a tabular or graphical form. Particle size distribution was plotted on the basis of the particle size extracted from the FESEM images. A number–frequency histogram presents particle size and its distribution. A histogram is a bar graph that illustrates the frequency of occurrence versus the size range (Carpenter *et al.*, 1998). Figure 3 shows the number–frequency histograms of particle size data. Hundreds of particles could be measured to present statistically reliable mean size data. For instance, measuring 500–1000 grains has been proposed to obtain optimum sample size (Akbari *et al.* 2011).

Effect of Filler Content on Composite Density:

Density is an important physical property of dental composites. Table 1 presents the densities of pure PMMA and reinforced PMMA composites. The table shows that the density of the PMMA composites slightly increased after the filler was incorporated. The slight increase in density may be attributed to the small filler amount in the composites. Furthermore, an increase in density is expected because the density of NBT is higher than that of PMMA (i.e., 6.08 g/cm³). When they were blended, the density of the PMMA composites was between both components and increased with increasing NBT content. This result is consistent with the previous findings that the composition density of composites is linearly related to the increase in filler percentage (Rojek *et al.*, 2011, Elshereksi *et al.*, 2014b).

In general, the material used to produce a denture base should have a low specific gravity value because the denture base should be as light as possible. Therefore, an increased denture density is considered undesirable especially for the upper denture base because a heavy denture base would be wobbly in the patient's mouth and would drop down during use.

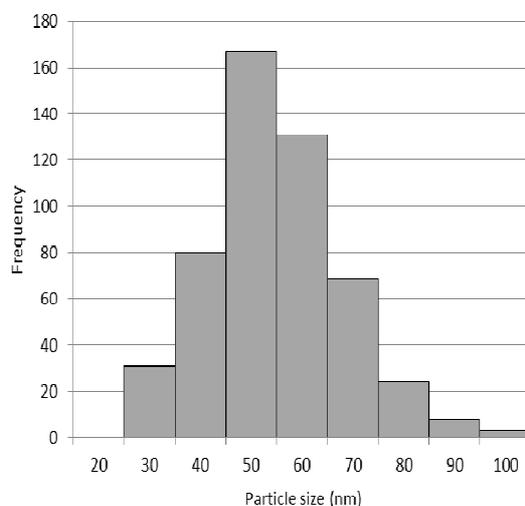


Fig. 3: Number-frequency histograms of NBT particle size distribution. The particle size data is based on the FESEM images analysis of more than 500 particles.

Table 1: Effect of NBT content on the density of the PMMA composites compared with the PMMA matrix.

| Formulations | Density (ρ_c) (g/cm ³) | Increase in density (%) |
|---------------|---|-------------------------|
| PMMA matrix | 1.189 | 0 |
| PMMA + NBT 1% | 1.193 | 0.3 |
| PMMA + NBT 3% | 1.209 | 1.7 |
| PMMA + NBT 5% | 1.225 | 2.9 |
| PMMA + NBT 7% | 1.238 | 4 |
| PMMA + NBT 9% | 1.256 | 5 |

Water absorption:

Dental composite resins for filling, acrylic denture base resins, and self-curing and heat-curing acrylic resins exhibit water sorption in the oral cavity. This phenomenon decreases the mechanical properties of the material. As a result, restoration durability is reduced, organic matrix properties are permanently altered, and material qualities are compromised. Therefore, water sorption should be suppressed (Soanca *et al.* 2011). The testing method and the maximum amount of water absorbed were standardized by International Standards as ISO 1567.

Figure 4 shows the water absorption of various denture base formulations immersed in water at 37 °C. The water uptake increases steadily with time until it reaches the saturation limit after 60 days of immersion. The PMMA matrix showed a higher water absorption capacity compared with the PMMA composites. This behavior can be attributed to the fact that the presence of polar and hydrophilic functional groups in acrylic resins may increase their polarity and facilitate the uptake of bound water to form primary and secondary hydration shells around the polar groups via hydrogen bonding. Addition of the treated NBT reduced water absorption, which restricted the neat polymer matrix with good adhesion. Therefore, the highest filler loading exhibited the lowest water absorption because of the great difference in the bulk materials used as compared with pure PMMA. Moreover, the samples notably showed significant water gain in the first 2 weeks and remained stable after the fourth week of immersion. Arora *et al.* (2011) analyzed PMMA-filled sapphire or silver and concluded that the water absorption of PMMA decreases with increasing filler powder. Factors that affect the amount of water absorption on the composite restoration materials include resin content, filler content, curing time, and coupling agent. In general, water absorption decreases with increasing filler content. In addition, proper bonding of the coupling agent corresponds to reduced water absorption (Biradar *et al.*, 2012). The presence of the coupling agent reduced the hydrophilicity of the ceramic filler and enhanced the adhesion between the filler and the PMMA matrix. The hydrophobic surface nature of the treated ceramic filler inhibited the capability of the composites to absorb water, thus corroborating the results of other studies (Pérez-Fonseca *et al.*, 2015, Yoshida *et al.* 2016).

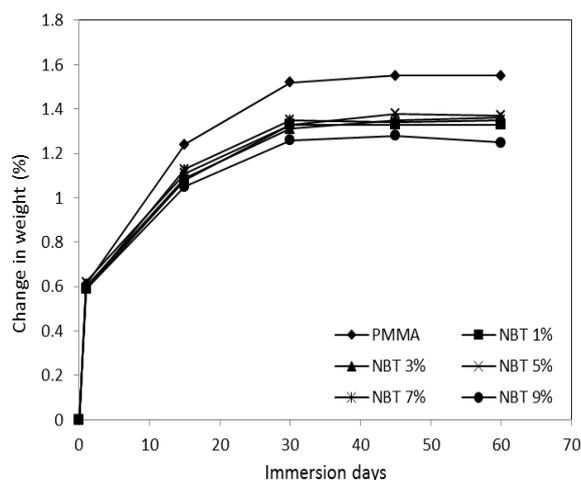


Fig. 4: Water absorption curves of NBT/PMMA compared with that of the PMMA matrix after 60 days of immersion at 37 °C.

Polymerization shrinkage (PS):

The PS of dental resins during polymerization is triggered by two phenomena. One is when the van der Waals distance between the monomer molecules is replaced by a covalent bond during polymerization. The other is when the intermolecular distance between polymer chains becomes smaller than that between monomers. Such shrinkage causes the buildup of a residual tensile stress around particles, which negatively affects the toughness of the material. Factors, such as monomer structure and content, as well as filler loading, type, and composition, influence shrinkage (Wang *et al.* 2015).

Polymerization in resin-based dental materials could be accompanied by a dimensional change that results in shrinkage. This resin shrinkage during curing affects density. Therefore, density variation was considered to indicate shrinkage. The curing shrinkage of the PMMA composites was evaluated by weighing each specimen in air and in water using an analytical scale balance. Figure 5 depicts the PS in the NBT/PMMA composites and pure PMMA. The shrinkage values of the PMMA composites were lower than those of the PMMA matrix. This result may be ascribed to the total organic matrix content of the composites. As mentioned above, the P/L ratio and presence of prepolymerized powder in PMMA resins may reduce shrinkage strain upon polymerization. However, the volumetric shrinkage strain is more dependent on the extent of polymerization or degree of conversion than on the size or amount of PMMA beads in the PMMA/MMA mixture (Kwon *et al.* 2014). Kobayashi *et al.* (2004) found that a reduction in the amount of strain before removing the samples from the mold is dependent on the cooling method and that gradual cooling reduces shrinkage strain with different cooling methods.

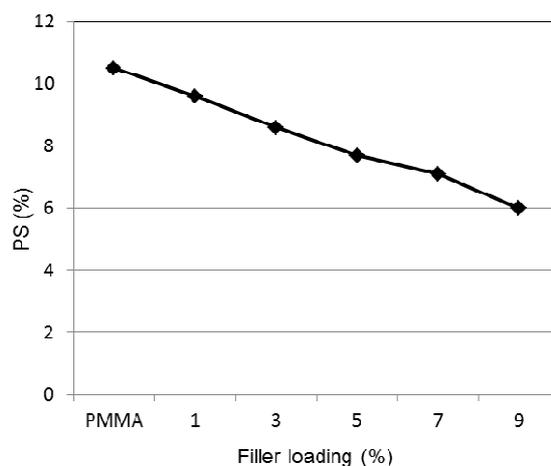


Fig. 5: Polymerization shrinkage of PMMA composites filled by different ratios of NBT.

The shrinkage in the NBT-reinforced composites decreased from 9.6% (NBT 1%) to 6% (NBT 9%). An inverse linear relationship was noted between NBT content and PS. PS considerably reduced with increasing

NBT content in the PMMA matrix because of the decrease in the number of reactive methacrylate groups in the PMMA composites with high filler levels (Razak and Harrison 1997). As the amount of NBT was increased, the interstices existing in PMMA could be assumed to be gradually embedded with these small fillers. Hence, the movement of the formed polymer chains might be further hindered during polymerization, and the shrinkage may decrease to the minimum under the highest filler loading. These outcomes agreed with the generally accepted conclusion that high inorganic contents are associated with low polymerization stress values, which are directly related to shrinkage reduction (Sudheer and Manjunath 2011, Wang *et al.* 2015). Others reported that epoxy resin-based nanocomposites demonstrate low shrinkage and high strength because of the strong interfacial interactions between resin and nanoparticles (Chen *et al.*, 2006).

During denture fabrication, the shrinkage that occurs when a liquid monomer is converted to a solid polymer can be largely compensated by keeping the curing dough under compression. However, hybrid systems deliberately start the polymerization of the dough on one side of the mold while forcing the uncured dough into the mold on the opposite side. As curing progresses across the mold, the mold space is continuously filled, consequently improving dimensional accuracy. The advantage of this system is the decreased tendency to raise the bite by overpacking the mold with dough, which can occur with conventional flasks. PS produces internal stress, which consequently creates microvoids or microcracks that impair the mechanical properties of composites. In composites used for removable prosthodontics, PS impairs the adaptation to the patient's mouth, which leads to the formation of rocking. The shrinkage of current filling composites ranges from 1.5% to 5% after curing (Pires-de-souza *et al.*, 2009). Thus, 6% of the denture base composites should be acceptable because of the great difference in the bulk materials used.

Effect of filler on surface hardness (VHN):

Hardness is an important measure of a restorative material's resistance to undergo permanent surface indentation or penetration. Indentation is the pressing of a hard round object or point with force against a material sample, which produces a depression. Depression or indentation is caused by plastic deformation beneath an indenter. Specific characteristics of indentation, such as shape or depth, are used as a measure of hardness (Abed *et al.* 2015). VHN is the number obtained by using the aforementioned formulae, Eq. (4). The hardness of dental composites is a significant issue to address because its resistance to abrasion is directly related to their longevity. Thus, the substantial surface hardness of restoration materials is a principal requirement in posterior stress-bearing areas.

Figure 6 shows the VHN values of the filled samples compared with those of the PMMA matrix. The filled samples exhibited a higher surface hardness than the PMMA matrix. Nevertheless, the PMMA composites exhibited increased hardness with increased filler content. This result indicates that a strong correlation exists between the VHN and the filler volume fraction in the PMMA composites. In other words, the amount of filler particles can influence and improve this property. This finding may be attributed to the high hardness value of the dispersed phase and the imposed restriction on matrix deformation caused by the uniform distribution of the dispersed phase. Similarly, Mathew *et al.* (2014) reported that increasing the filler content in a polymer matrix improves composite hardness. The results of the present study agreed with those of previous ones showing that dental composites have high hardness values because of their high filler contents (Vojdani *et al.*, 2012, Elshereksi *et al.*, 2016). Conversely, combining the lowest filler content with the smallest filler size resulted in the lowest hardness value. Furthermore, the white color of ceramic fillers is not expected to affect adversely the aesthetic appearance of denture base resin (Vojdani *et al.* 2012).

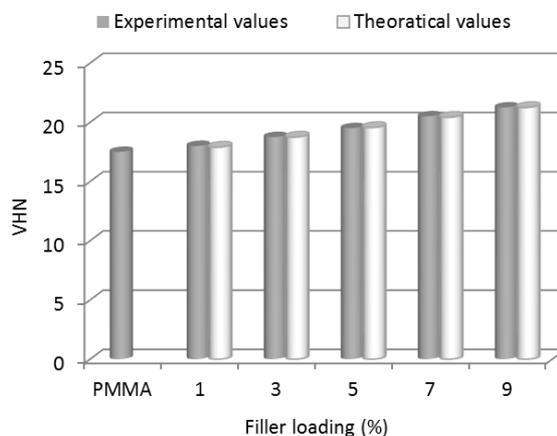


Fig. 6: Experimental and theoretical VHN values of NBT/PMMA composites.

Predicting the overall properties of composites is important for material design and applications. The ROM is the simplest and intuitive method to estimate the effective mechanical properties of materials in terms of their composition. The theoretical hardness values of the PMMA composites were calculated and plotted using ROM (Figure 6). The theoretical and experimental VHN values showed no clear differences. This consistency supported the homogeneous dispersion of the NBT, which improved the VHN by 21.5% at the highest filler content. The hardness of the composites approached that of the soft matrix especially at a low volume fraction of the hard particle.

Conclusion:

NBT inclusion remarkably lowered the water absorption and PS of the composites but evidently improved their surface hardness. Such enhancements could increase the longevity of the composites. This work provides a good foundation for future studies that aim to evaluate the mechanical and environmental properties of composites.

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