Water Sorption and Solubility of Light-Cured Dental Composites Prepared from Two Different Types of Matrix Monomers

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Abstract. Two groups of resins reinforced by different ratios of nano-hydroxyapatite were used to produce the novel dental composite, with the main purpose of measuring the water sorption (WS) and solubility (SO) of the prepared nanocomposites. Ten experimental specimens were prepared in disk-shaped with and without filler materials, in two groups, each group was classified according to filler contents (0, 1, 2, 3, and 4) wt. % of nano-hydroxyapatite. Each specimen was stored for 7 days in water, proceeding to measure the mass of each specimen. After drying process, the specimen masses were determined. Then WS and SO were calculated from these determinations. The results showed that values of water sorption after 1 week for group A composite and group B composite ranged (9.66 to 13.07 mg/mm³) and (5.11 to 8.52 mg/mm³), respectively, while solubility was (2.97 to 3.25 mg/mm³) for the composites specimens of group A and (1.83 to 2.26 mg/mm³) for the composites specimens of group B, respectively. For composite with different filler contents the results showed a high filler contents of 4% for each group with high value of water sorption (WS) and solubility (SO). It could be concluded that the percentage of sorption and solubility of composite based on acrylic and amide were significantly lower than that based on acrylate.

Keywords. Water sorption, Solubility, Dental composite, Filler content.

1. Introduction

Human teeth can be completely or partially damaged which negatively affects a person's health and appearance; therefore, they need restoration biomaterial continuously. Composite materials are the most common materials used not only because of their facility to adhere to tooth substance but also for their aesthetic and performance characteristics. Dental composite materials consist mainly of filler particles and a polymer matrix based on different monomers, dimmers and/or oligomers of methacrylate and/or acrylates, together with additives [1]. These materials stay for long time in the oral environment and will undergo an interaction with the oral fluids. Hence, they absorb water and release unreacted components.

According previous studies, the absorbed water weakens the mechanical properties such as fatigue limit, transverse strength, and hardness, because of the effect of water plasticizing [2,3]. In addition, water sorption has an effect on the dimensional stability through a three-dimensional volumetric expansion and causes occlusal changes [4]. The dimensional change of dental composite produces...
internal stress and harmful consequences such as crack development and denture fracture [5, 6]. Polymer is exposed to water sorption because of the polarity of molecular present in the polymer, unsaturated bonds of the molecules, or unbalanced intermolecular forces in the polymers [7]. It causes reversible rupture of ill-qualified inter-chain bonds as a result of absorbed water, as well as irreversible flaws in the polymer matrix [8]. The result of this effect is a degradation of the filler/matrix interface and deteriorating of the polymer network [9, 10]. The extent of generated degradation depends on the chemical composition of the monomers, dimers and oligomers, the cross-linking grade in the polymerized matrix, the conversion rate and influence of the environment [11]. The factors of time immersion and filler content of the composite material have influence on the activity of water sorption [12]. Degradation processes will also cause erosion of filler particles. These erosion processes will lead to the loss of mass of the dental composite material [13].

Hence, the networks of polymer should be insoluble materials especially those with high thermal and chemical stability. Despite the fact that most of the monomers of polymer used in dental composite can take up water and chemicals from the environment, they cause the releasing of components into the surrounding environment [14, 15]. There, as indicated by various researchers, the polymer composite materials can be used in different biomechanical application, such as prosthetic application, [16-28], bone application, [29-31], removable partial denture, [32-36], hip joint application, [37-40], and other applications [41-53]. Then, the polymer composite can be applied to manufacturing dental composite materials.

Another significant factor in dental composite that must be considered in their water sorption (WS) and solubility (SO), rate of wear, surface roughness, and esthetic results, is the filler type and characteristics [54 -56]. The main aims in this study is preparing a new nanocomposite material used for dental applications having desirable characteristics by using new types in the resin matrix materials. Also, the study aims to examine the effect of nano-filler content on some properties such as water sorption and solubility, and comparing the result properties of all the prepared composites to attain the best materials.

2. Materials and methods

In this work two groups of polymeric composite materials have been prepared, the most important monomers used in preparing the samples are:

- The 1,6 hexanediol methacrylate, bisphenol dimethacrylate (Bis_DMA) bisphenol-A-glycidyl dimethacrylate (BIS-GMA), dimethyl aminoethyl methacrylate (DMAEMA) and camphorquinone (CQ) which were supplied by the Sigma Aldrich (USA) company.
- Methacrylamide (MM), methacrylic acid (MA), 2-E thylhexylmethacrylate, polyethylglycol (PEG) provided from Merck (Germany).
- Nano Hydroxyapatite (Hualancheh.Co.China) and Zinc Oxide (ZnO) (GCC) are the fillers used to reinforced these monomers with an average particle size of 50 nm. Each group is made with five different fillers (nano-hydroxyapatite) at (0, 1, 2, 3, and 4) %. The chemical compositions of the two groups of composite resins prepared are listed in Table (1).

| Group | Resin Monomer | Filler Type | Filler wt% |
|-------|---------------|-------------|------------|
| A     | 1. BIS-GMA (40%) | Hydroxyapatite | 0,1,2,3,4 |
|       | 2. methacrylamide (20%) | | |
|       | 3. methacrylic acid (20%) | | |
|       | 4.1-hexanediolmethacrylate (20%) | | |
| B     | 1. BIS-GMA (40%) | Hydroxyapatite | 0,1,2,3,4 |
|       | 2.2-Ethylhexylmethacrylate(20%) | | |
|       | 3. polyethylglycol (10%) | | |
|       | 4. bisphenol adimethacrylate (10%) | | |
|       | 5.1-hexanediolmethacrylate (20%) | | |
2.1. Preparation of composite
Matrix monomers were selected as (Bis-GMA, MM, MA, and 1-6HNM) for group A and (Bis-GMA,2-EMA, Bis-AMA and1-6HNM) for group B. These samples components were weighed by a digital sensitive balance (Sarorius, BL210s, Germany) and then subjected to mixing process for approximately 90 minutes until the homogenous mixture are obtained. The process of adding zinc oxide (0.4gm) as antimicrobial agent, and Nanofillers (1,2,3,4) % as colorant to improve the mechanical characteristics have been implemented. Adding DMAEMA (0.5wt %) as accelerator and camphor quinine (0.5wt %) as initiator also have implemented continuously for twenty minutes as the last stage of mixing. Then, the paste has been inserted to the test moulds, and loosely hand pressed between two slides of glass to eliminate any excess material. The monomers molecules structure of matrix group (A) is shown in Figure 1 (a), which contains four different types of monomers materials as stated in Table (1). The new repeating unit for matrix group (A) is shown in Figure 1(b).

![Figure 1a. Monomers molecules structure of matrix group (A).](image1a)

![Figure 1b. Repeating unit for matrix group (A).](image1b)

On the other hand, the monomers molecules structure of matrix group (B) is shown in Figure 2 (a), which contains five different types of monomers materials as stated in Table (1). The new repeating unit for matrix group (B) is shown in Figure 2 (b).
Figure 2a. Monomers molecules structure of matrix group (B).

Figure 2b. Repeating unit for matrix group (B).

Finally, the composite resin was photo polymerized with the use of a light curing unit (EliparFreelight2LED, 3 M ESPE) at 1500 mW/cm² intensity. Light was illuminated on the two surfaces, bottom and top, via clear matrix strips for sixty seconds. The samples were exposed to irradiation in different positions for 60 s until the entire area was exposed. The distance between the specimen tip and the surface of light curing unit has been kept at 1–2 mm. After that, the specimens were removed from the mold, both sides were smoothed to a thickness of 0.85 ± 0.1 mm.

2.2. Test of specimens
Water sorption and solubility tests were determined according to the specification standard for composite (ISO 4049:2000) [57]. Fifty discs of specimens of about 15±0.2 mm in diameter and 1±0.1 mm in thickness were made as shown in figure 3. Five discs of specimens were prepared for each composite material, and the control sample (free from filler), were weighed by an analytical scale accurate up to 0.0001mg (Sarorius, BI210s, Germany). All specimens were kept on a vacuum desiccator at 37°C to obtain a constant weight (m₁). Specimens were next stored in 10 mL in distilled water for seven days. After this storage period, the samples were removed, and the excess water was removed with absorbent paper until water was no longer visualized. The weights were recorded again (M₂) after they were obtained of constant mass. Specimens were subsequently reinserted in the desiccator at 37°C and weighed daily until a constant mass of water release (M₃) was obtained.
Equations 1, 2 were used to calculate the values of water sorption (WS) and solubility (SL) respectively. [58]

\[
WS = \frac{M_2 - M_3}{V} \quad (1)
\]

\[
SL = \frac{M_1 - M_3}{V} \quad (2)
\]

Where; \(M_1\) is the initial dry constant mass (mg) prior to immersion in water, \(M_2\) is the mass of the specimen (mg) after immersion in water for seven days; \(M_3\) is the mass of the reconditioned specimen (mg), and \(V\) is the volume of specimen in mm\(^3\).

![Figure 3. The Prepared Specimens for Testing Water Sorption and Solubility.](image)

### 3. Results

Based on the ISO 4049 standard test, it was found that the maximum WS value dental composite was 40 mg/mm\(^3\)[57]. In this work the maximum SO value reached 13.07 mg/mm\(^3\). So, none of the prepared composite specimens exceeded the maximum WS value. The SO values for the composite resins tested in this study ranged from 5.11 to 13.07 mg/mm\(^3\). This result is related to the difference in the chemical composition of matrix material and the ratio of nano-hydroxyapatite content in composites materials. The mean WS and SO values for each specimen were listed in Table 2.

| Specimens | Water Sorption, mg/mm\(^3\) | Solubility, mg/mm\(^3\) |
|-----------|-----------------------------|-------------------------|
| A1        | 9.66733                     | 2.970927                |
| A2        | 10.80466                    | 2.970927                |
| A3        | 11.942                      | 3.1124                  |
| A4        | 12.51066                    | 3.253873                |
| A5        | 13.07933                    | 3.253873                |
| B1        | 5.117998                    | 1.839146                |
| B2        | 6.255331                    | 1.980618                |
| B3        | 6.823998                    | 2.122091                |
| B4        | 7.392664                    | 2.122091                |
| B5        | 8.529997                    | 2.263564                |

Through this test, it was found that all of the studied composite resins during immersion in water increased in their weight, as well as in their mean values of water sorption. Specimens of the second group (B) showed significantly lower values in these properties, while specimens of the first group showed the highest values in water sorption, which is lower than those required by ISO 4049 standard; 40 mg/mm\(^3\) [57].

Water sorption also showed difference within each studied groups. When adding nano-filler (Nano Hydroxyapatite) to matrix resin, they showed varying values of water sorption of composite samples of more than the reference specimen (free from filler). Specimen A5 within group A represents the
highest filler content with the higher value of water sorption, then decreasing is followed by A4, A3 and A2, respectively, with a decrease in the ratio of nano-hydroxyapatite in composite. For group B also, the data showed the lowest and highest mean solubility values for each specimen within this group. Increase was found in solubility values in B2, B3, B4 and B5, respectively, with increase in the ratio of nano-hydroxyapatite in composite. On the other hand, the mean values of solubility showed by the tested composite specimens varied from 1.8 to 2.263564 mg/mm\(^3\). Specimens of group A exhibited higher mean solubility than group B, varying from 2.97 to 3.83 mg/mm\(^3\). All these values were less than the maximum value mentioned by the ISO 4049 standard (<7.5 mg/mm\(^3\)) [59].

Figures (4) and (5) elucidate the effect of adding hydroxyapatite nanoparticle (for two groups different in the matrix monomers) on water sorption (WS) and solubility (SO) for nanocomposites, respectively. From these Figures, it was noticed that the addition of nanoparticles increased the values of WS and SO with addition of nano-hydroxyapatite to matrix material and these arrived to the highest values at 4\% wt. where the maximum values of WS and SO for first group (A) reached to 13.07933 mg/mm\(^3\) and 3.253873 mg/mm\(^3\), respectively, and the maximum values of WS and SO for second group (B) reached 8.529997 mg/mm\(^3\) and 2.263564 mg/mm\(^3\), respectively. On the other hand, it can be observed from these Figures, that the nanocomposite samples of first group (A) matrix material (see Table 1) have the highest values of water sorption and solubility as compared with their counterparts of the second group (B) of matrix material (see Table 2). This is related to the nature of the components of matrix material and molecules structure of repeating units for matrices A and B as shown in Figures 1b and 2b, respectively. Also, it depends on the compatibility range between hydroxyapatite nanoparticles and the components of matrix material.

![Water Sorption](image1.png)  ![Water Solubility](image2.png)

**Figure 4.** Water sorption as a function of nano-hydroxyapatite content in nanocomposites.  **Figure 5.** Water solubility as a function of nano-hydroxyapatite content in nanocomposites.

### 4. Discussion

The chemical composition of the prepared composites materials (i.e. hydrophobic and hydrophilic monomers, solvent material, and filler particles) of the restorative material have a direct effect on its degradation. Apparently, the extent and rate of water absorption is dependent on the nature of the polymer network and the potential binding of hydrogen and polar interactions, as proven by research [59]. The amount of water that composite resins can absorb depends on the chemical composition of hydrophilicity polymeric matrices materials and on the nature of the nanofiller materials in composite [56, 60]. So, the results obtained from the two experimental composite groups tested in this study i that differences in the indicate that polarity of each monomer has a significant role in determining the ability of absorbing water in polymers. Thus, all composites samples of group A showed high water sorption mean values after storage in water for one week, which is due to the chemical structure of its polymer repeat units being more polar because they contain carbonyl, amide, benzene and hydroxyl groups as shown previously in Figure (1b). Therefore, they are hydrophilic polymers. Hence, Figure
(4) shows the highest WS value for the specimens of group A, which has more amide groups (NH2) monomers per length of chain, which will increase the polar nature of the polymer group (A) specimens, because this functional group has more types of monomer. This leads to more hydrogen bonding between water, hence the polymer will be more hydrophilic and water soluble. On the other hand, the samples of group B contain methacrylate monomers as shown previously in Figure (2b). Therefore, they are hydrophobic polymers, which exhibited a lower value for water sorption than samples of group. A. Within the same resin group, the value of water sorption may also vary with the filler content (hydroxyapatite nanoparticle). It is indicated that there is extra weight gained by absorption and diffusion with increasing weight fraction ratio of hydroxyapatite nanoparticle, which may be due to hydroxyapatite nanoparticle which have a higher water penetrates, as well as, a higher water absorption percentage than the matrix material [61]. Additionally, the porous structure of nanocomposites materials increases with increasing the percentage of the hydroxyapatite nanoparticle content in the composite. This will increase the free volume within the polymeric matrix and create a tortuous path for the permeating molecules, and this increase is in water absorption [62].

In regard to solubility (SO) behavior in Figure 5, a number of factors affect the solubility values of composites, for example the monomers type, immersion time, number and the size of leachable species, the interface bond of resin-filler quality, the solvents, and temperature [63,64]. Another important factor is the residual monomers that do not react and are consequently lost. It is highly dependent on the degree of conversion, that is, the higher the value of the conversion degree, the lower the amount of non-reactive monomers, resulting in a lower solubility [60, 65].

The diffusion of a lot of water through the polymeric materials is undesirable because of its tendency to reduce the frictional forces between the polymeric chains, due to the problem of polymer swelling. At higher absorbed water intensity, polymeric chains may suffer a relaxation process, thus the elution of unreacted monomers and/or solvents that trapped in the polymer network become more facilitating [66]. These factors are likely responsible for the known degradation of the resin-dentin bonds over time observed by several research centers [60, 67]. Specimens noticeably materials listed in group B, are lower solubility value than the other tested specimens due to their hydrophobicity, where the highest value of water sorption is the highest solubility value, vice versa.

5. Conclusions

The following conclusions can be drawn:

1. All polymers absorb moisture in humid atmospheres and when immersed in water. The amount of water sorption and solubility of composite resins varies according to the hydrophilicity nature of the monomers and filler content.
2. The composite that contains the polymeric materials with more hydrophilic functional group have higher water sorption and solubility. Therefore, composite based on acrylic and amide had W.S and SOL% significantly higher than that based on acrylate.
3. Weight gain percentage of prepared composites increase with increase in weight percentage of fillers, where the percentage 4%wt represent the highest of water sorption and solubility values.
4. Water has a very important role in the interaction strength of filler and matrix; it merely accelerates polymer- filler failure which fails the restoration material.
5. All water sorption and solubility values were less than the maximum values mentioned by the ISO 4049 standard; therefore, the prepared composites are considered promising materials in dental applications.

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