

# Water Sorption, Solubility and Surface Properties of CAD-CAM Materials

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## Research Article

### Abstract

The purpose of this research was to examine water sorption, solubility, and surface properties of three widely used CAD-CAM materials: resin composite (Cerasmart; CS), polymer-infiltrated ceramic network (Vita Enamic; VE) and lithium disilicate glass ceramic (IPS e.max CAD; LS). Therefore, 45 rectangular-shaped specimens were prepared and immersed in deionized water for varying durations at 7 days, 1 month and 6 months. These specimens were then subjected to a range of tests, including water sorption, solubility, hardness and roughness. The result showed that immersion time significantly impacted water sorption among tested materials ( $p<0.001$ ). However, there were no significant differences in solubility between materials at each time point of the water immersion process ( $p>0.05$ ). The 2-way ANCOVA revealed that water immersions had significant effects on hardness among tested CAD-CAM blocks ( $p<0.001$ ), while there were no significant differences on roughness ( $p>0.05$ ). Moreover, the study found that there were no significant differences on roughness within IPS e.max CAD group at every time point ( $p>0.05$ ), while Cerasmart exhibited a similar trend compared to Vita Enamic, where surface properties showed significant differences among 7 days and 6 months of water immersions ( $p<0.05$ ). The correlation analysis showed statistically significant differences between water sorption and hardness, water sorption and roughness ( $p<0.001$ ). Overall, assessment of IPS e.max CAD, Vita Enamic, and Cerasmart, exhibited different levels of water sorption when subjected to prolonged water immersion for up to 6 months. Both aged and unaged specimens of these materials showed similar sorption and solubility during immersion process. However, water sorption influenced their hardness and roughness after immersion. Although these materials displayed varying degrees of water sorption, hardness, and roughness, they did not exhibit significant differences in solubility after 6 months of water immersion.

**Keywords:** CAD-CAM block/ Hardness/ Roughness/ Solubility/ Water sorption

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### Introduction

Computer-aided design-computer-aided manufacturing (CAD-CAM) technology is becoming a promising means of choice for indirect restoration due to its benefits, including reduced chair time, better infection control and patient's preference over intraoral scanning rather than taking impressions<sup>1</sup>. Software and milling devices also have greatly improved, especially with recent introduction to a new range of digitalization tools and scanners<sup>2</sup>. Additionally, CAD-CAM technology has enabled the use of polycrystalline ceramics and the development of new materials with a polymeric matrix<sup>3-5</sup>. There are a variety of commercially available CAD-CAM blocks in dental practice, with differences in composition. A modern kind of resin

composite materials for indirect restorations has been developed especially for CAD-CAM technology. This advanced technology produces highly polymerized resin composite blocks under high temperature and high pressure in order to increase monomer conversion, mechanical properties, and polymerization shrinkage stress<sup>6</sup>. Lithium disilicate (LS<sub>2</sub>), with long-term clinical research<sup>7</sup>, has been proven to be the most widely used material for ceramic restorations due to its pleasing appearance, excellent mechanical properties, high hardness, high wear resistance<sup>3</sup>, and user-friendly workability<sup>8</sup>. A polymer-infiltrated ceramic network or PICN has been introduced as an alternative to ceramics<sup>3</sup>. It is created by infusing a pre-sintered glass-

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ceramic scaffold with a monomer, which is subsequently polymerized. Unlike resin composite materials with dispersed fillers<sup>9</sup>, PICNs have a unique 3-dimensional scaffold of interconnected particles that forms a sturdy skeleton<sup>10</sup>. This structure distributes stresses effectively in all directions, making it highly resistant to breakdown phenomena<sup>10</sup>. High temperature increases chain mobility and polymerization, whereas high pressure compensates for shrinkage and reduces number and size of defects<sup>6</sup>.

Previous studies have demonstrated that water sorption and solubility were the origins of various changes in physical and mechanical properties of resin composites<sup>11, 12</sup>, which resulted in reduction of hardness and wear resistance due to material degradation<sup>13</sup>. Despite its excellent mechanical properties, CAD-CAM lithium disilicate glass ceramic degraded after exposure to various beverages, leading to decreased hardness, color changes, and increased roughness<sup>14</sup>.

Although several studies reported that CAD-CAM blocks exhibited good mechanical properties<sup>3,5,15</sup>, there was limited research regarding this topic<sup>3</sup>. For example, one study evaluated water sorption and solubility of CAD-CAM resin composite blocks after eight months of storage in water and artificial saliva. However, this study did not assess mechanical properties of these blocks in comparison with ceramics<sup>12</sup>. Another study evaluated sorption, solubility and color stability of CAD-CAM materials after immersion in various beverages<sup>16</sup>. Therefore, comparing water sorption and solubility among different commercially available CAD-CAM materials and their mechanical properties after long immersion up to 6 months can lead to understanding the characteristics of each material after a long period of testing.

There have been studies highlighting the significance of hardness and roughness of CAD-CAM materials after water immersion, since water sorption and solubility of these materials could lead to reduction of their hardness, followed by material degradation<sup>14,17,18</sup>. Furthermore, a decrease in mechanical properties of materials could compromise surface stability leading to increased roughness, compromising the esthetic outcomes of tooth-colored materials<sup>14</sup>. Therefore, it is imperative to assess hardness and

roughness of CAD-CAM materials to ensure their optimal performance and longevity. The aims of the present study were to evaluate water sorption, solubility, and the effect of water immersion on hardness and roughness of commercially available CAD-CAM materials. The first null hypothesis was that water sorption and solubility of aged CAD-CAM blocks were not different from those of un-aged CAD-CAM blocks. The second null hypothesis was that water sorption and solubility of CAD-CAM blocks did not affect hardness and roughness after water immersion. Lastly, the third null hypothesis was that types of CAD-CAM blocks did not exhibit different water sorption, solubility, hardness and roughness.

## Materials and methods

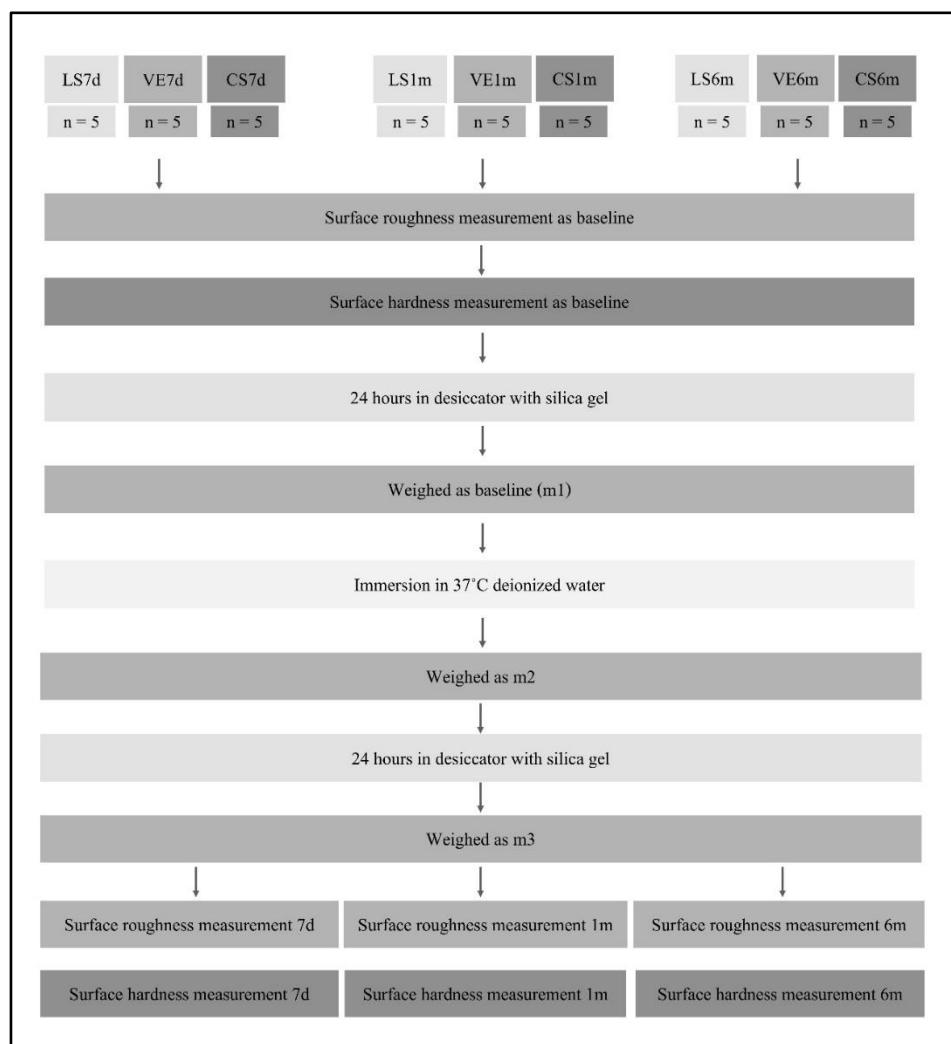
All CAD-CAM materials in this study were commercially available products, including resin composite (Cerasmart, CS; GC, Japan), polymer-infiltrated ceramic network (Vita Enamic, VE; Vita Zahnfabrik GMbH, Germany) and lithium disilicate glass ceramic (IPS e.max CAD, LS; Ivoclar Vivadent, Liechtenstein). G\*power 3.1 software was used to calculate sample size, and found that this study required 45 rectangular-shaped specimens<sup>12</sup>. All specimens were prepared, with an estimated dimension of 12×14×2 mm<sup>3</sup>, by a low-speed cutting machine (Isomet 1000, Buehler Co, USA) under constant water irrigation. Specimens were divided into 9 groups (n=5 per group) by material categories (CS, VE, LS) and immersion times (7 days, 1 month, 6 months) as illustrated in the flow diagram presented in Figure 1. Details about tested materials with their compositions and manufacturers are listed in Table 1.

Each test piece was polished on both sides using a polishing machine (Minitech 233, Presi, France) with a series of silicon carbide papers of decreasing grit size of 15 µm (P1200, 30 seconds), 10 µm (P2400, 30 seconds), and 5 µm (P4000, 30 seconds) (Buehler Co, USA) under continuous flow of water. All specimens were then divided equally into two separate surface areas with an estimated dimension of 12×14 mm<sup>2</sup> on each side, with a T-marker to identify specific areas for surface measurements at each time point (Figure 2).

**Table 1** Commercially available CAD-CAM blocks

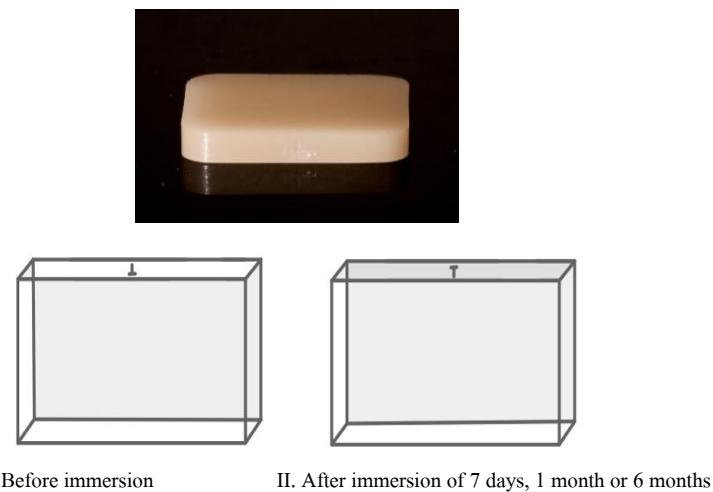
CAD-CAM blocks (Materials; Abbreviation)	Manufacturer	Composition
Resin composite Shade A3 HT (Cerasmart; CS)	GC Corporation, Tokyo, Japan	71% Silica and barium glass nanoparticles, Bis-MEPP, UDMA, DMA
Polymer-infiltrated ceramic network Shade 3M2 HT (Vita Enamic; VE)	Vita Zahnfabrik GMbH, Bad Säckingen, Germany	86% feldspar ceramic enriched with aluminium oxide, UDMA+TEGDMA
Lithium disilicate glass ceramic Shade A3 HT (IPS e.max CAD; LS)	Ivoclar Vivadent, Schaan; Liechtenstein	57.0-80.0% SiO <sub>2</sub> , 11.0-19.0% Li <sub>2</sub> O, 0.0-13.0% K <sub>2</sub> O, 0.0-11.0% P <sub>2</sub> O <sub>5</sub> , MgO, Al <sub>2</sub> O <sub>3</sub>

Abbreviations; Bis-MEPP: Bisphenol-A ethoxylate dimethacrylate; DMA: Dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate; UDMA: Urethane dimethacrylate.



(CS, Cerasmart; LS, IPS e.max CAD; VE, Vita Enamic; 7d, 7 days; 1m, 1 month; 6m, 6 months)

**Figure 1** Flow diagram of research process



**Figure 2** Rectangular-shaped specimen size 12x14x2 mm was divided into 2 separate parts with a T-marker

All specimens from each group were immersed in deionized water in separate containers. Then specimens were subjected to evaluate water sorption and solubility at 0, 7 days, 1 month, and 6 months. Water sorption and solubility were calculated in accordance with ISO 10477:2020.

Firstly, the specimens were stored in a desiccator with silica gel, maintained at  $37\pm1$  °C. After 24 hours, specimens were then removed and weighed as baseline ( $m_1$ ) using a digital balance (AS220/C/2, Radwag, Poland) with a resolution of 0.1 mg. After initial desiccation procedure, specimens were immersed in 70 mL of deionized water in plastic containers and remained there at  $37\pm1$  °C for 7 days, 1 month, and 6 months. Subsequently, specimens were removed and washed with tap water. Thereafter, each specimen was isolated, and any remaining water was absorbed on both sides of an absorbent paper (Raylabcon Inc., USA) until it was visually free from moisture. Afterwards, each specimen was air-dried for 15 seconds and weighed again ( $m_2$ ). Within one minute, the specimen was weighed once more and then returned to its respective vial. Finally, specimens were reconditioned to a constant dry mass ( $m_3$ ) in the desiccator using the abovementioned cycle.

Water sorption and solubility were calculated by using the following equations provided by ISO 10477:2020 standard. Water sorption of all materials within 1 month of the experiment was set not to exceed  $40\text{ }\mu\text{g/mm}^3$ , and solubility of all materials was set not to exceed  $7.5\text{ }\mu\text{g/mm}^3$ .

$$\text{Sorption} = (m_2 - m_3) / v$$

$$\text{Solubility} = (m_1 - m_3) / v$$

$m_1$  was the mass obtained after initial drying and before immersion of the specimen in deionized water.

$m_2$  was the mass of the specimen after immersion in deionized water at 7 days, 1 month, and 6 months.

$m_3$  was the final mass of the specimen.

$V$  was the initial volume of the specimen.

The roughness values were measured using a contact profilometer (TalyScan 150, Taylor Hobson Ltd., Leicester, England) following the recommendations of ISO4288-1985. The 2  $\mu\text{m}$  radius diamond stylus moved diagonally along defined specimen's surface at each time point from the upper left corner downwards to the lower right corner. Three profile measurements were made for each defined specimen's surface, with a 0.25 mm space between them. Mean roughness ( $R_a$ ) was determined with a cut-off value of 0.25 mm and a stylus speed of 0.1 mm/second.

Vickers hardness test was subsequently conducted to evaluate hardness (HVN). The test was performed by using a diamond indenter in accordance with the guidelines provided by ISO6507-1. Vickers hardness tester (Micro-hardness tester-FM810, Type D, Future-Tech, Japan) was used for hardness measurements, by applying a 200-g load with a 10 second dwell time for each indentation. Three indentations were made on the defined surface of each specimen, positioning towards the periphery of the square surface at each time point. The measured values were averaged to determine the hardness values of each specimen at the following timings: at 0, 7 days, 1 month, and 6 months.

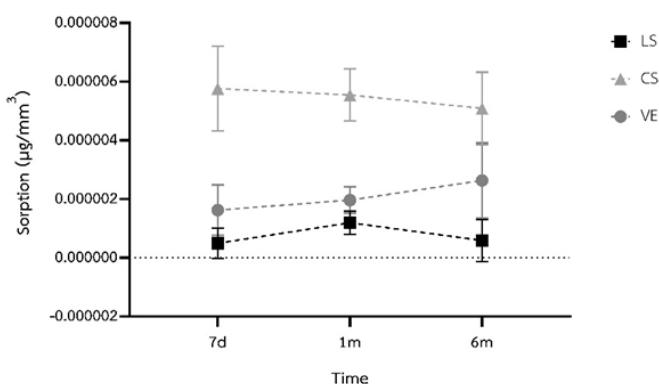
#### Statistical analyses

Data were analyzed using an SPSS statistical software, with 2-way Analysis of Variance (ANOVA) for sorption and solubility, 2-way Analysis of Covariance (ANCOVA) for hardness and roughness, followed by Spearman correlation test. The results of statistical analyses with p-values less than 0.05 were interpreted as statistically significant.

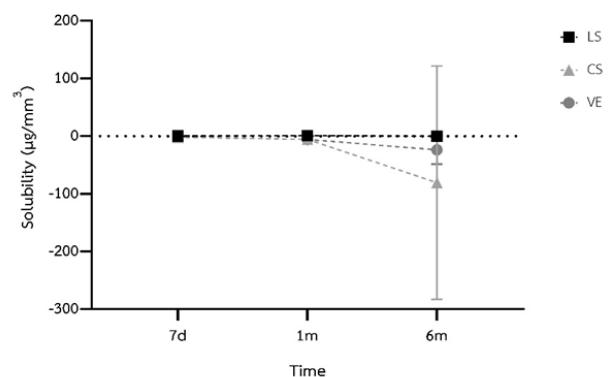
## Results

All specimens were immersed separately in deionized water by 3 groups of immersion periods; 7 days, 1 month and 6 months. From Table 2, the 2-way ANOVA demonstrated that water sorption of those materials was influenced by the variations in their mechanical properties ( $p<0.001$ ), irrespective of immersion time ( $p=1.000$ ). However, solubility was not influenced by their mechanical properties and immersion time ( $p=0.482$ ,  $p=0.326$ ), respectively. Furthermore, Table 3 and Figure 3 demonstrated that there were no significant differences in water sorption between LS and VE group at 7 days and 1 month of water immersion ( $p=0.064$ ,  $p=0.200$ ), respectively. In each group of CAD-CAM materials, there were no significant differences in water sorption observed between 7 days, 1 month, and 6 months of water immersion.

The 2-way ANOVA revealed that there were no significant differences in solubility properties between LS, CS and VE at each time point of water immersion process ( $p>0.05$ ). Furthermore, water immersion did not influence solubility properties of tested CAD-CAM materials ( $p>0.05$ ), regardless of immersion period, as shown in Table 3 and Figure 4.



**Figure 3** Mean water sorption of tested CAD-CAM materials; IPS e.max CAD (LS), Cerasmart (CS), Vita Enamic (VE) at 7 days (7d), 1 month (1m), and 6 months (6m)



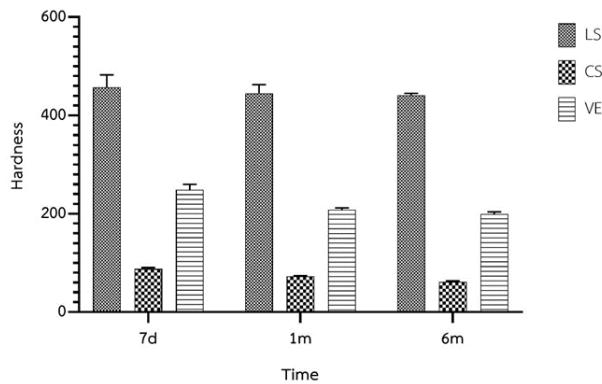
**Figure 4** Mean solubility of tested CAD-CAM materials; IPS e.max CAD (LS), Cerasmart (CS), Vita Enamic (VE) at 7 days (7d), 1 month (1m), and 6 months (6m)

The 2-way ANCOVA revealed that hardness of tested CAD-CAM materials was significantly affected by variations in their mechanical properties and immersion time ( $p<0.001$ ). However, roughness was not found to be influenced by different mechanical properties ( $p=0.526$ ), but rather by immersion time ( $p<0.001$ ), as shown in Table 2.

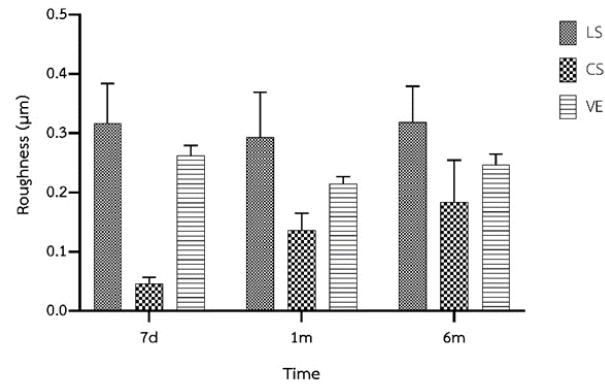
The 2-way ANCOVA illustrated that each duration of water immersion process showed a significant impact on hardness among tested CAD-CAM blocks ( $p<0.001$ ). Moreover, as shown in Table 3 and Figure 5, specifically, in the LS group, hardness significantly decreased after 6 months compared to 7 days ( $p=0.010$ ), but no significant differences were observed between 7 days and 1 month ( $p=0.066$ ) or between 1 month and 6 months ( $p=0.352$ ). For the CS group, hardness significantly differed between 7 days and both 1 month ( $p=0.031$ ) and 6 months ( $p<0.001$ ), with no significant difference between 1 month and 6 months ( $p=0.148$ ). The VE group showed significant differences in hardness between 7 days and both 1 month and 6 months ( $p<0.001$ ), but no significant difference between 1 month and 6 months ( $p=0.209$ ).

From Table 3 and Figure 6, the 2-way ANCOVA indicated significant roughness differences between CS and LS, and between CS and VE, after 7 days of water immersion ( $p=0.032$ ,  $p=0.034$ ), with no significant difference between LS and VE ( $p=0.601$ ). However, no significant differences were found among the CAD-CAM blocks after 1-month and 6-month immersion periods ( $p>0.05$ ). Within the LS group, roughness remained consistent across all immersion periods. In contrast, the CS group showed significant roughness differences between 7 days and both 1 month and 6 months ( $p<0.001$ ), but not between 1 month and 6 months ( $p=0.343$ ). Similarly, the VE group had no significant difference between 7 days and 1 month ( $p=0.616$ ), but showed significant differences between 6 months compared to both 7 days and 1 month ( $p=0.006$ ,  $p=0.012$ ).

Additionally, negative correlations between water sorption and hardness, and water sorption and roughness were observed ( $p<0.001$ ).



**Figure 5** Mean hardness of tested CAD-CAM materials; IPS e.max CAD (LS), Cerasmart (CS), Vita Enamic (VE) at 7 days (7d), 1 month (1m), and 6 months (6m)



**Figure 6** Mean roughness of tested CAD-CAM materials; IPS e.max CAD (LS), Cerasmart (CS), Vita Enamic (VE) at 7 days (7d), 1 month (1m), and 6 months (6m)

**Table 2** 2-way ANOVA and 2-way ANCOVA for CAD-CAM materials(A), immersion time(B), on water sorption, solubility, hardness and roughness of tested CAD-CAM materials

	Sum of squares	df	Mean Square	F	p-value
<b>Sorption</b>					
CAD-CAM(A)	1.768x10 <sup>-10</sup>	2	8.839x10 <sup>-11</sup>	100.914	<0.001 *
Time(B)	0.000	2	0.000	0.000	1.000 *
AxB	0.000	4	0.000	0.000	1.000 *
<b>Solubility</b>					
CAD-CAM(A)	6.877x10 <sup>-9</sup>	2	3.438x10 <sup>-9</sup>	0.745	0.482 *
Time(B)	1.066x10 <sup>-8</sup>	2	5.329x10 <sup>-9</sup>	1.155	0.326 *
AxB	1.048x10 <sup>-8</sup>	4	2.619x10 <sup>-9</sup>	0.568	0.688 *
<b>Hardness</b>					
CAD-CAM(A)	4863.586	2	2431.793	20.507	<0.001 **
Time(B)	7838.782	2	3919.391	33.052	<0.001 **
AxB	1837.036	4	459.259	3.873	0.010 **
<b>Roughness</b>					
CAD-CAM(A)	0.001	2	0.000	0.654	0.526 **
Time(B)	0.023	2	0.012	17.333	<0.001 **
AxB	0.020	4	0.005	7.394	<0.001 **

\*2-way ANOVA; \*\*2-way ANCOVA.

**Table 3** Mean±SD of water sorption, solubility, hardness (HVN) and roughness (Ra) of tested CAD-CAM materials

	CAD-CAM	7 days	1 month	6 months
Mean Sorption ( $\mu\text{g}/\text{mm}^3$ )	LS	0.49±0.52 <sup>A,a</sup>	1.19±0.39 <sup>A,a</sup>	0.59±0.71 <sup>A,a</sup>
	CS	5.76±1.44 <sup>B,a</sup>	5.54±0.89 <sup>B,a</sup>	5.09±1.23 <sup>B,a</sup>
	VE	1.62±0.86 <sup>A,a</sup>	1.97±0.45 <sup>A,a</sup>	2.63±1.28 <sup>C,a</sup>
Mean Solubility ( $\mu\text{g}/\text{mm}^3$ )	LS	0.38±0.45 <sup>A,a</sup>	0.76±0.39 <sup>A,a</sup>	0.02±0.75 <sup>A,a</sup>
	CS	-2.11±0.96 <sup>A,a</sup>	-5.62±1.65 <sup>A,a</sup>	-80.67±202.24 <sup>A,a</sup>
	VE	-0.96±0.49 <sup>A,a</sup>	-5.78±0.80 <sup>A,a</sup>	-23.72±24.73 <sup>A,a</sup>
Mean HVN	LS	457.00±25.40 <sup>A,a</sup>	445.07±17.41 <sup>A,ab</sup>	441.07±3.71 <sup>A,b</sup>
	CS	87.74±2.72 <sup>B,a</sup>	72.50±1.39 <sup>B,bc</sup>	61.36±2.04 <sup>B,c</sup>
	VE	248.46±11.24 <sup>C,a</sup>	208.24±3.60 <sup>C,b</sup>	199.50±4.32 <sup>C,b</sup>
Mean Ra (μm)	LS	0.32±0.07 <sup>A,a</sup>	0.29±0.08 <sup>A,a</sup>	0.32±0.06 <sup>A,a</sup>
	CS	0.05±0.01 <sup>B,a</sup>	0.14±0.03 <sup>A,b</sup>	0.18±0.07 <sup>A,b</sup>
	VE	0.26±0.02 <sup>A,a</sup>	0.22±0.01 <sup>A,a</sup>	0.25±0.02 <sup>A,b</sup>

Abbreviations; CS: Cerasmart; LS: IPS e.max CAD; SD: standard deviation; VE: Vita Enamic.

Values with the same superscript letters represent a non-significant difference.

Capital letter denotes statistical comparison in column. Small letter denotes statistical comparison in row.

According to the equation provided by the ISO 10477:2020 standard, when the final mass (m3) was greater than the initial mass (m1), it might result in a negative solubility value.

## Discussion

This study investigated water sorption, solubility and surface properties of 3 commercially available CAD-CAM materials: Cerasmart, Vita Enamic, and IPS e.max CAD, which were immersed in deionized water for varying periods of time. The correlation between sorption, solubility and surface properties of CAD-CAM blocks were analyzed. The findings indicated that there were no significant differences in water sorption and solubility of each material type during multiple periods of water immersion, therefore, the first null hypothesis was consequently accepted. Furthermore, water sorption of CAD-CAM materials had a significant effect on hardness and roughness in all materials tested. Thus, the second null hypothesis was partially rejected. On the other hand, the results showed significant differences in water sorption, hardness and roughness between different types of CAD-CAM materials, while there were no significant differences in solubility among those tested CAD-CAM blocks, thus, the third null hypothesis was partially rejected. Water sorption and solubility of three distinct CAD-CAM materials were evaluated over a six-month period in deionized water. The findings indicated that there were no significant differences in water sorption and solubility of each material during multiple periods of water immersion. Furthermore, water sorption had a significant effect on hardness and roughness in all materials tested. The results showed significant differences in water sorption, hardness, and roughness between different types of CAD-CAM materials, while there were no significant differences in solubility among those tested CAD-CAM blocks.

In this present study, there were no statistically significant differences of water sorption and solubility of all tested materials between un-aged and aged CAD-CAM blocks, irrespective of immersion time. Water sorption had no significant effect comparing between groups of LS and VE at 7 days and 1 month of immersion periods. However, regardless of immersion time, CS group exhibited a significant water sorption compared to LS and VE groups. As mentioned above, CAD-CAM resin composite blocks were

produced by incorporation of filler particles in monomer mixture<sup>15, 19</sup>, and polymer-infiltrated ceramic network (PICN) blocks comprising a pre-sintered glass-ceramic network infiltrated with a monomer which was then polymerized<sup>15, 20</sup>. Compared to ceramics, resin composites exhibited lower strength and inferior color stability, resulting mainly from their solubility and water sorption<sup>12</sup>. Storage in water induced hydrolytic degradation of the interfacial silane-coupling agent, causing molecular instability<sup>21, 22</sup>. Even though CAD-CAM resin composite blocks have been improved during the past several years, degradation of resin composite could still be observed<sup>18</sup>. Water immersion, therefore, affected mechanical and physical properties of resin composites and induced hydrolytic degradation<sup>23-25</sup>. However, Hibino et al.<sup>26</sup> reported that resin blocks used in their study were considered to have low water sorption because the main monomers was UDMA and Bis-MEPP<sup>26</sup>. Vita Enamic and Cerasmart were made from similar resin matrix monomers, whereas Cerasmart used Bis-MEPP in addition to UDMA. The resin components of Cerasmart blocks were mainly composed of Bis-MEPP, hydrophobic in nature, and UDMA, still hydrophobic but rather hydrophilic compared to Bis-MEPP<sup>27</sup>. Mourouzis et al.<sup>28</sup> stated that TEGDMA and UDMA were the main eluted monomers. However, at the end of the 60-day experimental period, no monomers were detected in distilled water. Leaching pattern of monomers in CAD-CAM resin blocks, as reported in a previous study, decreased over time because the materials were highly polymerized into ready-to-mill blocks and possessed better chemical properties<sup>28</sup>. Although water was likely to penetrate into resin blocks when immersed in water, matrix resin components would absorb less water because of their hydrophobic nature. Therefore, water may have had a greater effect in reducing bonding of resin matrix and filler in Cerasmart blocks<sup>26</sup>. Water sorption of CAD-CAM composite blocks was dependent on the resin-matrix composition and influenced by the weight percentage of the fillers<sup>12</sup>. As the filler percentage increased, the polymer matrix percentage decreased, resulting in less water diffusion<sup>29, 30</sup>. The study by Goujat et al. revealed that mechanical properties of resin blocks were more dependent

on their structural composition, including filler content and matrix distribution, rather than just chemical properties of the monomers used<sup>5</sup>. As a result, despite the hydrophobic nature of Bis-MEPP in Cerasmart, it showed higher water sorption compared to Vita Enamic, highlighting the importance of structural factors over purely chemical ones in determining material performance. Alamoush et al.<sup>12</sup> who reported that CAD-CAM composite blocks exhibited hydrolytic stability during long-term storage. However, their stability was found to be comparatively lower than that of ceramics<sup>12</sup>. In addition, according to ISO standards, the initial and final dehydration of specimens could affect solubility of the tested materials<sup>31</sup>. Some of the water absorbed might interact with resin matrix<sup>32</sup> and, therefore, was not completely removed by desiccation after water storage<sup>33</sup>. Furthermore, the negative solubility values of Vita Enamic and Cerasmart did not necessarily imply that these materials were insoluble, as it could be the result of incomplete dehydration<sup>12</sup>. However, previous studies reported that negative values might be the result of water penetrated into the surface of materials. This phenomenon might be ascribed to hydrophilicity of resin matrix and/or hydrolytic instability of the interfacial coupling between filler and resin matrix<sup>11, 16, 30</sup>. Additionally, water molecules penetrated into spaces between chains of polymer molecules, causing changes in dimension and volume of material<sup>34</sup>. The solubility variations observed in the CS and VE groups after six months of immersion could be attributed to several factors. Söderholm et al.<sup>35</sup> noted that barium-containing filler particles were susceptible to leaching, and in the case of Cerasmart, the filler particles were relatively large, small, and uniformly distributed<sup>36</sup>. Extended immersion might weaken the bond between resin matrix and filler particles, leading to filler detachment<sup>26</sup>. This detachment, particularly of fillers of varying sizes, might allow water to penetrate, contributing to observed solubility variations after six months. Beyond filler content, polymer matrix exhibited a crucial role in solubility after prolonged immersion. Mourouzis et al.<sup>28</sup> found that Vita Enamic was the only material to release TEGDMA in a distilled water solution over different experimental periods due to its molecular structure and higher solubility<sup>28</sup>. This

leaching of TEGDMA, likely due to hydrolytic degradation, could explain the solubility changes in Vita Enamic after six months. Alamoush et al.<sup>12</sup> monitored the pH of the solution during long-term immersion in water and artificial saliva, a factor that could affect monomer elution and the degradation of CAD/CAM resin composite materials<sup>28</sup>. However, the current study did not monitor the pH levels of the storage medium, which might have influenced monomer elution, contributing to material degradation and increased water uptake.

It was more difficult for water to penetrate into ceramics in comparison to polymer-based materials under test<sup>37</sup>. However, various studies reported significant changes in surface properties of ceramic materials following immersion procedures<sup>14, 21, 38-40</sup>. The present study found a significant change in roughness of LS group between 7 day and 6-month immersion times, which coincides with some previous studies. For example, Milleding et al.<sup>38</sup> stated that once a ceramic material was exposed to an aqueous environment, leaching out of easily-released alkali oxides (sodium and potassium) from glass matrix might occur, creating porosities and channels within the glass matrix allowing further diffusion of water molecules. Subsequently, this process might lead to disruption of the bonded glass network (Si-O-Si)<sup>38</sup>. According to Alencar-Silva et al.<sup>14</sup> who reported that significant changes in roughness, hardness, and color stainability were observed in CAD-CAM lithium disilicate ceramic after immersion in tested beverages<sup>14</sup>, which was in line with Musanje et al.<sup>21</sup> who reported that water sorption not only affected physical and mechanical properties, especially of resin composite, but also decreased hardness and elastic modulus<sup>21</sup>. Al-thobity et al.<sup>39</sup> also reported that it might be due to leaching of alkali ions from exposed surfaces of ceramics which made them rougher. Furthermore, roughening of ceramic surfaces could jeopardize the structure of the material and increase plaque accumulation<sup>39</sup>. In addition, Kukiatrakoon et al.<sup>40</sup> evaluated ion leaching of 4 different ceramics after being immersed in acidic agents and deionized water. The crystalline phase of ceramics was found much higher in acidic

agents. They demonstrated that there was a large number of ions leaching out of various ceramics immersed in all acidic agents and deionized water<sup>40</sup>. One possible explanation that Al-thobity et al.<sup>39</sup> reported was that aqueous solutions substantially weakened the structure of glass-based ceramics (feldspathic porcelain) while structures of crystalline-based ceramics (zirconia and lithium disilicate) were not affected<sup>39</sup>. However, Kukiatrakoon et al.<sup>40</sup> reported that all aqueous solutions including saliva had a significant effect on roughness of ceramics including lithium disilicate and zirconia<sup>40</sup>. Although chemical degradation of ceramics has been demonstrated to be associated with elution of alkali ions<sup>41</sup>, due to their homogeneous structure and toughness, and lithium disilicate showed much greater long-term resistance to chemical and mechanical degradation<sup>7</sup>.

The present study found that VE group showed no significant change on roughness within 30 days. However, there was a significant difference in comparing short-term and long-term immersion time as reported in a previous study. In the same trend, Mourouzis et al.<sup>28</sup> stated that surface of Vita Enamic after 60-day immersion did not differ significantly<sup>28</sup>. As a consequence of the study, it was noted that using a single method to evaluate roughness could be misleading<sup>42</sup>, which was consistent with Amaya-Pajares et al.<sup>43</sup> who stated that highly variable Ra values have been reported for ceramics because they relied on the composition of the material, method of fabrication, measurement methods, and surface treatment<sup>43</sup>. They also relied on instrumentation-dependent factors including stylus or probe size, scanning speed, frequency response and sampling rate of the recording instrumentation, limitations due to feature slope or sharpness, sampling length or area and type of software used to filter and refine the raw data<sup>43</sup>. Roughness in dentistry is commonly described using the Ra parameter, which is determined by various machines and techniques. However, direct comparison of Ra values could be challenging due to variations in techniques. Ra value does not completely describe the surface of a material. However, this Ra value, which is easy to calculate, gives a representative estimate of roughness<sup>43</sup>. Moreover, previous studies also mentioned that

high crystal content in a ceramic material could produce an uneven surface when polished<sup>44, 45</sup>. Morphology of a surface depended on the scale of observation and different measuring methods used. Numerical characterization of roughness varied depending on the roughness parameter chosen and the measuring equipment used<sup>46, 47</sup>. Therefore, SEM analysis was performed after profilometer measurements in the evaluation of roughness as shown in many studies<sup>48, 49</sup>. This study did not prepare the specimens in a disk shape as specified by ISO 4049 due to the limitations of using a Cerec milling machine and its associated diamond milling tools. However, this did not affect the required flat surface area for the experiments.

There have been a few in vitro tests that seemed to reveal limited correlation of short-term clinical performance of ceramics and resin composite indirect restorations<sup>50, 51</sup>. There were no methods which could reliably predict long-term clinical performance<sup>50</sup>. Clinical studies, that have evaluated lithium disilicate glass ceramic or resin composite indirect restorations separately, found good clinical short-term and long-term performance with slightly different success rates in favor to ceramics<sup>7, 52-54</sup>. The properties of a material, including its sorption, solubility, and surface properties, are highly dependent on various external factors including surface finishing treatments, chemical structure of the material itself, and type of solutions it is exposed to. However, limitations in the study included the inability to replicate factors such as saliva, consumption habits, thermal changes, oral hygiene or tooth brushing habits, smoking, and both functional and parafunctional forces. Therefore, it is necessary to conduct studies that closely simulate clinical situations to obtain more comprehensive results.

## Conclusion

Within the limitations of this study, three commercially available CAD-CAM blocks, IPS e.max CAD, Vita Enamic, and Cerasmart, exhibited different levels of water sorption when subjected to prolonged water immersion for up to 6 months. Both aged and unaged specimens of these materials showed similar sorption and solubility during the immersion process. However, the water sorption properties

influenced their hardness and roughness after immersion. Although the three materials displayed varying degrees of water sorption, hardness, and roughness, they did not exhibit significant differences in solubility after 6 months of water immersion.

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# การดูดนำ การละลายตัว และคุณสมบัติพิเศษของวัสดุแอดแคม

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บทความวิจัย

## บทคัดย่อ

งานวิจัยฉบับนี้มีวัตถุประสงค์เพื่อศึกษาเปรียบเทียบคุณสมบัติการดูดนำ การละลายตัว และคุณสมบัติพิเศษของวัสดุแอดแคม 3 ชนิด. เรซินคอมโพสิตเชร่าสมาร์ท เรซินเซรามิกวีตี้เอ็นามิก และลิทียัม ไดซิลิเกตคลาสเซรามิก โดยเครื่องวัดคุณภาพสีเทาลี่มีค่าเฉลี่ย 45 ชั้น จากนั้นนำไปแข็งในน้ำปราศจากไอออนในระยะเวลา 7 วัน, 1 เดือน และ 6 เดือน เพื่อนำไปทดสอบคุณสมบัติการดูดนำ การละลายตัว ความแข็งพิเศษและความหมายพื้นพิว การทดสอบพบว่าระยะเวลาที่ใช้ในการแข็งวัสดุส่งผลต่อการดูดนำของวัสดุที่ถูกทดสอบทั้ง 3 ชนิดอย่างมีนัยสำคัญทางสถิติ ( $p<0.001$ ) อย่างไรก็ตามระยะเวลาที่ใช้ในการแข็งวัสดุไม่มีผลต่อการละลายตัวของวัสดุอย่างมีนัยสำคัญทางสถิติ ( $p>0.05$ ) เมื่อวิเคราะห์ข้อมูลโดยใช้ 2-way ANCOVA พบว่าการแข็งวัสดุแอดแคมมีผลต่อความแข็งพิเศษของวัสดุแต่ละชนิด ( $p<0.001$ ) แต่ไม่มีผลต่อความหมายพื้นพิวของวัสดุอย่างมีนัยสำคัญทางสถิติ ( $p>0.05$ ) จากการศึกษาพบว่าความหมายพื้นพิววัสดุกลุ่มลิทียัม ไดซิลิเกตคลาสเซรามิก ไม่มีความแตกต่างอย่างมีนัยสำคัญทางสถิติ เมื่อเปรียบเทียบแต่ละช่วงเวลา ในขณะที่เรซินคอมโพสิตเชร่าสมาร์ท เรซินเซรามิกวีตี้เอ็นามิก ให้ผลการศึกษาคุณสมบัติพิเศษมีความแตกต่างอย่างมีนัยสำคัญเมื่อเปรียบเทียบระหว่างกันที่แข็งตัวที่สุด ( $p<0.05$ ) จากการศึกษาพบว่าคุณสมบัติการดูดนำของวัสดุแอดแคมมีความสัมพันธ์กับความแข็งพิเศษและความหมายพื้นพิวของวัสดุ ( $p<0.001$ ) ทำให้สรุปได้ว่าวัสดุทั้ง 3 ชนิด มีคุณสมบัติการดูดนำที่แตกต่างกันหลังจากแข็งในน้ำ 6 เดือน และเมื่อเปรียบเทียบระหว่างก่อนแข็งและหลังแข็งวัสดุทั้ง 3 ชนิด ไม่มีการเปลี่ยนแปลงคุณสมบัติการดูดนำและ การละลายตัว อย่างไรก็ตามการแข็งน้ำปราศจากไอออนส่งผลต่อคุณสมบัติพิเศษของวัสดุแอดแคมทั้ง 3 ชนิด และแม้เมื่อวัดวัสดุทั้ง 3 ชนิดมีความแตกต่างในการดูดนำ ความแข็งพิเศษ และความหมายพื้นพิว แต่กลับไม่พบการละลายตัวที่แตกต่างกันหลังแข็งนาน 6 เดือน

คำนำรักษ์: วัสดุแอดแคม/ การละลายตัว/ ความแข็งพิเศษ/ ความหมายพื้นพิว/ การดูดนำ

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