J Nanostruct 15(1): 114-121, Winter 2025

RESEARCH PAPER

The Effects of Different Camphorquinone Concentrations on the Water Sorption and Solubility of an Experimental Green-Based Flowable Resin Composite

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ARTICLE INFO

Article History:
Received 13 September 2024
Accepted 25 December 2024
Published 01 January 2025

Keywords:
Camphorquinone
Experimental flowable composite
Nanohybrid silica
Novel composites
Photoinitiator
Water sorption and Solubility

ABSTRACT

Water sorption (WS) and solubility (S) of the experimental flowable composites (EFCs), made from silica filler of rice husk are important to explore the reaction of EFCs against different oral environments. To investigate the effects of different camphorquinone concentrations on the WS and S of the EFCs. EFCs were fabricated from nanohybrid silica and involved different weights of camphorquinone (CQ) (0.03 g, 0.05 g and 0.075 g) and other materials. 3 groups of 7 disk-like samples of each of the three EFCs and Revolution Formula 2 (RF2) were tested. Before immersion, the volumes were recorded and the mass of each sample was recorded, as m1. During the immersion phase in three different media for 7 days, the weight was recorded as m2. During the drying phase at 37◦C for 7 days, the weight was recorded as m3. The WS and S of EFCs and RF2 were calculated and compared. Data from all tests were analyzed using one-way ANOVA and post hoc Dunnett T3 tests at a significance level of 0.05. In all immersion media, WS and S of EFC CQ 0.05 were significantly higher than EFC CQ 0.03 and EFC CQ 0.075. However, the three experimental groups showed significantly higher values compared to RF 2 ($p < 0.05$). Although the outcomes were suboptimal, EFCs have the potential to be improved and be a sustainable product that can benefit the dental field and the environment.

How to cite this article
Jie G., AL-Jobory A., Fatalla A. et al. The Effects of Different Camphorquinone Concentrations on the Water Sorption and Solubility of an Experimental Green-Based Flowable Resin Composite. J Nanostruct, 2025; 15(1):114-121. DOI: 10.22052/JNS.2025.01.011

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INTRODUCTION

Flowable resin composite (FRC) was first introduced in late 1996 [1] as a way to modify the putty-like consistency of resin-based composites while also increasing their handling ability. Reducing the filler quantity or changing the viscosity of the monomer mixture is one of the ways to make the composite less viscous [2]. Due to its lower viscosity, FRC has better wettability to the tooth surface, allowing it to flow into the desired locations; however, the stress produced during polymerization shrinkage of composite resin can be high [2]. FRCs have versatile clinical uses and are quite beneficial in the restorative and other dentistry fields [3].

Commercial FRC and other types of resin composites normally contain fillers such as silica, silicate glass (borosilicate glass, barium or lithium aluminium silicate), quartz, strontium, barium, zinc, or zirconia. None of these fillers come from biological sources. A good alternate source of silica is rice husk, an agricultural biowaste. Bio-based materials have the benefits of being eco-friendly, renewable, and sustainable with little negative impact on the ecosystem [4].

Recently, scientists developed a project to make experimental flowable composites (EFCs) from the rice husk's silica [4-7]. New EFCs were made using nanohybrid silica from rice husk through the sol-gel technique by combining triethylene glycol dimethacrylate (TEGDMA) monomer with bisphenol A-glycidyl methacrylate (Bis-GMA) monomer. Then, camphorquinone (CQ) and 2-(Dimethylamino)ethyl methacrylate (DMAEMA) are added during the preparation of EFC samples. During the initiation step of resin composite polymerization process, the CQ, which acts as the photo-initiator, absorbs the energy produced when exposed to irradiation light, which causes the CQ to enter an excited "triplet state". After this, CQ reacts with DMAEMA (the photo-initiator system) to produce a reactive complex which is the responsible for the composite polymerization [5].

Water sorption (WS) and solubility (S) of the EFCs are important for us to study the reaction of the EFCs against different oral environments. Previously, a study [6] showed that the water sorption of EFCs was significantly higher than that of the commercial flowable composite. Hence, future improvements to the materials' qualities should be made in order to meet the ISO standard and make them competitive with their commercial products. In this study, different concentrations of CQ were added during the preparation to produce three different materials. The water sorption and solubility of each material were evaluated after immersion in three different media, which are distilled water, orange juice, and sports drink. Therefore, the aim of this study was to investigate the effects of different camphorquinone concentrations on the water sorption and solubility of an experimental flowable resin composite.

MATERIALS AND METHODS

Sample size calculation

G* power 3.1.9 software was used to determine the minimum acceptable sample size for this study. The sample size was calculated based on a one-way ANOVA (Fixed value, Omnibus). The parameters required for the calculation were: effect size of 0.5, α error probability of 0.05, power of 0.8, and number of groups of 12 (3 groups of each tested material, which are 3 experimental material and one commercial composite). The total sample size was 84, so each of the 12 groups has a sample size of 7. The quantity of material tubes needed was calculated to provide the correct sample quantity in the water sorption and solubility experiment. Since the sample was of a cylinder shape (the mold used was 10 mm in diameter and 1mm in height), a mathematical equation for cylinder volume was used to obtain the volume of each sample, and then the result was converted to milliliters as shown below. A syringe of 1 ml capacity was used to hold the material inside. Calculation of the volume needed for each sample:

$$\text{Volume (V)} = \pi * R\text{adius}(r2)^* \text{ Height (h)}$$

Fabrication of experimental FRC

Weight measurements were carried out with (Sartorius BSA 4235). Total 2 g Bis-GMA and TEGDMA (Esstech Inc., USA) were added in a mortar to produce 40/60 wt.%. Afterward, 3 different concentrations of CQ and 0.02g of DMAEMA (Merck, Germany) were added to the mix. At the final step, 2 g of nanohybrid silica (Produced locally at Universiti Sains Malaysia by following the steps demonstrated by Noushad et al., 2016 [7] particles processed from rice husks were applied incrementally and then combined and stirred using a pastel until they appeared as a homogeneous paste. The vortex was used for a more homogeneous mixture after the manual mixing process. The obtained composite paste was packed in a disposable syringe wrapped with aluminum foil (Terumo Corporation, Tokyo, Japan) without a needle tip and then the syringe was stored in a refrigerator. The steps of fabrication of the experimental FRC were adopted from [4].

Preparation of test specimens

Stainless steel mold with a thickness of 1 mm and a diameter of 10 mm was used to prepare disc shaped samples (n=7) which were prepared with two slides of glass, lined with two mylar strips. The prepared EFCs and the commercial flowable composites were injected into the mold and pressed from the top surface of the upper glass slide to ensure creation of samples without defects or air bubbles. A light-curing device was used to irradiate all specimens from the top surface for 40 seconds at a power of 1450 mW/cm2. The sample was exposed to 40 seconds 4 times in four different locations to obtain 160 seconds total over one surface. The cured samples then were stored in an oven at 37ºC ±1 and then a desiccator at 23ºC for 24 hours one day before the immersion process, and their masses (m1) were reported by using Sartorius BSA 4235 balance. These processes were repeated until constant masses (m1) were reached. To determine sample volumes, diameter and thickness of each sample were measured by using a digital caliper (Mitaka, Japan). Seven specimens of each material were then located in a sealed 10 ml filled with immersion media plastic container (distilled water, sports drink and orange juice). All the containers were positioned in a water bath at 37◦C±1. In the first week, measurements of weight for each sample were conducted on the 1st, 3rd and 7th day after sample preparation phase. Throughout the tests, each sample was collected, dried to remove excess fluid, weight measured, and returned back to its container in the water bath.

In the second week, which the immersion phase, the m2 weight measurements were carried out on the 1st, 3rd and 7th day of immersion to ensure full absorption of the composite. In the third week, which is the drying phase, reconditioning of samples was done in the oven at 37º ±1 and weighed on the 1st, 3rd and 7th day of drying phase to obtain a constant mass of m3. Water sorption and solubility for FCs were determined using the following formulas in accordance with ISO 4049:2000:

$$\text{Water sorption equation } WS = (m2 - m3)/v$$

$$\text{Solubility equation } S = (m1 - m3)/v$$

From the equations, the conditioned mass refers to m1, while the mass after 7 days immersion

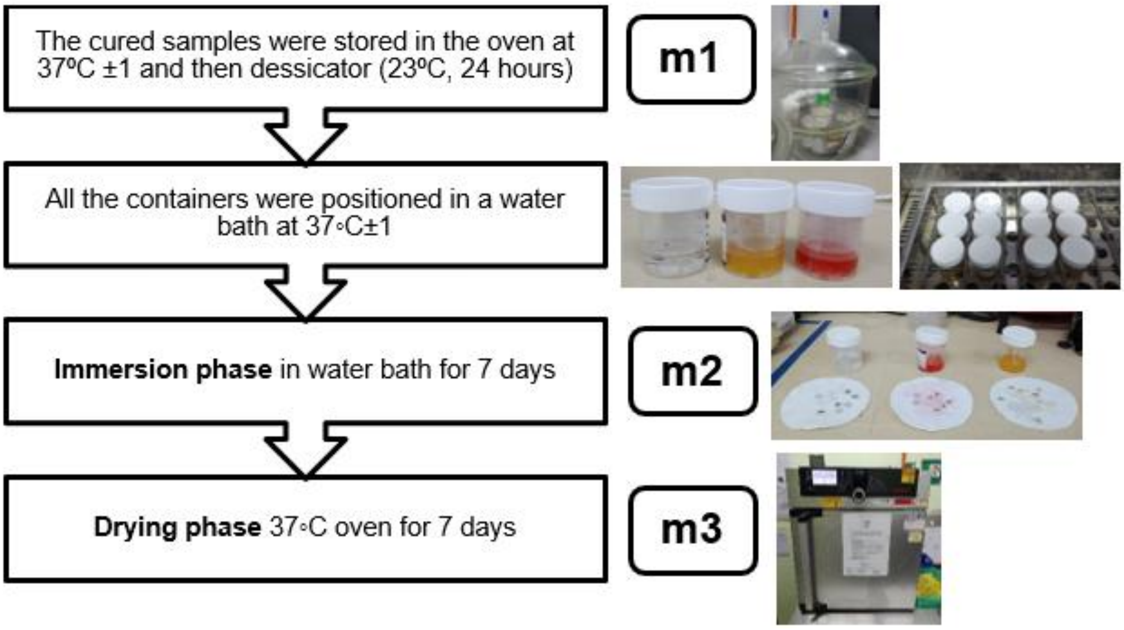

Fig. 1. Summary of the steps in the water sorption and solubility procedure.

refers to m2 and the mass of the reconditioned specimen refers to m3. Under ISO 4049:2000 for polymer-based material guidelines, the values of WS should be either equal to or less than 40 µg/mm3 to pass the ISO requirements, while for the S values, the values should be either equal to or less than 7.5 µg/mm3. The above methodology was adopted from Rahim et al. [8]. Summary of the steps are shown in Fig. 1.

Statistical Method

The statistical analysis was performed using IBM SPSS software version 27.0. The data from the experiment was presented as mean and standard deviation. The parameters were tested for normality by using Kolmogorov-Smirnov test. After running a normality test, the results were normally distributed. One-way ANOVA with Dunnett t3 post hoc tests at p = 0.05 significance level were used.

RESULTS AND DISCUSSION

One-way ANOVA was chosen to test the significance of the results because there were four test groups and the results were normally after running a normality test. Table 1 displays the water sorption (WS) means and standard deviations for EFCs and RF2, while table 2 displays the solubility (S) means and standard deviations for EFCs and RF2. One-way Anova with Dunnett t3 post hoc tests at p = 0.05 significance level were used and detected a significant difference among all test groups for all immersion media. However, for the multiple groups' comparison (post hoc), some groups showed non-significant differences. In all media, water sorption of EFC CQ 0.05 was significantly higher than EFCs CQ 0.03 and CQ 0.075 ($p < 0.05$) as shown in Table 1. However, the three experimental groups showed significantly higher values compared to RF 2 ($p < 0.05$) as shown in Table 1.

In distilled water, sports drink and orange juice media, the solubility of EFC CQ 0.05 was significantly higher than EFC CQ 0.03, EFC CQ 0.075 and RF2 ($p < 0.05$) as shown in table 2. However, the three experimental groups showed higher values compared to RF2.

Fig. 2 shows the plots of mass 1, 2 and mass 3 changes before and during the immersion and drying process. Mass 2 is related to water sorption

Table 1. Water sorption results of four different testing groups in three different immersion media.

Media	Water sorption (µg/mm^3) means and standard deviations				F (df) or Statistic (df1, df2)^	p-value*
	RF2	EFC CQ 0.03	EFC CQ 0.05	EFC CQ 0.075		
Distilled water	8.18 $^{(0.94)}$	111.16 $^{(14.60)}$	231.78 $^{(33.08)}$	169.72 $^{(28.19)}$	120.441 (3, 24)	<0.001
Sports drink	8.32 $^{(1.57)}$	113.87 $^{(8.31)}$	186.65 $^{(22.67)}$	125.51 $^{(10.89)}$	218.229 (3, 24)	<0.001
Orange juice	7.71 $^{(1.24)}$	126.82 $^{(18.92)}$	201.26 $^{(36.37)}$	115.34 $^{(34.16)}$	62.478 (3 ,24)	<0.001

^Data analysis was done by using one-way Anova and Dunnett T3 post hoc tests.

*The mean difference is significant at the 0.05 level.

The differences between all groups in Dunnett t3 post hoc test were significant, except for the differences between sports drink group (EFC CQ 0.03 vs. EFC CQ 0.075) and orange juice group (EFC CQ 0.03 vs. EFC CQ 0.075).

Table 2. Solubility results of four different testing groups in three different immersion media.

Media	Solubility (µg/mm^3) means and standard deviations				F (df) or Statistic (df1, df2)^	p-value*
	RF2	EFC CQ 0.03	EFC CQ 0.05	EFC CQ 0.075		
Distilled water	-1.17 $^{(8.08)}$	62.74 $^{(14.72)}$	139.41 $^{(41.02)}$	137.58 $^{(61.64)}$	22.345 (3, 24)	<0.001
Sports drink	-0.60 $^{(0.90)}$	67.87 $^{(21.17)}$	94.19 $^{(31.02)}$	59.56 $^{(19.51)}$	25.066 (3, 24)	<0.001
Orange juice	-1.48 $^{(1.35)}$	63.48 $^{(19.29)}$	108.79 $^{(37.23)}$	57.75 $^{(39.88)}$	17.120 (3 ,24)	<0.001

^Data analysis was done one-way Anova and Dunnett T3 post hoc tests.

*The mean difference is significant at the 0.05 level.

The differences between all groups in Dunnett t3 post hoc test were significant, except for distilled water group (EFC CQ 0.03 vs. CQ 0.075 and EFC CQ 0.05 vs. CQ 0.075), sports drink group (EFC CQ 0.03 vs. CQ 0.05, EFC CQ 0.03 vs. CQ 0.075 and EFC CQ 0.05 vs. CQ 0.075) and orange juice group (EFC CQ 0.03 vs. CQ 0.05, EFC CQ 0.03 vs. CQ 0.075 and EFC CQ 0.05 vs. CQ 0.075).

of the FCs that were immersed in different media. The plots showed a linear increase in earlier stages with a degree of difference between the test groups depends on the previous results shown. However, they became plateau at the end of the immersion process in most of the groups. Mass 3 changed after immersion (drying process) which are related to the solubility of the FCs immersed in different media. This part of graphs reflected the linear changes in downward directions due to loss of weight during or after immersion. However, both parts of graphs became plateau at the end. There was a noticeable loss of mass of the tested EFCs when comparing day 1 mass measurement to the last day of drying process. This mass change was not evident with regard to commercial FC group.

Any kind of dental composite that is present in the oral environment is periodically or continuously exposed to substances that can alter its chemical breakdown, such as those found in foods, drinks, and saliva [6].

Although the International Organization for Standardization (2000) established ISO 4049:2000 guidelines specifying that WS and S testing for polymer-based filling materials should be conducted using water, results from a number of studies indicated that the behavior of resin composites when submerged in oral fluids, like acids, is also essential for identification and appreciation [9].

The absorption of water into the polymer matrix is regulated by the process of Fickian (type I) diffusion [10]. Diffusion in polymers is described by the two main models. According to the “free volume theory,” water enters nanopores without reacting chemically with polymer chains. According to “interaction theory,” water diffuses through the substance and binds to the hydrophilic groups progressively [11]. Consequently, there are two different types of absorbed water: “unbound water,” which fills the space between the chains of polymers and the nanopores formed during polymerization [12]; and “bound water,” which is bonded to the chains of polymers by hydrogen bonds [13]. A decrease in shrinkage stress is correlated with the quick elution of unbound water molecules into free volume between the chains and crosslinks. A further drop in stress levels is the consequence of the slow uptake of water till saturation [14].

Table 1 shows that WS results are ranged from 111.16 to 112.82 µg/mm3 for EFC CQ 0.03, from 186.65 to 231.78 µg/mm3 for EFC CQ 0.05, from

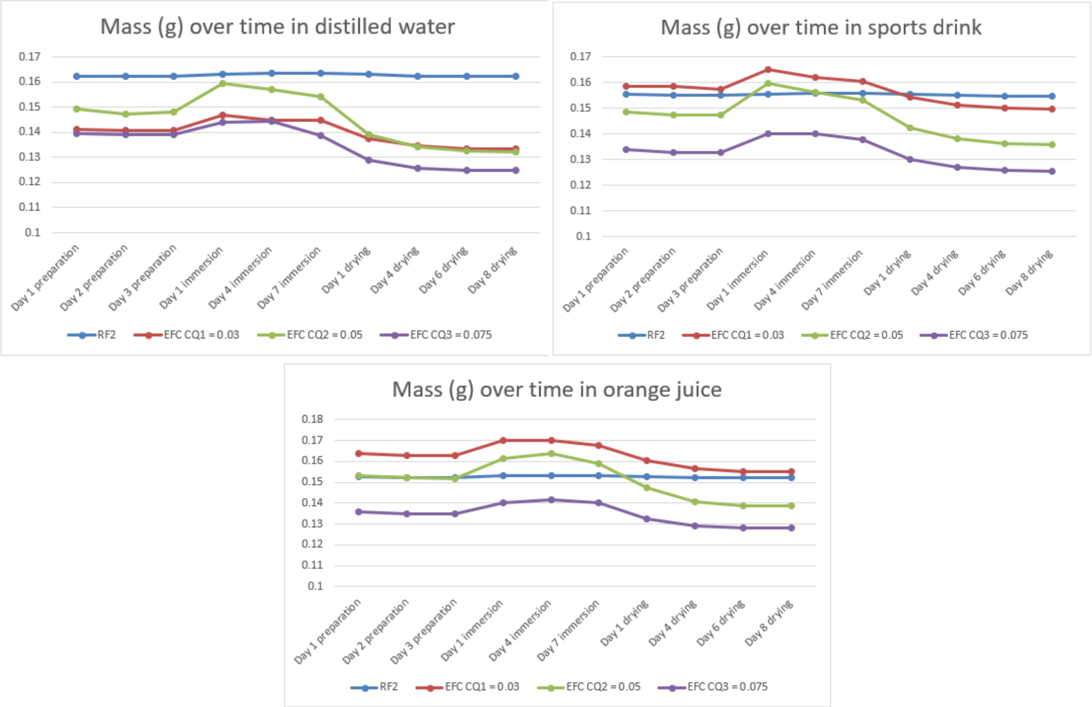

Fig. 2. Mass 1, 2, 3 changes before, during and after immersion of EFCs and RF2 in three different immersion media.

115.31 to 169.72 μg/mm3 for EFC CQ 0.075 and from 7.71 to 8.32 μg/mm3 for RF2. The water sorption values for the three experimental test groups did not pass the ISO 4049 standard, which stated that maximum water sorption is equal or below 40 μg/mm3 (International Organization for Standardization, 2000). All samples related to RF2, which were immersed in four different media, did pass the ISO standard. The null hypothesis was rejected.

Post hoc analysis detected statistically significant difference of water sorption ($p<0.05$) among all the test groups in all immersion media except for group sports drink (EFC CQ 0.03 vs. EFC CQ 0.075) and orange juice (EFC CQ 0.03 vs. EFC CQ 0.075).

When compared to other EFC test types, RF2 showed the lowest WS values. This could be as a result of the resin matrix combined with glass fillers, which are less hydrophilic than fillers based on silica. Even though other studies showed that RF2 has irregularly shaped fillers, and this is supported by other findings [1], which may restrict its packing density. Better filler packing in the polymer matrix with a smooth, homogenous network and few microvoids can also result in lower water sorption and solubility values.

The porous nature of the silica fillers in EFC groups can account for a higher degree of WS [8]. According to Curtis et al. [15], the more surface area per volume of nanosized fillers, the more water-susceptible hydrophilic sites there are, which allows fluids to pool near the filling-polymer interface. A higher level of silanization is essential because of the high-volume percentage of nanoparticles.

In resin-based composites systems, the photoinitiator concentration should ideally be kept to a minimum to encourage maximal monomer conversion [16]. Based on experimental findings by Maciel et al. [17], increasing the CQ amount up to 1% resulted in a greater degree of monomer conversion, which in turn decreased polymer breakdown and increased cure depth, meaning that the lower concentration had better attributes. The inclusion of light-absorbing photoinitiators in resin composites, according to Asmusen et al. [18], invariably causes attenuation of the light intensity along the radiation channel and frequently restricts the depth of cure of these materials. Nevertheless, absorbance decreases and attenuation reduces when the photoinitiator photo bleaches because the photoinitiator consumption is accompanied by a deeper penetration of light through the composite thickness. With greater concentrations of CQ, the excess of unreacted photoinitiator in composites prevents photobleaching and prevents the lowering of light attenuation. As a result, depth of cure is lower in composites with higher concentration of CQ. Yap et al. [19] state that a polymer system's cross-link density has a significant impact on the mechanical and physical characteristics of the final material. Less depth of cure means less number of bridges are formed between linear macromolecules, less crosslinking lowers the polymers' permeability and increase their water sorption and solubility [20]. This could be the reason why there is higher water sorption and solubility results when there is an increasing concentration of CQ.

High WS can also result from the erosive effect of acidic fluids on the material surface [21]. When materials are subjected to acidic solutions such as in 100 plus sports drink and orange juice, the number of barriers preventing water molecules from passing through the material structure decreases, resulting in a higher WS value. Water molecules encourage composite deterioration by two mechanisms. The first mechanism is the monomers elution. Initially, water molecules seep into the polymer network and fill the gaps in the structure, which can cause the matrix to become plasticized and inflate as well as break the chains, which results in the elusion of monomers [15]. Another mechanism is filler debonding. It results from the hydrolysis reaction of the siloxane bond (silica surface and coupling agent bond) being broken down by the water molecules [15]. The ultimate outcome is a deterioration or softening of the resin composite, which may reduce some of the material's physical properties such as strength, hardness, and elasticity modulus. The high acidity of 100 plus ® sport drink, and orange juice (pH=3.88 and 3.68 respectively) may be the ideal plasticizer for accelerating water absorption, by decreasing interactions in polymers [6].

According to the results in this study, the levels of WS in water for EFC CQ 0.05 and EFC CQ 0.075 were greater than those of other acidic media. The outcomes of EFC CQ 0.03 water sorption in orange juice and 100 plus® were comparable, but higher than the values in the water medium. The previously stated information may help to explain the causes of these variations in the results. The compositions and ratios of the monomers, the surface treatment of the fillers, and the composite preparation procedures can all be improved. As a result, more research is required to address the problem of water sorption so that the newly developed materials can satisfy the ISO standard for clinical applications.

According to Da Silva et al. [9], resin composite S denotes the release of unreacted monomers, filler particles, and ions from their surfaces. Solubility is measured based on the weight loss during the drying process of the samples. The resin either softens due to the plasticizing effect of water or breaks down chemical connections within the resin, causing hydrolytic breakdown. Certain components of resin, such unreacted monomers or fillers, disintegrate and leach out of the samples when they are submerged in water [6].

Compared with ISO requirements, all EFC CQ 0.03, EFC CQ 0.05 and EFC CQ 0.075 from all groups (distilled water, 100 plus, orange juice) results showed S higher than the maximum requirement, which is 7.5 µg/mm3. All RF2 results exhibited S inferior to the maximum value from the ISO standards (7.5 µg/mm3) as shown in Table 2. High composite S values are frequently associated with low packaging density and degree of conversion [22,23].

Some negative values for RF2 and experimental flowable nanocomposites suggested that not all of the absorbed water had been eliminated during the drying process. According to Ortengren et al. [24], the desorption of water in the fillers was thought to be complicated by the high porosity of the silica filler and agglomeration.

Acids were originally intended to assist in the removal of unreacted monomers and fillers. However, they can also lead to corrosion of filler surfaces, causing particles to detach and increasing the overall loss of composite material [6]. Furthermore, acids can create a lower pH environment within the resin matrix, triggering ester hydrolysis of dimethacrylate monomers into carboxylic acid and alcohol molecules [9]. This reaction speeds up degradation and promotes the release of monomers.

Another possible cause of the variation of WS and S is the curing procedure of EFCs. Considering the sample's large (10 mm) diameter in relation to the light curing system's head diameter, possibly, several of the EFCs had partially cured. Rather than white sample layers, the presence of light yellowish layers may suggest partially cured parts. The consistent hue produced by RF2 signifies that the components had reached full cure. The difference in the nanohybrid silica refractive index and that of the monomers resulted in an incomplete polymerization process (conversion degree) because of visible light scattering, absorption, and refraction, which is another possible explanation for this variation between test groups [27-29].

In our study, it was found that the WS and S of EFC CQ 0.05 were higher compared to EFC CQ 0.03 and EFC CQ 0.075. This is most probably due to the use of old silica in the preparation of samples of EFC CQ 0.05 due to insufficient material available in the laboratory. A properly kept old silica still has the possibilities of getting exposed to aging environment, such as humidity. Ageing is defined as the irreversible changes over time in the original properties that are inevitable and limits the operating life of the restoration, despite sophisticated design methods and great care in production [25]. Aged silica may contain water molecules, which can be absorbed into the resin matrix during the sample preparation. As time goes by, the mechanical qualities gradually deteriorate over time due to component leaching, swelling, deterioration of the cross-linked matrix in the dental composite, and hydrolysis of the filler-matrix interfaces [26,30]. As a result, the produced EFCs will have a greater permeability, hence a greater water sorption and solubility properties.

A few limitations were found in this study. First, hand mixing method during the preparation of EFCs may affect the filler distribution and lower the characteristics of the EFCs. Besides, a small diameter head light cure device was used to light cure the samples, which had a diameter of 10 mm. This constraint resulted in some disturbance in the polymerization and degree of conversion of the EFCs. Furthermore, the usage of old silica due to insufficient material also affected the final EFCs.

A few recommendations can be done to improve the results of this study. First, instead of hand mixing, advanced equipment and method can be used, such as the speed mixer. A light cure device with a greater tip size can be used instead. A same batch of fresh silica fillers should be used in this study to ensure an equal quality of the fabricated EFCs.

CONCLUSION

The WS and S of experimental flowable nanocomposites were significantly higher than those of the commercial flowable composite. Most of EFCs' findings did not meet the minimum ISO standards specifications. Overall, it is evident that while synthetic nano-silica was used to successfully create experimental flowable nanocomposites, their qualities still fall short of those of commercial flowable composites. EFCs have the potential to be a sustainable product that can benefit the environment and further research is needed to overcome the suboptimal results.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

ACKNOWLEDGEMENT

Special thanks for the financial assistance to Universiti Sains Malaysia, Short-Term Grant with Project No: 304/PPSG/6315680.

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