



EFFECTS OF STIRRING SPEED AND TIME ON WATER ABSORPTION PERFORMANCE OF SILICA AEROGEL/EPOXY NANOCOMPOSITE

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ABSTRACT

Aerogel reinforced epoxy nanocomposite are known as efficient materials in a broad range of high performance application in many industries including aviation and marine industries. This paper investigated on the effect of stirring speed and time, as separate factors on water absorption performance silica aerogel/epoxy nanocomposite. Silica aerogel/epoxy nanocomposites were prepared variously by mixing silica powder, epoxy resin and hardener at a fixed ratio. The uncured solutions of aerogel/epoxy were then subjected to different combination of parameters of stirring speeds and stirring times. Resulted weight gained from water absorption analysis of the cured aerogel/epoxy nanocomposite was recorded at various time intervals up to 99 days. Presence of trapped air bubbles during fabrication of silica aerogel/epoxy nanocomposite was examined morphologically under Scanning Electron Microscope (SEM). Fickian diffusion analysis was used to predict water uptake by the epoxy nanocomposites. Results showed that blank epoxy stirred at higher speed and lower time exhibit the lowest rate of water absorption with only 7.30%. As for the epoxy filled nanocomposite samples, they obtained higher rate of water absorption approximately more than 9.10% compared to blank epoxy. For mix 1 (silica and epoxy resin), lower stirring speed and higher stirring time provided good result. For mix 2 (mix 1 and hardener), higher stirring speed and lower stirring time provided good result. It can be indicated that water absorption rate were influenced by stirring speed, time and adding of filler.

Keywords: water absorption, stirring speed, stirring time, trapped air bubbles, silica aerogel.

INTRODUCTION

Epoxyes are the most studied thermoset materials with a very wide range of industrial applications, such as for aerospace, automobile, sporting equipment, surface coatings, printed circuit boards, as well as anti-scratch and anti-corrosive materials with high adhesive strength, high strength and hardness, outstanding chemical and heat resistance [1, 2]. Such a broad range of practical applications is obtained by the proper selection of the resin-hardener pair and also by employing the right curing conditions [3].

Silica aerogels are highly porous (microporous) materials, which have large surface area and pore volume. Due to its excellent properties with about 98% porosity [4], comparative large surface area with approximately 1500 m²g⁻¹ and extremely low density, silica aerogels often incorporated as filler in epoxy resins. Inclusion of silica aerogel in polymer system is able to improve the mechanical performances such as flexural modulus, fracture toughness and impact strength, which have been the subject of intensive research in the last decade [5-7]. Polymer materials can absorb water in a wet atmosphere, and the amount absorbed depends on the composition and hydrophobicity of the polymer [8]. There were a few studies on analysis of water absorption on composite materials [8-16]. The mechanism of water absorption is based on climatic conditions [17]. At high level of humidity, water can penetrate into polymeric composites in three ways; primarily via the inter-polymer chain micro-gaps [18], second is via fibre and matrix interfaces through capillary transport tracing any gaps and/or flaws caused by

incomplete wettability and impregnation [19, 20], and third is via micro-cracks in matrix which result from natural composite swelling (particularly in the case of natural fiber composites) [21].

Previous studies had reported that absorption of water in epoxy composites do effect the mechanical strength of the composites [21-23]. It is a challenge to to totally remove the trapped air in the produced composite. The formation of bubbles and air trapped were due to several parameters, such as the stirring speed, stirring time and the reaction time. However, until now there are no systematic studies on the effect of stirring speed and time on the properties of the nanocomposite product. In this study the effect of stirring speed and time, and water absorption performance of silica aerogel/epoxy nanocomposites was investigated and discussed.

METHODOLOGY

In this experiment, EpoxAmite® 100 Resin and 102 Medium Hardener, manufactured by Smooth-On, Inc, were used as resin and curing agent respectively. Resin to hardener weight ratio is 100: 29 as recommended by the manufacturer. Silica aerogel which is derived from rice husk ash was used as filler and was manufactured by Maero Tech Sdn Bhd (MTSB). Silica aerogel has an impressive dendritic microstructure with high specific surface area of approximately 900 m²/g and average nanoscale size of 20-50 nm which is fused into clusters. These clusters result in very strong structure [24].



Preparation of blank epoxy

Blank epoxy samples were prepared as control to provide baseline data on properties of blank epoxy. Epoxy resin was mixed with hardener. Three samples were prepared, namely samples A, B and C with 25 gram each. The mixture was stirred using mechanical overhead stirrer at different speed and stirring time (see Table-1). The mixture was degasified by placing it in a vacuum oven (at room temperature for 30-45 minutes) to remove the air trapped. The mixture was poured in square mold and left for over 24 hours at room temperature to cure.

Table-1. Blank epoxy samples with different stirring speed and time.

Sample	Stirring speed (rpm)	Stirring time (min)
A	300	2
B	150	7
C	55	3

Constant weight of 0.5 wt% of silica aerogel was added in epoxy resin for every different nanocomposite samples. The mixture was stirred using mechanical stirrer at different speed and stirring time (see Table-2). The prepared mixture was then placed in a vacuum oven for 30-45 minutes (at room temperature) for complete degasification of uncured nanocomposite samples. Formation of air bubbles can be either from the resin mixture or during the mixing process [25]. It is believed that the air bubbles appeared during stirring due to reaction between silica powder (filler) and epoxy in open air condition. The degasification process was conducted to obtain good quality and improve microstructural properties of the resulting composite [26, 27]. After complete degasification, hardener was mixed with the silica aerogel/epoxy solution mixture and stirred at varying time for 0.5, 1 and 2 minutes. The prepared mixture was again placed in the vacuum for complete degasification. Following this, the final solution was cast and cured at room temperature for 24 hours. Table-2 shows six samples of silica/epoxy nanocomposite with different stirring speed and time.

Preparation of silica/epoxy nanocomposites

Table-2. Silica/epoxy nanocomposite with different stirring speed and time.

Sample	Mix 1 (Silica + Epoxy)		Mix 2 (Mix 1 + Hardener)	
	Stirring speed (rpm)	Stirring time (min)	Stirring speed (rpm)	Stirring time (min)
D	500	5	150	1
E	300	8	150	0.5
F	700	5	150	1
G	1000	3	150	1
H	700	5	55	2
I	1000	3	55	2

Water absorption test

The water absorption capacity of blank epoxy and silica aerogel/epoxy nanocomposite when immersed were determined in accordance with ASTM D570-98. A total of 10 sets of specimens including six sets of silica aerogel/epoxy nanocomposites and four sets of blank epoxy, were subjected to the test. The nanocomposite samples were prepared to approximately of 57 mm x 10 mm x 3 mm dimension. Readings on mass for every sample were taken after 30 minutes, 1 hour, 2 hours, 4 hours, 8 hours, 16 hours and 24 hours.

The samples were weighed and immersed in distilled water with controlled temperature of 30 ± 1 °C. For taking readings, specimens were removed from water and wiped off dry with a dry cotton cloth. Then, weighed the specimen to the nearest 0.001 g immediately and replaced the samples back in water. Readings were taken

at various time intervals and every 14 days thereafter, until weight is constant. To calculate the amount of water absorbed (M_A) by the samples, the Equation (1) was used, where M_D is the initial mass (dry mass) and M_t is the mass of sample after immersed in distilled water for time, t .

$$M_A(\%) = \frac{M_t - M_D}{M_n} \times 100 \quad (1)$$

Optical microscope inspection

In order to examine the trapped air bubbles formed in the composite, an Olympus BX51 microscope was used with magnification of microscope 20 X for all samples.



RESULTS AND DISCUSSION

Fickian diffusion

To describe moisture absorption of materials, Fickian diffusion was considered as suitable models to predict water uptake by epoxy resin [28-32]. The presence of fillers was proven to affect water absorption behavior of the composite [33, 34]. The water absorption curve derived from the experiment can be considered Fickian diffusion only if the curve follows the linear Fickian diffusion curve as shown in Figure-1.

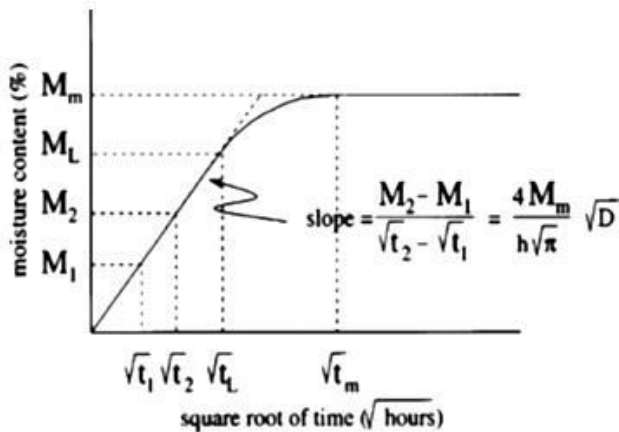


Figure-1. Illustration on the change of moisture content with the square root of time for Fickian diffusion. For $t < t_L$ the slope is constant [28].

Additionally, maximum content and mass diffusivity can be calculated from the graph using Equation (2) below where M_t is the amount of water absorbed at time, t . D is the diffusion coefficient, M_m is the maximum water uptake, and l is material thickness [35].

$$\frac{M_t}{M_m} = 1 - \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp \left[\frac{-(2n+1)^2 \pi^2 D t}{4l^2} \right] \quad (2)$$

Water absorption

Water absorption test was conducted with three replicates for each sample. An average value of the three replicates was taken to represent the test results.

Figure-2 shows water absorption behavior of the blank epoxy and silica aerogel/epoxy nanocomposite for 3 months. It can be seen that water absorption for all samples increase with immersion time. The curve showed that water absorption followed the typical Fickian diffusion behavior. According to the classical diffusion theories [36], the curve can be described as pseudo-Fickian at a shorter time period where the initial rate of absorption is higher for a shorter time and continue to slow down until reaching an equilibrium state. At this state, it was proved that total percent of water uptake is approximately the same whether immersion is conducted at room or higher temperature [37].

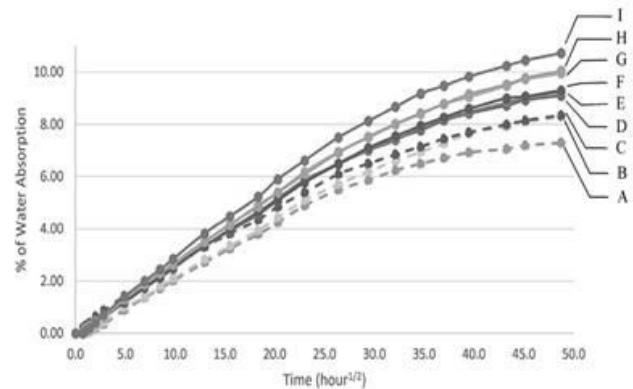


Figure-2. Water absorption behavior of blank epoxy (A, B, C) and silica/epoxy nanocomposite (D, E, F, G, H, I).

According to Crank [38], the difference is only in the time taken to reach the equilibrium. At initial stage, an increase in penetration of water will caused the absorption coefficient to increase instantly. When approaching equilibrium, the absorption coefficient will slow down due to relaxation process characteristic of the glassy state.

Figure-3 shows the percentage decrement of water absorption for each sample. All samples were found to absorb high amount of water up to 14 days. This is due to the strong cohesive force between water molecules in which the water molecules continuously and rapidly diffuse into trapped air bubbles. Later, amount of water absorbed by samples was found gradually decreased over a period of 85 days starting from day 22 for about 1.0 -1.4 percent and continuously reduce to approximately 0.2 percent in day 78 till day 99. When the composite weight have become constant, it indicated that the composite have saturated with water as less interaction sites of composite left with water molecules. This stage is called quasi-equilibrium. Prolong the immersion time of sample in water will only allow water molecules to fill in the trapped air bubbles or microporosity, thus forming a clusters. Finally, real equilibrium may be achieved when all trapped air bubbles or microporosity are filled [37].

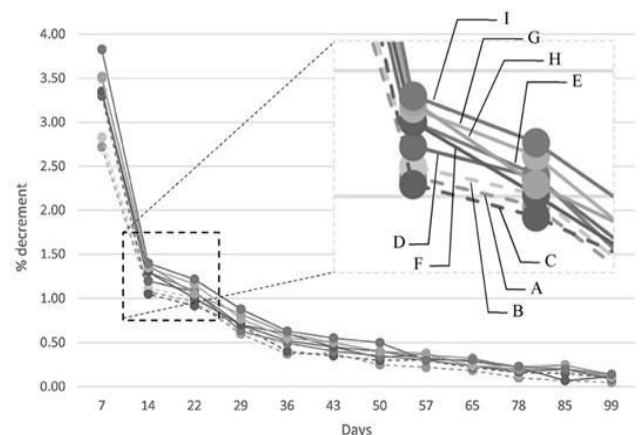


Figure-3. Percentage decrement of water absorption by samples.

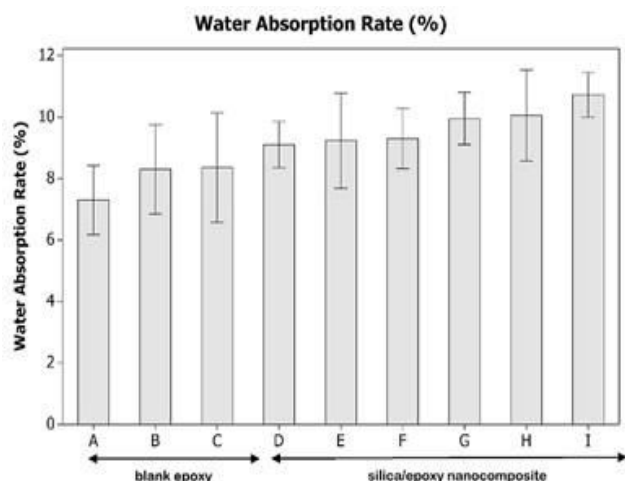


Figure-4. Water absorption rate on blank epoxy and silica/epoxy nanocomposites.

Figure-4 shows the total water absorption rate by all samples. Overall, silica aerogel/epoxy nanocomposites shows higher absorption rate compared to blank epoxy samples. With addition of silica aerogel, high absorption of water is mostly associated with formation of H-bonded with hydroxyl groups besides formation of air bubbles. The composite became hydrophilic (extremely hygroscopic) when the silica surface was totally hydroxylase and therefore able to absorb water, with 20 percent increase in mass [39, 40]. Since silica is synthetic and generally amorphous, water desorption can also be greatly influenced by the number, size and morphology of the silica pores [41].

All samples exhibit direct-fast diffusion of water molecules through structural imperfections in the composite such as presents of voids or trapped air. For blank epoxy sample, sample A showed the lowest absorption rate with 7.3% followed by sample B and C

(Figure-4). Based on Figure-5, the contour plot shows that high stirring speed with low stirring time provided good results in reducing the moisture absorption from the composite.

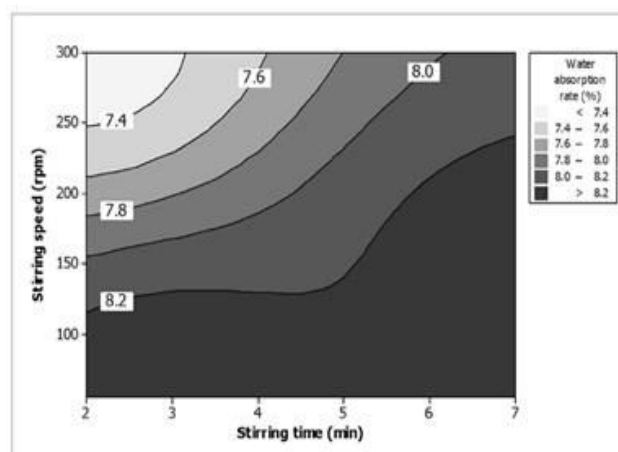


Figure-5. Contour plot of stirring speed vs. stirring time for blank epoxy.

In order to obtain homogeneous resin with less bubble formation, shortening the stirring time and increase the stirring speed provide better result. Formation of air bubbles during stirring process were able to be remove before the epoxy resin started to cure by degassing in vacuum oven. Not all bubbles can easily move upward following the air suction by the vacuum. Some of the bubbles failed to move upward due to the increase of epoxy viscosity with formation of gel on the epoxy surface. At this stage, the epoxy resin starts to cure and small bubbles trapped in the epoxy resin after sample totally cured.

Table-3. Effect of stirring speed and time on water absorption rate for silica/epoxy nanocomposite.

Water Absorption rate (%)	Sample	Mix 1 (Silica + Epoxy)		Mix 2 (Mix 1 + Hardener)		% difference with sample D
		Stirring speed (rpm)	Stirring time (min)	Stirring speed (rpm)	Stirring time (min)	
9.10	D	500	5	150	1	-
9.23	E	300	8	150	0.5	1.4
9.30	F	700	5	150	1	2.2
9.95	G	1000	3	150	1	9.3
10.05	H	700	5	55	2	10.4
10.73	I	1000	3	55	2	17.9

Figure-6 shows the effect of stirring speed and time on rate of water absorption for silica/epoxy nanocomposites. For silica/epoxy nanocomposites, the relationship between stirring time and stirring speed were investigated for mix 1 and 2 separately. For mix 1, sample

D, F and G were chosen for analysis. Stirring speed and time for mix 2 were fixed at 150 rpm and 1 minute, respectively. Result from Figure-6 shows that low stirring speed with increase in time, resulted in low water absorption rate. According to Aravand *et al.*, effect of



stirring speed is inversely proportional to the viscosity of the mixture. Therefore, increase the stirring speed can produce low mixture of viscosity and vice versa [42]. Liquid with low viscosity has high surface tension. In addition, surface tension is proportional with the formation of bubble size. Hence, increasing the stirring speed will not only result in producing high surface tension, but also producing big and wide distribution of the bubbles [43, 44].

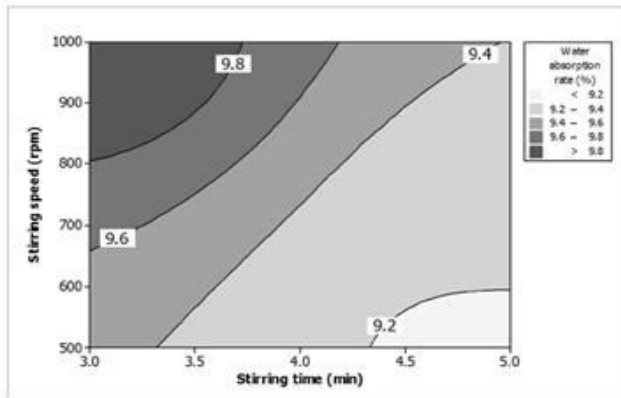


Figure-6. Contour plot of stirring speed vs. stirring time for sample D, F and G.

Interestingly, although sample E produced with the lowest stirring speed, the sample was found to exhibit higher water absorption rate compared to sample D. This can be due to the lack of silica dispersion into the epoxy matrix. Increasing the stirring speed will subsequently increase the turbulence in the fluid and result in high volume of void fraction. This situation leads to an increase the drag of the bubbles and caused more bubbles holdup [45, 46]. By prolong the mixing process between silica and epoxy resin, more bubbles were formed. It was found that optimum stirring speed and processing time in this work were 1, 500 rpm and 5 minutes, respectively. As the stirring time increase more than 8 minutes, the formation small bubbles increase. Small bubbles have higher internal pressure, thus large size of nucleation bubbles will be formed by diffusion of gas through the liquid matrix [47].

For mix 2, sample F and H (Figure-7a) and sample G and I (Figure-7b) were chosen for analysis. Result shows that increasing the stirring speed and reducing the stirring time gave lower water absorption rate. This result is similar with blank epoxy due to the rapid curing process after adding the hardener to the solution mixture of epoxy and silica aerogel.

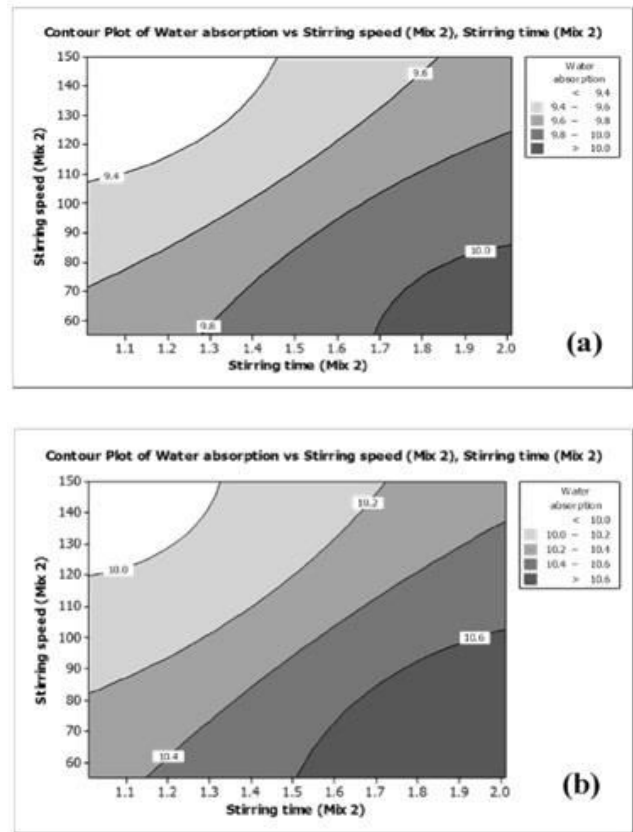


Figure-7. Contour plot of stirring speed vs. stirring time for sample (a) F and H, and (b) G and I.

Trapped air bubble content

Optical microscope was used to investigate presence of trapped air bubble content in the nanocomposite samples. During degasification process, some of the bubbles which produced during processing of the nanocomposite were sucked out. The remaining bubbles which is fail to remove after the resin start to cross link were trapped and formed small hole or known as trapped air bubble. Presence of trapped air bubble are likely to be related with absorption of water by the material. High content of trapped air bubbles indicate high amount of water being absorbed into the composite and vice versa. Water which is present in the trapped air bubbles was observed. The microscopic images are shown in Figure-8 (for blank epoxy) and Figure-9 (for silica aerogel/epoxy nanocomposite). Trapped air bubbles in each sample are randomly selected and measured as shown in Table-3 and Table-5.

Table-4. Size of trapped air bubble for blank epoxy.

Trapped air bubble	Size of trapped air bubble (μm)		
	Sample A	Sample B	Sample C
A	380.58	3488.55	4837.35
B	447.21	2830.19	2780.29
C	-	3860.05	4248.53
D	-	2102.38	2617.25
E	-	5325.41	-

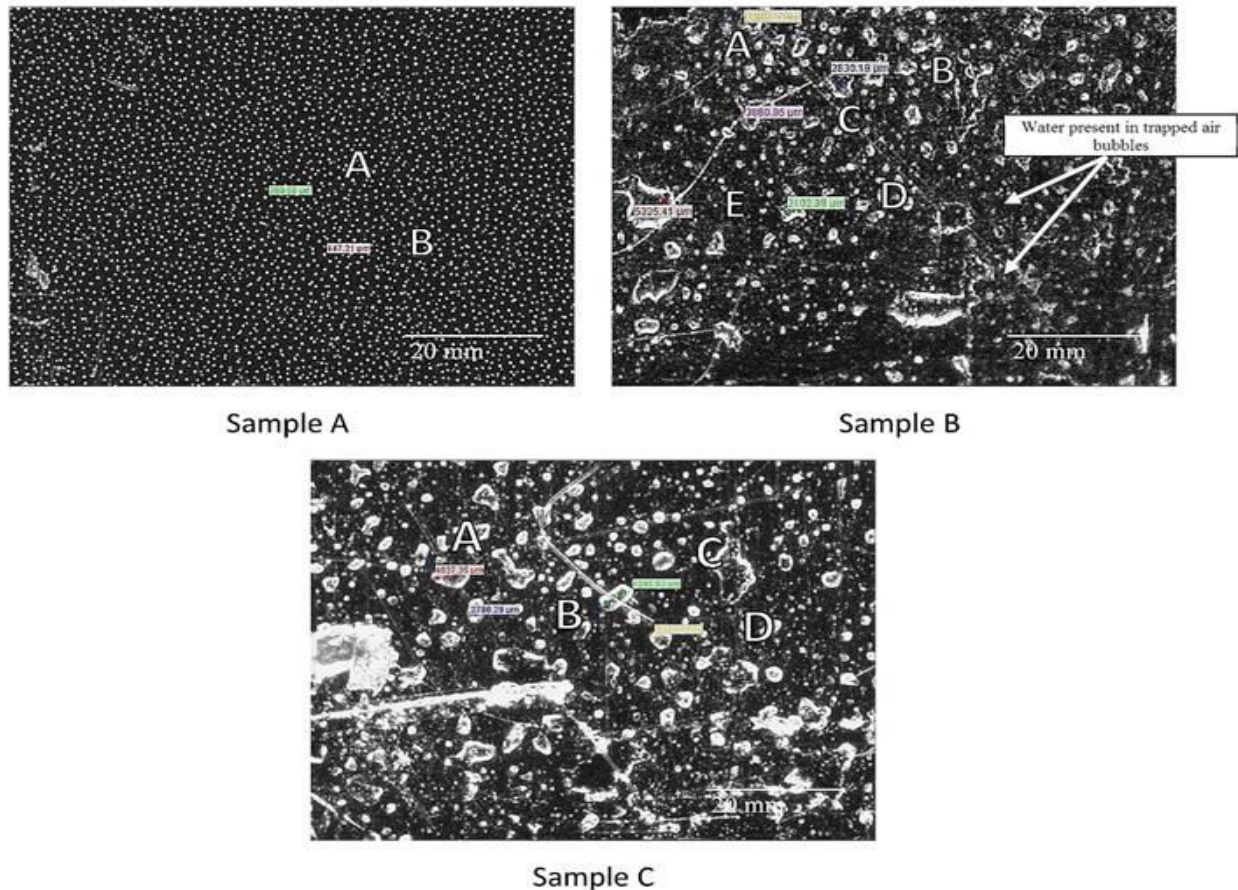


Figure-8. Microscopic images for blank epoxy.

Figure-8 reveals that trapped air bubbles content in sample A is approximately nine times smaller compared to those in sample B and C. The trapped air bubbles were scattered uniformly across the sample. Sample A was found to absorb the least amount volume of water due to small size of trapped air bubbles. However, sample B and C produced bigger size of trapped air bubbles compared to sample A. The size of trapped air bubbles for both samples were nearly similar thus producing similar water absorption results.

Formation of trapped air bubbles in silica aerogel/epoxy nanocomposite for sample D (Figure-9) were small and the trapped air scattered compared sample B and C. Although sample B and C (Figure-8) had more trapped air bubbles compared to sample D, it was found that sample D has high water absorption rate due to the presence of silica in the resin system. All silica aerogel/epoxy nanocomposites exhibit higher rate of water absorption compared to blank epoxy as evident in the results shown in Figure-3.

Table-5. Size of trapped air bubble for silica/epoxy nanocomposites.

Trapped air bubble	Size of trapped air bubble (μm)					
	Sample D	Sample E	Sample F	Sample G	Sample H	Sample I
A	2807.13	4134.01	1769.18	2549.51	5142.96	8982.76
B	2915.48	3847.08	2780.29	1726.27	5819.08	6735.73
C	3008.32	3342.15	4070.63	6684.31	4924.43	4876.47
D	2607.68	3220.25	2302.17	2816.83	4534.31	6003.33
E	3101.81	3201.56	2418.68	2262.74	3512.83	6074.54
F	-	3323.42	2692.58	-	16497.58	-

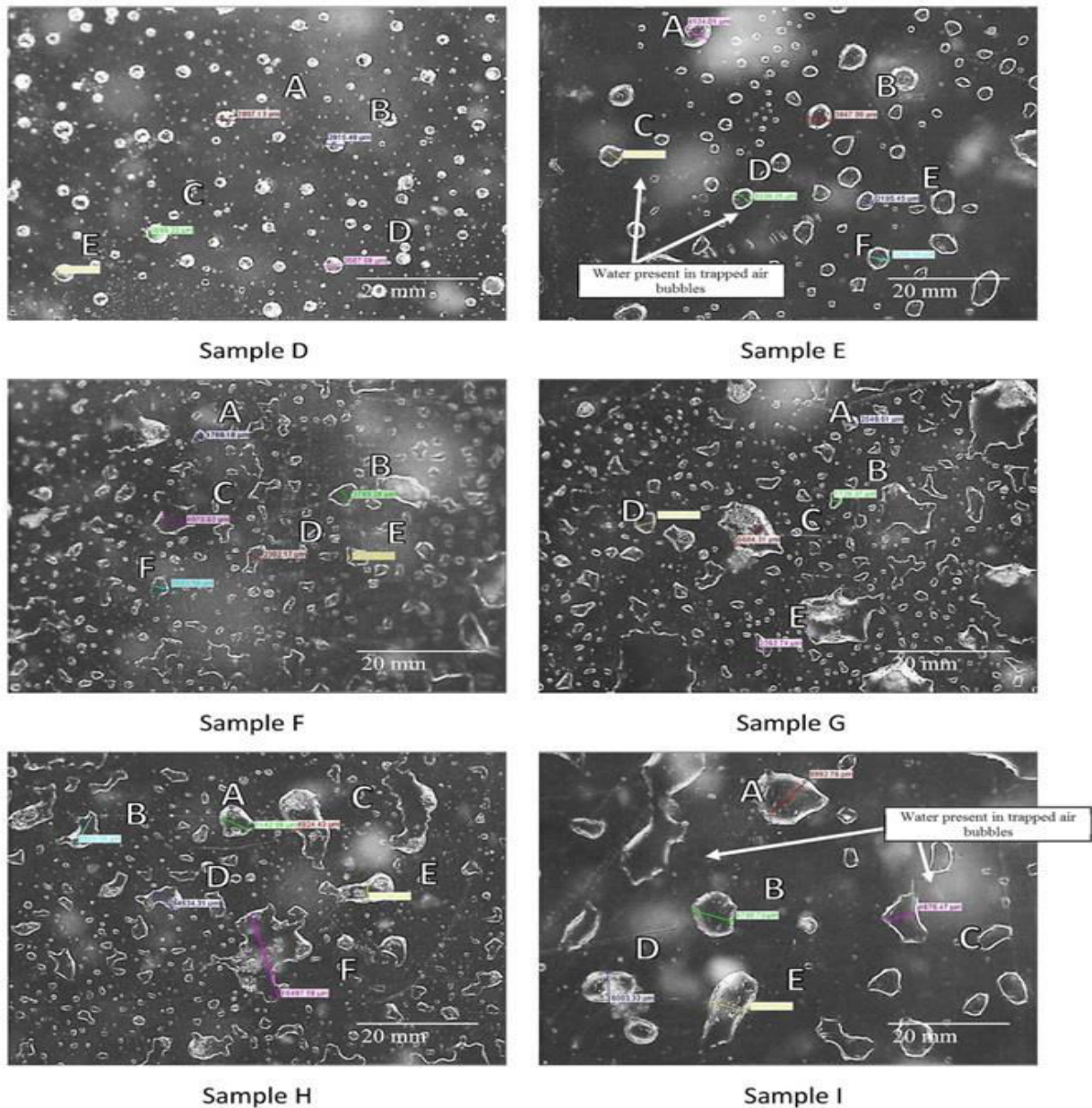


Figure-9. Microscopic images for silica/epoxy nanocomposites.

Other than that, it was observed that sample E and F showed approximately similar trapped air bubbles size. However, sample F exhibited slightly higher rate of water absorption probably due to higher trapped air bubble density with more uneven distribution trapped air bubble. Sample G and H shows larger trapped air bubble sizes compared to sample E and F, in which larger size of trapped air bubbles surrounded with the tiny bubbles. The size and frequency of trapped air bubbles in sample G and H are almost similar, as reflected in a mere of 0.1 % difference in water absorption between those two samples. Sample I which exhibited the highest rate of water absorption rate which is 10.73% (Figure-4) and it comprised with mainly large trapped air bubbles.

CONCLUSIONS

It was observed that from the water absorption curves for blank epoxy and silica aerogel/epoxy nanocomposites, both type of samples followed the typical Fickian Diffusion behaviors in which the trend increased linearly at the beginning before reaching equilibrium state. For processing blank epoxy, increasing the stirring speed with shorter stirring time gave better results. As for silica aerogel/epoxy nanocomposites, good results were obtained by reducing the stirring speed and prolong the stirring time for mix 1 (when mixing silica and epoxy resin) and followed by increasing the stirring speed and shortening the stirring time for mix 2 (when mixing hardener with mix 1). Results shows that water absorption rate were primarily influenced by stirring speed and time and together with incorporation of filler.



The trapped air bubble sizes obtained using optical microscope supported the results on water absorption rate. For blank epoxy, trapped air bubble size of sample with lowest amount of water absorbed is nine times smaller and scattered uniformly compared to the sample with highest amount of water absorbed. The same results were obtained for silica aerogel/epoxy nanocomposite, in which sample with lowest absorption rate contained small volume of trapped air bubbles that were evenly and closely scattered. Large trapped air bubbles mainly can be seen in sample with the highest absorption rate.

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