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# The Effect of Methyltriethoxysilane (MTES) Concentration on Hydrophobic Properties of Silica Thin Layer

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**Abstract.** One of the hydrophobic materials is silica thin layer in which a hydrophobic silica precursor is superimposed on the material surface that makes it hydrophobic. Methyltriethoxysilane (MTES) is a favorable hydrophobic silica precursor because it has a methyl group that functions as a hydrophobicity guiding agent. In this study, the synthesis of thin layer and hydrophobic silica xerogel from MTES using the sol-gel method with ethanol-ammonium hydroxide solvent was carried out. The MTES concentration was varied to 1.42, 2.85, 5.70, 11.40, and 22.80 M to assess the effect of MTES concentration on the hydrophobic nature of the silica thin layer. The MTES solution was coated on the glass surface with a dip-coating method to measure the contact angle then, xerogel preparation was carried out by drying the MTES solution at room temperature. It continued by drying in an oven at 60 °C for 2 hours then calcined at 300 °C. The xerogel generated were analyzed using FTIR. It was found that the variation of MTES concentration had a considerable effect on the hydrophobic nature of the silica thin layer. Increased MTES concentration, from a concentration of 1.425 M to 11.4 M, escalated the contact angle of silica thin layers. The highest contact angle was achieved at MTES concentration of 11.4 M and calcination temperature of 300 °C, which reached 82.53°. Nevertheless, the thin layer produced was still hydrophilic, which was characterized by the contact angles of less than 90°, and the Si-OH/Si-O-Si ratio increased.

## INTRODUCTION

Glass is a transparent material that has been widely used in everyday life, one of which is as window glass. Glass in windows of tall buildings requires special cleaning from dust that sticks to the surface, and this requires a high cost. For this reason, efforts are needed to develop glass coating materials that have waterproof properties. Hence the water droplets do not attack the glass surface and carry pollutants. In nature, plants and animals are discovered that the water does not stick to their surface, where one of them is a lotus leaf. Water that falls on the lotus leaf does not stick and is easy to roll and carry specks of dirt on the lotus surface, which is known as the self-cleaning mechanism and is often referred to as the lotus effect [1].

A hydrophobic surface can be obtained by changing micro and/or nano-sized structures on a substrate or changing the surface structure with materials that have low surface energy [2]. The solid surface, which has low surface energy, will reduce wettability so that it will produce a hydrophobic surface [3].

Methyltriethoxysilane (MTES) with the molecular formula  $C_3H_9O_3Si$  has a methyl group ( $-CH_3$ ), which can add to the hydrophobicity of a surface [4]. Tsuru and coworkers [5] conducted a study on the manufacture of nanopore methylated hydrophobic membranes and their application for hexane solution nano-filtration. The methylated  $SiO_2$  solution was obtained by hydrolysis and condensation reaction from a mixture of tetraethoxysilane (TEOS) and methyltriethoxysilane (MTES) using ethanol as a solvent and  $NH_3$  as a catalyst. Organic/inorganic hybrid membranes were prepared by coating a porous alumina substrate with a modified  $SiO_2$  solution and calcined at 400-600 °C in the  $N_2$  atmosphere. The results showed that the membrane was hydrophobic and produced nano-sized

pores of 1.7-4.3 nm. The hydrophobic nature of a membrane can be accessed by adding methyltriethoxysilane (MTES) and tetraethylorthosilicate (TEOS) to the sol solution through the hydrolysis reaction and condensation reaction [6].

Research conducted by Rao, Rao and Pajonk [7] making a thin layer using MTES precursors with differences in molar ratio of sodium silicate: water:  $\text{NH}_4\text{OH}$ : MTES i.e.  $1 : 45 : 4.3 \times 10^{-2} : 5$  showed that in the form of aerogel, MTES was able to produce a density of  $0.164 \text{ g/cm}^3$ , pore volume of  $5.51 \text{ g/cm}^3$ , porosity of 91.45%, thermal conductivity of  $0.17 \text{ Wm}^{-1}\text{K}^{-1}$  and the contact angle that was relatively low reaching only  $65^\circ$ .

Nadargi and Rao [8] made silica thin layers using MTES precursors with a variation of MTES/methanol molar ratios (1: 6.45; 1: 9.65; 1: 16.12; 1: 19.35) catalyzed with both acid and base through the sol-gel method followed by supercritical drying. It was identified that the contact angle of the silica thin layer increased with increasing MTES content with the highest contact angle of  $163^\circ$ . Hence it was concluded that MTES is a pretty good hydrophobic steering agent.

Referring to the research of Nadargi and Rao [8], this research carried out the synthesis of silica layers using MTES as the silica precursor. A study of the effect of MTES concentration (the amount of MTES was varied while the amount of ethanol was made constant) was carried out on the hydrophobicity of the resulting silica thin layer and the correlation for the functional groups was then formulated. The synthesis was carried out using the sol-gel method with variations in the MTES concentration of 1.42, 2.85, 5.70, 11.40, and 22.80 M using ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) as a base catalyst while the solvent used was ethanol. Furthermore, water contact angle analysis and xerogel characterization were performed using FTIR instruments.

## EXPERIMENTAL

### Materials and Equipment

Equipment used included: glassware, oven, furnace Vulcan 3-130, magnetic stirrer, Fourier Transform Infra-Red (FTIR), and DSLR camera. While the materials used were Methyltriethoxysilane (MTES) Sigma-Aldrich, ethanol 99.99% (Merck), ammonium hydroxide 25% (Merck), and glass plates with a thickness of  $\pm 3 \text{ mm}$ .

### Preparation of Methyltriethoxysilane (MTES) Solution

Silica solution preparation refers to the research described elsewhere [9-12]. MTES solution with a concentration variation of 1.42, 2.85, 5.70, 11.40, and 22.80 M were synthesized using 70 mL ethanol-ammonia solvent at constant pH, then stirred using a magnetic stirrer for 2 hours at a speed of 300 rpm. The stirring process was carried out on an ice bath to avoid partial hydrolysis occurred due to temperature.

### Silica Xerogel Preparation

Xerogels were prepared by evaporating the MTES solution at room temperature for one week, followed by drying at  $60^\circ\text{C}$  in an oven to remove the remaining solvent. The dried xerogel was then calcined at  $300^\circ\text{C}$  using a furnace (Vulcan TM 3-1300) for 30 minutes at a ramp rate of  $2^\circ\text{C/minute}$  to be further analyzed by FTIR to analyze their functional group.

### Formation of Silica Thin Layer

The glass plate was dipped in the MTES solution through a dip-coating process until the entire surface was immersed. Slowly removed from the MTES solution and dried at room temperature, then calcined at a temperature of  $300^\circ\text{C}$  for 30 minutes at a ramp rate of  $2^\circ\text{C/minute}$ . These steps were repeated four times to obtain a thicker layer. These processes were also carried out on glass plates for various concentrations of MTES.

## Measurement of Water Contact Angle

The surface of the glass plate that has been coated with a silica thin layer was dripped with drops of water, then taken using a DSLR camera. Contact angle values were calculated using the arc-tangent method based on the height and radius of the resulting water drop.

## RESULTS AND DISCUSSION

### Water Contact Angle

To find out the interaction between water and hydrophobic silica thin film MTES, water contact angle measurements were taken. After water was dripped on the surface of a glass plate that has been coated with an MTES silica thin layer. The image of water drops on the surface of the glass plate is presented in Fig. 1.

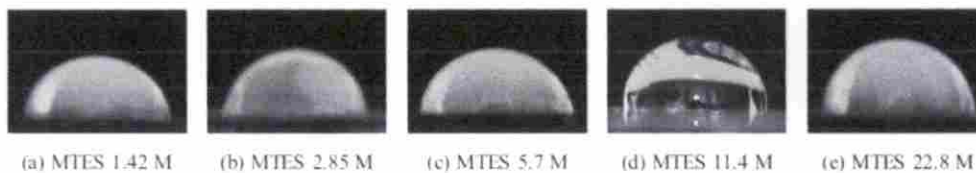


FIGURE 1. Water drops on MTES silica thin layers with varying concentrations of MTES : (a) 1.42 M, (b) 2.85 M, (c) 5.70 M, (d) 11.40 M, and (e) 22.80 M.

Figure 1 shows the contact angle of water dropped on a glass surface that has been coated by an MTES silica thin layer. With increasing MTES concentrations, the water contact angle tends to increase. The highest contact angle was obtained at an MTES concentration of 11.40 M with a contact angle of 82.53°, and from this point, for higher MTES concentrations, the water contact angle decreases.

Qualitatively, this result shows the differences in water contact angles due to differences in MTES concentrations. These results indicate that MTES concentration affects the hydrophobic nature of the silica thin layer produced. Quantitatively, the relationship between MTES concentration and water contact angle can be seen in Fig. 2.

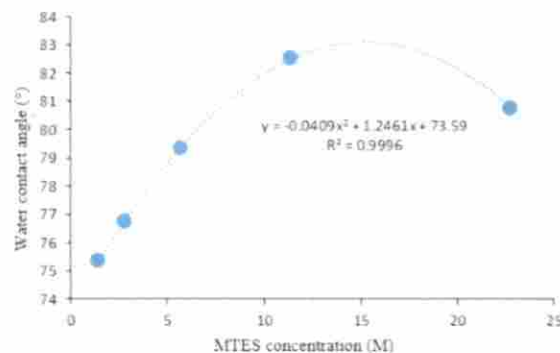


FIGURE 2. Relationship between MTES concentration and water contact angle dropped on a glass plate

Figure 2 shows a polynomial curve of order 2 (quadratic function) in the form of a parabola that has a peak point at a concentration of MTES 11.40 M. From the relationship of water contact angle with MTES concentration, a parabolic graph is obtained with the equation  $y = -0.0409x^2 + 1.2461x + 73.59$  with a very good regression coefficient value of 0.9996. According to Sarwono [13], the higher the value of  $R^2$  ( $R^2 \sim 1$ ), the alignment of the



regression model closer to the original data. This result indicates that there is an excellent correlation between MTES concentrations and the magnitude of the water contact angle.

Increased water contact angle indicates an increase in the hydrophobic nature of the thin film. The increase of water contact angle because of the increasing MTES concentration is predicted due to the increasing number of non-polar groups ( $\text{Si-CH}_3$ ) in MTES, which is a hydrophobic group [14]. The more non-polar groups, it increases the hydrophobic nature of a surface.

In silica polymers, there are silanol ( $\text{Si-OH}$ ) and siloxane ( $\text{Si-O-Si}$ ) groups. According to Rao, Rao and Pajonk [7] the  $\text{Si-OH}$  group is hydrophilic, while the  $\text{Si-O-Si}$  group tends to be hydrophobic. It is predicted that with increasing MTES concentration, the presence of the  $\text{Si-CH}_3$  group competes with the  $\text{Si-OH}$  group in the tip silica group, which eventually decreases the hydrophilic nature and improve the hydrophobic nature of the silica polymer. Therefore, increasing MTES composition upsurgs the hydrophobic properties of silica thin layers.

However, at higher MTES concentrations (22.8 M), the water contact angle was lower than of 11.4, this is counter-intuitive with the understanding that the contact angle will continue to increase with increasing MTES concentration. This case might be similar to the case raised by Sohn, Kim, Hong, Sohn and Lee [15] that found a similar case when synthesizing hydrophobic coatings using poly (oxyethylene)  $(-\text{OCH}(\text{alkyl}), \text{CH}_2)_n-$  polymers with alkyl thioether ( $\text{CH}_3\text{-nTE}$ ;  $n$  = carbon atoms in the side chain). Polymers synthesized with  $\text{CH}_3\text{-12TE}$  and  $\text{CH}_3\text{-14TE}$  have lower contact angles compared to  $\text{CH}_3\text{-8TE}$  and  $\text{CH}_3\text{-10TE}$  despite having more carbon atoms. The surface shape of the polymers influences this in  $\text{CH}_3\text{-12TE}$  and  $\text{CH}_3\text{-14TE}$ , which is flatter compared to polymers made from  $\text{CH}_3\text{-8TE}$  and  $\text{CH}_3\text{-10TE}$ . According to Wenzel [16], on rough surfaces, water droplets maintain contact at all points below. Sohn, Kim, Hong, Sohn and Lee [15] argued that the  $\text{CH}_3\text{-8TE}$  and  $\text{CH}_3\text{-10TE}$  polymers were deformed on the surface; hence they become jagged (rough) so that the contact angle was greater whereas in the  $\text{CH}_3\text{-12TE}$  and  $\text{CH}_3\text{-14TE}$  polymers the surface deformation disappears and the surface becomes smoother, causing smaller contact angles.

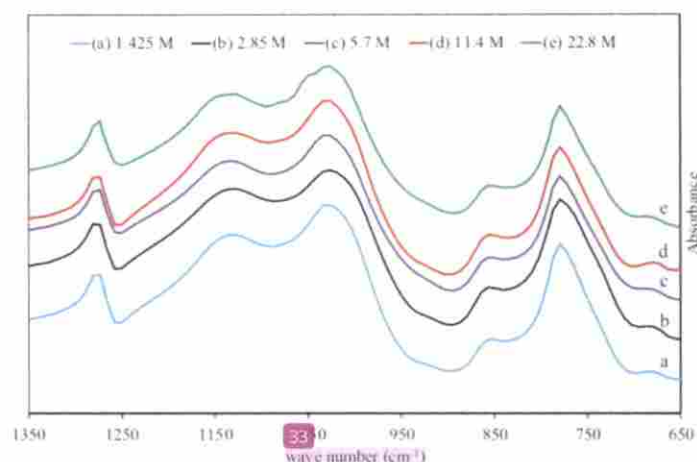
It was estimated that the surface roughness of the silica thin layer increased from an MTES concentration of 1.42 M to 11.40 M; therefore, with increasing MTES concentration, the magnitude of the contact angle became greater. However, after an MTES concentration of 11.40 M, the  $\text{Si-CH}_3$  group became more congested, which caused the  $\text{-CH}_3$  group to become denser and produce a smoother surface, the implication of which eventually resulted in a smaller contact angle.

According to Nakajima et al. [17], the coarser of the surface morphology, the hydrophobic nature of the surface increases. The surface roughness of the glass-coated by an MTES silica thin layer may be caused by  $\text{Si-CH}_3$  groups that are spread on the surface of the thin film. However, at specific concentrations, the amount of  $\text{Si-CH}_3$  becomes increasingly crowded and denser to produce a more delicate structure. According to Bhushan et al. [18], the hydrophobic nature of a surface is influenced by surface roughness factors where the surface roughness decreases (the narrower the contact area) and the magnitude of the contact angle increases.

### Fourier-Transform Infrared Spectroscopy

Bond types and functional groups contained in the MTES xerogel were identified as through by FTIR analysis. The FTIR spectra are presented in Fig. 3.

Figure 3 shows the presence of FTIR spectra in the region of wave numbers between  $1350\text{ cm}^{-1}$  -  $650\text{ cm}^{-1}$ . The peak of the wave number around  $779\text{ cm}^{-1}$  indicates that the symmetric stretching vibration of  $\text{Si-O-Si}$  [14]. The peak of the wave number around  $880\text{-}840\text{ cm}^{-1}$  indicates the existence of stretching vibration of the  $\text{Si-C}$  group. The peak of the wave number around  $955\text{-}835\text{ cm}^{-1}$  shows the vibrations of  $\text{Si-OH}$  stretching. The peak at the wave number  $\sim 1000\text{-}1200\text{ cm}^{-1}$  shows the existence of asymmetric stretching vibrations of the  $\text{Si-O-Si}$  group. Moreover, the peak at the wave number  $\sim 1280\text{-}1250\text{ cm}^{-1}$  is the vibration of the bending  $\text{CH}$  group [7, 19]. At wave number of  $955\text{-}835\text{ cm}^{-1}$ , the peak is not visible because the peak is in the form of a weak absorption that overlaps with strong absorption, which shows a stretching vibration of the  $\text{Si-OH}$ .



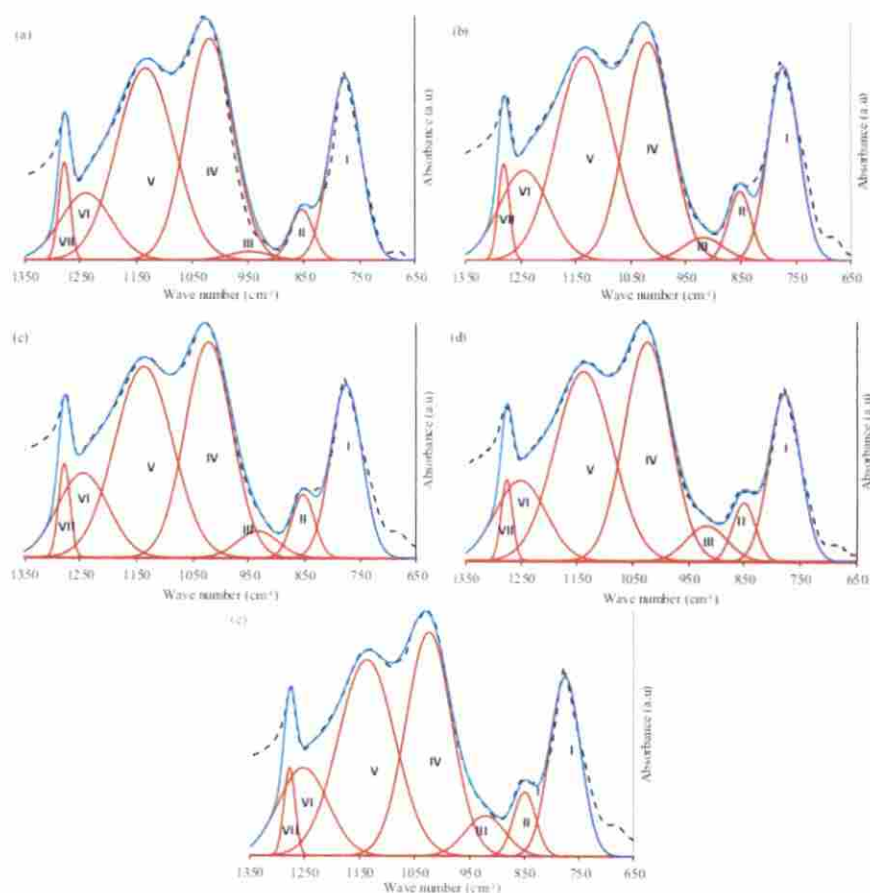
**FIGURE 3.** FTIR spectra of MTES silica xerogels with varying concentrations (a) 1.42 M, (b) 2.85 M, (c) 5.70 M; (d) 11.40 M (e) 22.80 M

Qualitatively, FTIR spectra can find out the absorption band of Si-O-Si, Si-C groups, and Si-OH groups in silica xerogels. However, the five FTIR spectra did not show apparent differences and were similar to each other. To find out the differences in the five spectra, it is necessary to deconvolute each FTIR spectra into their derivative spectra. From this, the difference between each spectrum can be seen. Deconvolution of FTIR spectra was carried out using Fityk software in which spectra in the wavelength region of 650  $\text{cm}^{-1}$  to 1300  $\text{cm}^{-1}$  were reduced to seven constituent spectra using the Gaussian approach. Derivation curves were obtained by equalizing the value of Half width at half maximum (HWHM) for each peak of the derivative by referring to the peak of one of the five spectra. In this case, the group sought was Si-OH, Si-O-Si, and Si-C groups in silica structures. After the peak of the derivative was obtained, then the area of the Si-OH group to the Si-O-Si group was compared; hence, the ratio of Si-OH/Si-O-Si obtained. Furthermore, the area of the Si-C group to the Si-O-Si group was compared to obtain the Si-C/Si-O-Si ratio. Deconvolution spectra in MTES silica xerogel samples with a concentration variation of 1.42, 2.85, 5.70, 11.40, and 22.80 M are presented in Fig. 4.

As a wave function, a peak can originate from one wave or resonance (both constructive and destructive) of various waves. From this, it can be assumed that a peak in the FTIR is composed of derivative peaks where each vertex of the derivative represents a functional group. Thus, the ratio of the area of derivative peak area can be used to determine the quantitative comparison of functional groups.

Figure 4 shows the seven components of the derivative peak, which results from deconvolution from the main peak. From this, it is found that the peak component (I) is an asymmetrical stretch of the Si-O-Si group at wave number  $\sim 779 \text{ cm}^{-1}$  [14]. The peak component (II) in the wave number  $\sim 850 \text{ cm}^{-1}$  shows the Si-C group [20, 21]. The peak component (III) at wave number  $\sim 950 \text{ cm}^{-1}$  is a stretching vibration of the Si-OH group (called silanol), which is the primary source of hydrophilic character [22]. The peak components of (IV) and (V) in wave numbers around 1000-1200  $\text{cm}^{-1}$  are derived from the asymmetric stretching of Si-O-Si groups, which are siloxane groups [23,24,25]. The presence of peaks in these wave numbers shows that in the synthesis process, there is a silylation reaction [26]. The peak component (VI) is the stretching vibration of the Si-O group, while the peak component (VII) represents the C-H group [19].

From the deconvolution of FTIR spectra, the ratio of the area of the Si-OH/Si-O-Si and Si-C/Si-O-Si groups was determined. The choice of the ratio of Si-OH/Si-O-Si and Si-C/Si-O-Si functional groups is since Si-OH groups contribute to hydrophilic properties while Si-C groups contribute to hydrophobic groups. In contrast, Si-O-Si groups are the main group on the silica framework. Thus, the functional group ratio of Si-OH/Si-O-Si and Si-C/Si-O-Si can be used to make a quantitative approach to the contribution of hydrophilic and hydrophobic groups to the hydrophobicity of silica surfaces. The relationship between MTES concentration and the Si-OH/Si-O-Si ratio and the Si-C/Si-O-Si ratio is presented in Fig. 5.

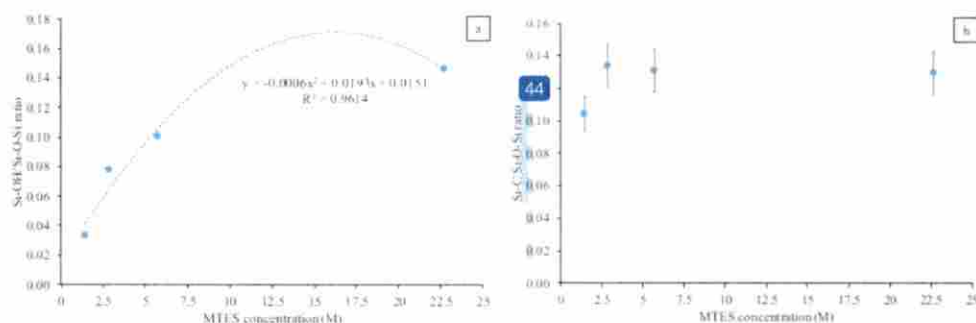


**FIGURE 4.** Deconvolution of FTIR spectra from MTES silica xerogels with variations in MTES concentration of (a) 1.425 M; (b) 2.85 M (c) 5.7 M (d) 11.4 M and (e) 22.8 M

Figure 5a shows that an increase in the ratio of Si-OH/Si-O-Si with increasing MTES concentrations. The increase in the Si-OH ratio is in line with the increase in the contact angle value in the thin layer. This result is averse to the initial hypothesis, which estimates that the increase in contact angle is inversely proportional to the Si-OH/Si-O-Si ratio. This is predicted to occur because the side of the Si-OH group in the silica framework binds to the glass surface, while the  $-CH_3$  group from MTES is pointing up, resulting in a hydrophobic surface. This case might be the same as what happened in the study of Royne et al.[27], where repulsive forces act between the surfaces of calcite in the presence of water, which is caused by water adsorption. It was discovered that there was a strong repulsive force between the surfaces of calcite mixed with water, while there was a tug of attraction (the surfaces becameched) with increasing concentrations of ethylene glycol.

The increase in the Si-OH/Si-O-Si ratio may be caused by an overly high calcination temperature (300 °C). According to Mahadik et al. [28], at high-temperature calcination of 300 °C, there is a decomposition of hydrophobic groups such as alkyl and tend to be hydrophilic. Hydrophobic silica can maintain its hydrophobicity up to 275 °C. Above that, the temperature the nature changes to hydrophilic; this is because, above that temperature, the methyl group turns into Si-OH groups, which lead to water adsorption [29]. This answers the fact that all MTES silica thin layers produce surfaces with angles that are still less than 90°.





**FIGURE 5.** Relationship between MTES concentration and ratio of (a) Si-OH/Si-O-Si and (b) Si-C/Si-O-Si

According to Wang et al. [30], the sol-gel reaction is influenced by the temperature and evaporation of the solvent; therefore, it is possible that the character of thin films and xerogel can be different. Xerogel has low thermal stability, and it has relatively many Si-OH groups. In contrast, thin layer properties have a uniform surface, where it can coat the surface of the substrate uniformly with minimal defects and has a stable surface temperature [31]. Therefore, xerogel may be more hydrophilic due to the presence of Si-OH groups that are present in it, in which Si-OH groups are easy to interact with water [32] while thin layers may be more hydrophobic. However, to collect thin layer samples is not technically possible, so FTIR analysis was carried out on silica xerogels with the same content.

Figure 5b shows that the Si-C/Si-O-Si ratio for all MTES concentrations tends to be flat and still within the error margin. There is no significant change in the Si-C/Si-O-Si ratio for the five FTIR spectra, or it can be said that the Si-C/Si-O-Si ratio to be unchanged for all MTES concentrations. This result shows that variations in MTES concentrations do not affect the content of Si-C groups in silica polymers. It can be said that the increase in hydrophobic properties of the thin layer is not affected by the Si-C group.

## CONCLUSIONS

A silica thin layer, which was superimposed on the glass and xerogel surface, was synthesized using methyltriethoxysilane (MTES) precursors with variations in the precursor concentration of MTES 1.42, 2.85, 5.70, 11.40, and 22.80 M using ethanol and sol-gel method at a calcination temperature of 300 °C. The hydrophobicity of silica thin films increased with increasing MTES concentration. This was characterized by an increase in the contact angle of silica thin films from MTES concentrations of 1.42 M to 11.40 M. However, the resulting surface was still hydrophilic, which was characterized by contact angles of less than 90° for all MTES concentrations. With increasing MTES concentration, the Si-OH/Si-O-Si ratio, while the Si-C/Si-O-Si ratio tends not to change.

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