



Manufacture of a Hydrophobic Silica Nanoparticle Composite Membrane for Oil-Water Emulsion Separation

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Abstract. The superhydrophobic composite membrane was successfully manufactured by a sol-gel method by drying the surrounding pressure. Tetraethylorthosilicate (TEOS) was used as a hydrophobic agent, while waterglass was used as a source of silica. The effect of the water to waterglass ratio (noted at 16:1 and 19:1) was evaluated to study the hydrophobic properties of the silica film coated composite membrane surface. By measuring the water contact angle on the film surface, the highest contact angle was found to occur at the ratio of 19:1, which is 143.86°. The stability of the composite membrane was also investigated by immersing the membrane in water until day 6. The results show that the synthesized composite membrane has good stability until day 6. The hydrophobicity of the surface of the silica film membrane was found to be unaffected by immersion time. Furthermore, the hydrophobicity increased after 6 days due to the interaction of alkyl groups with the humidified environment, and the surface was more stable in hydrophobicity (i.e., the contact angle of water is 153.79°). In addition, hydrophobic properties were obtained, confirming that this film has the potential to be applied to the separation of oil-water emulsions.

Keywords: Composite membrane; Hydrophobic membrane; Oil-water emulsions; Silica film; Sol-gel

1. Introduction

The separation of oil-water emulsions has become a major concern since the increase in oil-producing industries, which generate liquid waste and oil-contaminated water bodies due to oil spills. Oil pollution has caused severe environmental problems. The technology of oil-water separation is also important for the purification of biodiesel production (Atadashi et al., 2012). One of the most developed methods for oil-water separation is the absorption of oil using porous adsorbent materials, such as aerogels (Xue et al., 2014), sponges (Su et al., 2017), and sawdust (Jung et al., 2008). These adsorbents must have special properties, namely hydrophobicity. This property allows adsorbents to absorb oil without absorbing water. Besides its use for oil-water separation, the hydrophobic membrane is also applicable for other separation systems (Kartohardjono et al., 2017; Kartohardjono et al., 2019). Recently, it was found that hydrophobic properties can be formed using chemicals by means of surface modification of adsorbents with hydrophobic

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material such as organosilanes, thus forming hydrophobic films.

Various methods have been developed to prepare these hydrophobic films, such as sol-gel processes (Xiu et al., 2008; Xue et al., 2010), layer by layer of self-assembly (Ismijan et al., 2012), etching (Guo et al., 2005; Dong et al., 2011), chemistry (Rezaei et al., 2014), and electrochemical deposition (Benoit et al., 2013; Khorsand et al., 2014). Among these methods, the sol-gel method is a relatively simple and inexpensive option for hydrophobic film preparation. In addition, this method does not require high temperatures for the preparation process, so it can be applied to large-scale production.

Silica is widely known as a basic ingredient in making porous adsorbents (Mahadik et al., 2010; Bhagat et al., 2008), and the synthesis of hydrophobic silica films using the sol-gel method has also been reported (Bois et al., 2003; Rao et al., 2009; Liu et al., 2014). The hydrophobic properties are obtained during the silylation process by modifying the surface of the silica film in a hydrophobic material, a process that increases the hydrophobic properties through the replacement of silanol groups on the surface of silica with alkyl groups (Roach et al., 2008; Celia et al., 2013; Prihandana et al., 2015;). Three alkoxy silanes are common hydrophobic agents, including tri methyl chloro silicate (TMCS), which is widely used in a variety of applications to improve adhesion between organic matter and inorganic substrates. This alkoxy silane molecule has two key clusters. One of these clusters is an organic compound that can be hydrolyzed and can react with other chemicals or groups that are not reactive and hydrolyzed (Corriu, 2003; Lung and Matinlinna, 2012) and the other is silica. The drawback is that most of these hydrophobic agents are expensive and limited in supply.

In a previous study, tetraethylorthosilicate (TEOS) was generally preferred as a precursor to synthesize silica (Jyoti et al., 2009). TEOS is alkoxy silane that has four alkyl groups, one of which is C_2H_5 , meaning that it is possible to use TEOS as a hydrophobic agent. TEOS is more economical than most of the other available hydrophobic agents, and its abundance is greater than silylation agents with three functional groups. Concerning silica sources, waterglass is the cheapest source of silica; there is also an abundance of large raw waterglass sources and it does not endanger the environment, which gives it good potential for industrial scale applications. On the other hand, the drying process of the silylation process is usually carried out through supercritical methods that require large amounts of energy, which limits commercial applications for this material. To reduce production costs, drying at atmospheric pressure can be applied.

Previous studies reported that waterglass can be made from rice husk extract and successfully applied as a precursor to produce hydrophobic silica aerogels by drying through ambient pressure using TMCS and hexamethyldisilazane (HMDS) as silylation agents (Nazriati et al., 2014). However, aerobic silica is made in the form of particles, not as a film. In this work, we report on the manufacture of hydrophobic silica composite membranes using the sol-gel method by drying atmospheric pressure. Waterglass was chosen as a source of precursor silica while TEOS was applied as a silylation agent. Silylation conditions are carried out at a certain time while the temperature and water ratio to waterglass are varied to get the optimum composite membrane characteristics with the highest hydrophobic properties and good stability. It was found that the manufactured composite membrane has the potential to be applied in the separation of oil-water emulsion. In addition, the method we propose promises to prepare hydrophobic silica-coated composite membranes while maintaining a low cost and relatively easy production. In sum, this study focuses on applying a novel method for hydrophobic silica composite membrane synthesis and determining its performance in the oil-water separation process.

2. Methods

Hydrophobic composite membranes with a support of cotton cloth are synthesized through a three-step procedure: (i) sol-gel, a synthesis process to prepare silica sol; (ii) a coating process to synthesize silica film; and (iii) a silylation process to obtain hydrophobic films. The raw materials such as water glass ($\text{SiO}_2:\text{Na}_2\text{O}$ with ratio of 3.02), ammonium hydroxide, acetic acid, n-hexane, and ethanol that were used as precursors in this research were analytical grade and purchased from Merck. TEOS was purchased from Sigma Aldrich in analytical grade. All the materials used in this research were directly used without further purification.

In the first step, silica soles were prepared using waterglass as the source. Waterglass was mixed with deionized water to prepare a 10% waterglass solution (%volume). The initial pH of the solution was 12. Then, 1 M of acetic acid was added dropwise into a waterglass solution until the pH of the solution reached 4. Then 1 M of ammonium hydroxide was added drop by drop into the solution until the pH of the solution increased to 6. The solution was mixed with a magnetic stirrer to get a homogeneous solution.

In the second step, the prepared silica sol was coated onto a cotton cloth substrate by the dip-coating method. Before the process, the cotton cloth was cleaned with acetone for 15 minutes and rinsed with methanol for 15 minutes, followed by drying in an oven at 105°C for 1 hour. After cooling the cotton cloth to room temperature, the cloth was ready for use as a substrate in the dyeing process. The cleaned cotton cloth was soaked in silica soles and pulled at a speed of 8 cm/min, followed by aging at 500°C for 1 hour. The cotton cloth coated with silica film was then immersed in ethanol for 1 hour at room temperature in a closed glass before being used in the next step.

Finally, in the third step, the silylation process was carried out by immersing the silica film coated on the glass into a mixture of TEOS and n-hexane. The TEOS and n-hexane ratios were 1 to 2 v/v. The temperature was varied between $30\text{--}50^\circ\text{C}$ over the processing time of 12 hours. Then, the coated substrate was dried at 120°C for 5 hours.

After the experiment was conducted, the chemical properties of silica films were investigated by Fourier Transform Infrared (FTIR) spectroscopy, while the composition of the silica film elements was examined by X-ray fluorescence (XRF). Scanning electron microscopy (SEM) was applied to check the morphology of the silica films. For measurement of the contact angle using a goniometer, water was dripped over the coated composite membrane, and the contact angle was then calculated. This Silica aerogel film was also coated onto a cotton cloth to evaluate its hydrophobicity and stability.

3. Results and Discussion

The membrane produced in this study was characterized according to membrane material. The characterization includes hydrophobicity, silica composition, functional group, and surface morphology of the membrane as determined by contact angle measurement, XRF, FTIR, and SEM, respectively. Table 1 shows the elemental composition of the silica film-coated composite membranes examined by XRF.

Table 1 Elemental composition of composite membrane

Compound	Si	Ca	Sc	Ti	Cr	Fe	Ni	Cu	Zn	Ba	Re
%	93.7	3.51	0.04	0.26	0.16	0.94	0.14	0.31	0.09	0.2	0.67

According to these results, the silica component was the highest element (up to 93.7%) found in the silica film layer in accordance with the basic material composition of

waterglass. Other elements were also detected in relatively small percentages, which may be caused by dirt and materials used during the sol-gel process.

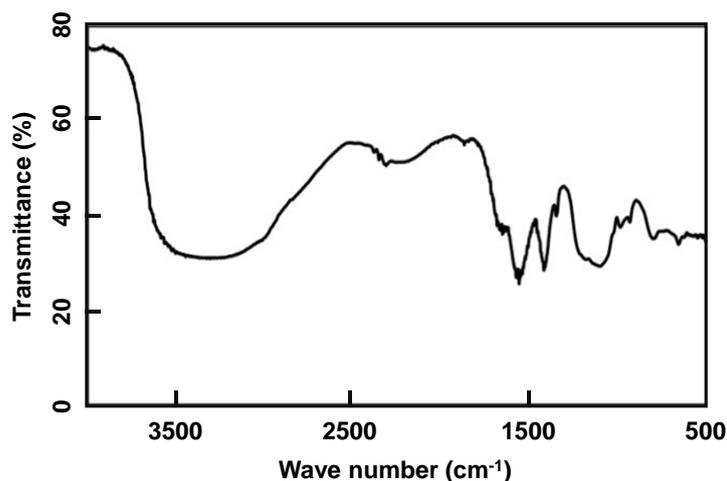


Figure 1 FTIR spectra of coated silica film

Figure 1 shows the FTIR spectrum of the hydrophobic silica layer prepared in silylation conditions at 30°C for 12 hours. This spectrum shows general bands that are characteristic of functional groups that exist on the surface of the film and interact with supporting materials. This spectrum analysis reveals a broadband centered around 3470–3450 cm^{-1} according to the overlap of O-H, which shows the water molecule functional groups bonded to hydrogen and Si-O-H stretching silanol (Brinker and Scherer, 1990). Furthermore, Si-O in the plane stretching Si-OH silanol groups appears at about 960 cm^{-1} (Gopal et al., 2004), and absorption bands associated with the deformation vibrations of adsorbed water molecules appear at 1653–1634 cm^{-1} (Socrates, 2001). The strong silicon-oxygen covalent bond appears mainly in the range of 1200–1000 cm^{-1} , which reveals the presence of solid silica groups on the tissue, where oxygen atoms play a bridge role between each of the two silicon sites (Duran et al., 1986). The symmetrical stretching of Si-O-Si appears at 469–467 cm^{-1} (Bertoluzza et al., 1982). Low energy bands around 560 cm^{-1} indicate Si-O stretches of SiO₂ tissue defects (Brinker and Scherer, 1990). The spectrum also shows peaks at 2970–2850 cm^{-1} and 1470–1340 due to C-H stretch vibration of -CH₃ groups. Si-C vibration stretching appears in the range 834–831 cm^{-1} . Bands at 788–783 cm^{-1} may overlap from Si-C and Si-O stretch vibrations. In addition, at 698–692 cm^{-1} , there is a Si-O symmetrical stretch vibration, and there is another one at 910 cm^{-1} (Al-Oweini and El-Rassy, 2009).

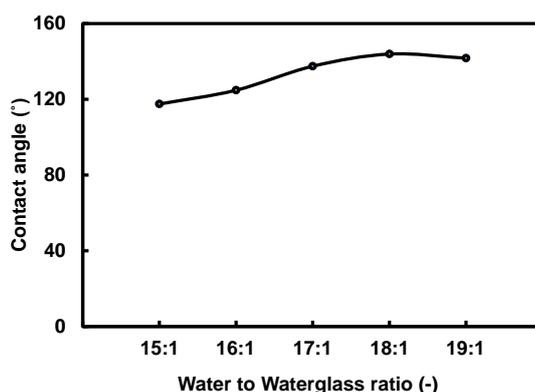


Figure 2 Effect of the ratio of water to waterglass and the contact angle of the silica hydrophobic membrane

The effect of the ratio between water and waterglass was shown in Figure 2. Measurements of the contact angle to water were used to evaluate hydrophobic properties. The measured contact angles of the silica film layer on the composite membrane prepared with 12 hours silylation time were $117.47 \pm 1.22^\circ$, $124.77 \pm 1.49^\circ$, $137.44 \pm 0.50^\circ$, $143.86 \pm 0.43^\circ$, and $141.70 \pm 0.63^\circ$, at water to waterglass ratio of 15:1, 16:1, 17:1, 18:1, and 19:1, respectively. The contact angle increased with an increase in the ratio of water to waterglass in the solution; however, the fourth variable showed the highest contact angle (143.86°). The ratio of water to waterglass also affected the properties of colloids in the mixture; the nature of the colloid is a major factor for the formation of nano-sized particles. The formation of nanoparticles affects hydrophobicity—the smaller the size of the particles formed, the larger the surface hydrophobicity. This hydrophobicity property, due to the surface of the silica, was modified by the exchange of hydroxyl groups with alkyl groups from TEOS. The alkyl group is a non-polar group that causes repulsion of water, thereby forming hydrophobic films.

The greater the water to waterglass ratio, the more the silica surface was modified by the alkyl group and the less hydroxyl remained, increasing the hydrophobicity of the silica film layer. However, when it reached 10 hours the hydrophobic layer of the silica film decreased, as shown by the decrease in the water contact angle. This was because the presence of water fractions was increasing, which led to the surface—which has hydrophobic properties—being distributed less evenly. There was a chance that the surface was not hydrophobic. The gap space filled with water easily interacts with ethanol molecules, thus weakening the hydrophobic structure of silica. When pore silica contains ethanol during the drying process, ethanol evaporates together with Si-C₂H₅, reducing the hydrophobicity of silica film. According to the results above, the fourth variable was chosen as the optimal composition for the process of making composite membranes. To study the stability of composite membranes, the composite membrane was immersed in water until day 6.

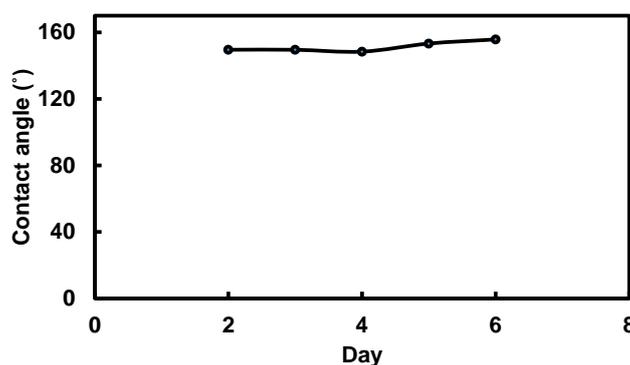


Figure 3 Effect of immersion time on the stability of the composite membran

The measurement of the water contact angle for the stability of the composite membrane with the prepared silica film layer was shown in Figure 3. This showed that there was no significant change in the contact angle of the silica-water as immersion time increased. The statistical method confirms this result through a calculation of the Least Significant Difference (LSD). LSD was 0.795 with a probability of 5%. This showed that the immersion time did not have a significant effect on the stability of the hydrophobic properties of the silica film layer on the composite membrane.

The highest measured water contact angle was 153.79° , which shows that superhydrophobic properties (Roach et al., 2008; Celia et al., 2013; Drelich and Marmur,

2013; Law, 2014) are obtained at day 6. The immersion time until day 6 gives an opportunity for the aging of hydrophobic groups on the surface of the composite membrane, and the hydrophobic groups were evenly distributed on the surface so that the hydrophobicity properties increased until day 6. However, the changes were not statistically significant.

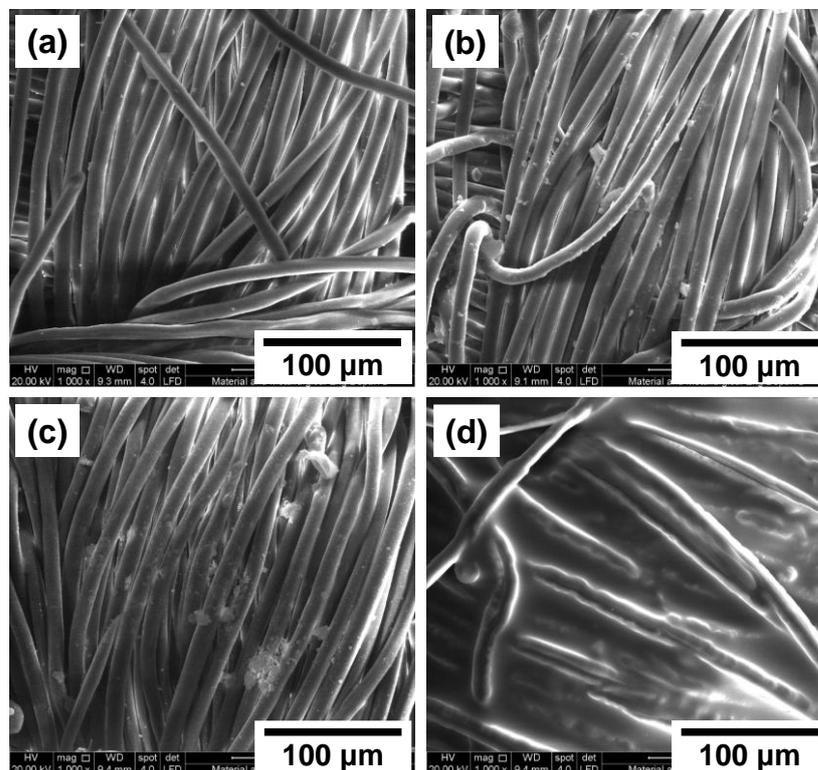


Figure 4 SEM images of the surface morphology of: (a) cotton fabric after initial preparation and composite membrane; (b) after silylation; (c) after surface modification; and (d) after separation

Figure 4a showed the surface morphology of the cotton fabric after initial preparation; the surface of cotton fibers was relatively smooth and there was no patch of other material. Figure 4b showed the morphology of the surface of the cotton fabric that had been coated by silica sol with conditions at the silylation temperature and time of 30°C and 12 hours, respectively. Some parts have patches of silanol compounds found on the surface and evenly distributed. The combination of cotton cloth that has been coated with silanol groups on its surface can be referred to as a composite membrane. The cotton fibers surface was roughened by the silica coating and further modified by TEOS, providing roughness to allow the composite membrane to become superhydrophobic. In this study, we successfully obtained superhydrophobic properties using only one precursor, TEOS, as hydrophobic agent instead of using a mixture of two precursors, such as TEOS with 1,1,1,3,3,3-hexamethyl disilazane (HMDS), cetyltrimethoxysilane (CTMS), or Polydimethylsiloxane (PDMS) (Xue et al., 2013; Xue et al., 2017).

The morphology of the composite membrane surface modification of the surface to make it more hydrophobic by immersing it in a mixture of TEOS, n Hexana, and ethanol in a ratio of 1:1:1, using TEOS as a hydrophobic agent or silylation was shown in Figure 4c. The role of silylating agents was not only to provide hydrophobic groups but also to strengthen the structure of silica by filling the silica pores because TEOS is a tetra silylating agent function in which silica is bonded to 4 alkyl groups—silica is more stable than the

trifunctional silylation group. In the case of tri-functional silylation groups such as TMCS, silica is bound to 3 alkyl groups and 1 Cl group where Cl makes the silica bond unstable and makes it easy to bind with silica groups in the pores. In the case of tetra-functional silylating agent, because silica is bound by four alkyls, silica becomes more stable and difficult to bind with silica groups in the pores. Therefore, the use of TEOS may not strengthen the pore structure of silica. Silica stability of hydrophobic films was also evaluated by measuring the contact angle of water droplets on a silica surface coated with hydrophobic glass for four days. Figure 4d showed the surface morphology of hydrophobic membranes after being used for the separation process of the oil and water mixture. The surface of the fiber seems to be covered by a layer of oil; this was due to the hydrophobic nature of the fiber surface. Water molecules could not interact with the membrane surface because of the repulsive force between the two. This was because the structure of the porous silica is weak, as described in Figure 5. The weak bond between silica and alkyl groups makes the alkyl group easy to cut when exposed to the wet environment. Therefore, the wet environment decreases the hydrophobicity of the surface of the silica film.

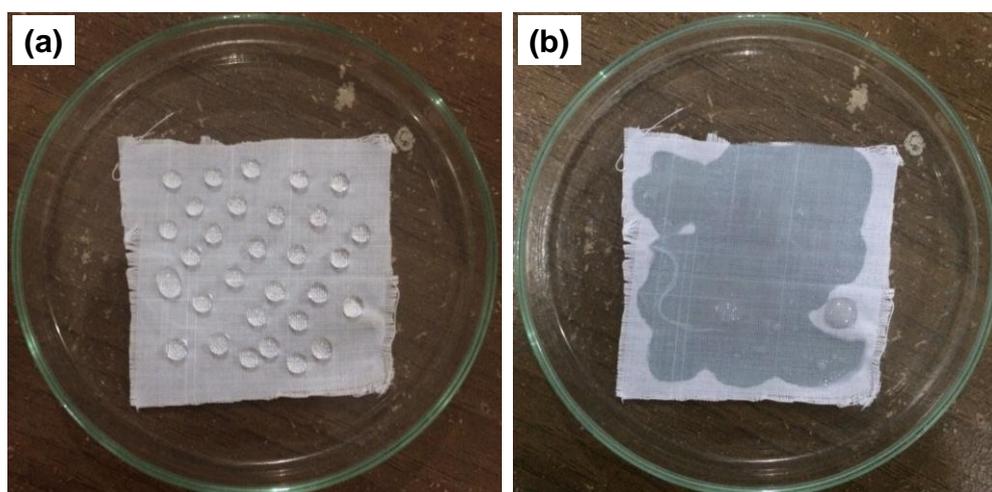


Figure 5 Shape of water (a) and oil droplet (b) on the cotton fabric coated by hydrophobic silica

Figure 5a and 5b showed the shape of water and oil droplets, respectively, on the cotton fabric coated by silica hydrophobic. In the case of water droplets on the coated cotton fabric (Figure 5a), there was a contact angle of about 144.25° , indicating that this coated cotton fabric has hydrophobic properties. In the case of the oil droplet on the coated cotton fabric (Figure 5b), the oil spread quickly and penetrated directly on the cotton coated cloth. This may be due to the full coverage of alkyl groups modified by silica on the surface of cotton fabrics and the capillary effects of cotton fabrics (Xue et al., 2013). This phenomenon showed the hydrophobic and oleophilic properties of coated cotton fabrics, which to our knowledge have never been reported before. Therefore, this film can be applied in the field of oil-water separation.

4. Conclusions

A composite membrane consisting of cotton cloth coated with silica film was successfully manufactured. It was found that this composite membrane with the support of cotton cloth combined with coating with silica film using TEOS as a surface modification agent was effective for delivering hydrophobic properties. The optimal ratio of the water to waterglass in the solution occurred at 19:1—that is, at a contact angle with water of

143.86°—which provided the best hydrophobicity. The hydrophobicity test results on day 6 increased to 153.79°, and this membrane was stable until day 6. This hydrophobicity provides the main factor for a more efficient oil-water emulsion separation. In addition, the method we propose can be used to prepare superhydrophobic silica-coated composite membranes with relatively easy production for oil-water separation.

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