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Hydrophobicity Properties of Silica-Dimethylsilicone Oil-Stearic Acid Based Materials Applied as Waterproof Coating Materials

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Abstract. Geothermal solid waste is one of the byproduct materials from the geothermal energy process. The silica extracted from this solid waste can be utilized as a raw material for waterproof coatings. The waterproof or hydrophobic properties in this study have been studied with a formulation of silica, stearic acid, and dimethyl silicone oil. The technique of extracting silica from geothermal waste is carried out using the sol-gel method. Preparation of hydrophobic materials is carried out by mixing silica with stearic acid at a ratio of 1:3, 1:5, and 1:7 on a mass basis with the addition of dimethyl silicone oil as an emulsifier. The hydrophobic silica-dimethyl silicone oil-stearic acid materials were synthesized and characterized by functional group analysis using FTIR. The best hydrophobic properties are shown in a ratio of silica and stearic acid of 1:5. On a glass substrate, the silica-dimethyl silicone oil-stearic acid coating produced a contact angle of 110.2 °. On a fabric substrate, the coating exhibited a superhydrophobic phenomenon with a contact angle of 153.7 °.

Keywords: *superhydrophobic, water repellent coating, stearic acid, silica*

Abstrak. Limbah padat geotermal merupakan salah satu material hasil samping dari proses pengolahan energi yang bersumber dari panas bumi. Kandungan silika dalam limbah padat ini dapat dimanfaatkan dengan cara diekstrak dan direkayasa, salah satunya adalah sebagai bahan baku pelapis anti-air pada permukaan material. Sifat anti-air atau hidrofobisisitas dalam penelitian ini telah dipelajari dengan formulasi antara silika, asam stearat dan dimetilsilicone oil. Metode ekstraksi silika dari limbah geotermal dilakukan dengan metode sol-gel. Pembuatan material hidrofobik dilakukan dengan pencampuran antara silika dengan asam stearat pada perbandingan 1:3; 1:5; dan 1:7 berbasis massa dengan ditambahkan dimetilsilicone oil sebagai emulsifier. Hasil yang didapatkan adalah material hidrofobik silika-dimetilsilicone oil-asam stearat mampu disintesis dengan karakteristik yang ditunjukkan dari analisis gugus fungsi dengan FTIR. Sifat hidrofobik terbaik ditunjukkan pada perbandingan silika dan asam stearat 1:5. Pada substrat kaca, pelapis silika-dimetilsilicone oil-asam stearat menghasilkan sudut kontak sebesar 110,2°. Pada substrat kain, pelapis dapat menunjukkan fenomena superhidrofobik dengan nilai sudut kontak sebesar 153,7°.

Kata kunci: *superhidrofobik, pelapis anti air, asam stearat, silika*

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INTRODUCTION

In geothermal energy processing, the geothermal well drilling process is carried out to a depth of 2000 m which passes through lava flows in volcanic areas. In practice, the process involves the flow of geothermal fluids that contain a lot of silicic acid at high temperatures. The cooled silicic acid will settle on the walls of the geothermal well pipe as solid SiO_2 (Gallup

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& von Hirtz, 2015; Svavarsson et al., 2014). The solid silica has no use value in the geothermal energy extraction process, so it is considered waste in geothermal power generation units.

The utilization of silica waste converted into silica has been carried out in the fields of construction, forensics, textiles, bioanalysis, adsorbents, and as a catalyst (Gupta, 2020; Irshad & Rahul, 2017; Jenie et al., 2020; Naat, 2022; Paramitha et al., 2019; Untoro et al., 2020). The wider utilization of silica is by modifying its hydrophobic properties so that it can be applied as a coating material (Crucho, 2024; Ghodrati et al., 2023; Taurino et al., 2008). Silica-based coating materials then continue to develop, especially in the use of added hydrophobic agents and coating techniques on silica, resulting in hydrophobic characteristics of the coating material that reach superhydrophobic levels (Al-Husseny et al., 2022; Ismail et al., 2021; Jiang et al., 2025; Latthe & Rao, 2012; R. Sutar et al., 2020; R. S. Sutar et al., 2019). Hydrophobic agents commonly used in the synthesis of hydrophobic materials are derivatives of silane compounds, which are still relatively expensive and difficult to obtain.

In this study, an alternative to hydrophobic agents, stearic acid, is used, which is more widely available. The use of stearic acid as a hydrophobic agent has been used as a bitumen coating and is superhydrophobic (Ismail et al., 2021). The use of stearic acid as a hydrophobic agent has also been implemented on the surface of printed circuit boards (PCBs) and produces a superhydrophobic coating (X. Li et al., 2023). Based on the research that has been done, the manufacture of superhydrophobic materials based on geothermal waste silica in this study will be applied to a wider range of substrates, such as glass and polyester fabrics.

MATERIAL AND METHODS

Materials

The raw materials used were solid geothermal waste samples from PT. Geo Dipa Energi Dieng unit. The reagents used in the study had pro analysis levels, namely sulfuric acid, nitric acid, hydrochloric acid, sodium hydroxide, and stearic acid. Supporting reagents in the synthesis of silica and coatings included dimethylsilicone oil (DSO) from Shin-etsu Japan, ethanol at technical levels, and distilled water.

Instrumentation

Characterization of the silica layer includes the morphology of the hydrophobic silica layer in the substrate using the image method from the results of the Zeiss Scanning Electron Microscopy (SEM) with a magnification of 20,000 times, the presence of functional groups using the Shimadzu Prestige 21 Fourier Transform Infrared (FTIR) spectroscopy method. Contact angle to see the level of hydrophobicity of silica with the substrate using the Oppo 12 cellphone camera with data processing using ImageJ and Microsoft Excel software.

Procedure

The research work procedure is divided into four steps that consist of raw material preparation, silica synthesis, coating on the substrate, and characterization of the coating product on the substrate.

Raw material preparation

The geothermal solid waste is homogenized in size to 325 mesh using a sieve shaker, which is then leached with 25% sulfuric acid. The leaching product is then filtered and dried to be processed to the silica synthesis stage.

Silica synthesis

The procedure for silica synthesis using the sol-gel method follows the reference (Ismail et al., 2021).

Coating materials preparation

The coating step of the glass substrate is carried out by adding a hydrophobic agent of stearic acid to silica. The addition of DSO is carried out in a constant amount to the three mixtures. Technically, the composition of the coating material is stated in Table 1.

Table 1. Composition of silica, stearic acid, and DSO for the preparation of hydrophobic silica-DSO-stearic acid material

Number Code	Silica (g)	Stearic acid (g)	DSO (g)
1.	0.2	0.6	1
2.	0.2	1	1
3.	0.2	1.4	1

Coating to glass and polyester fabric substrate

The next step is stirring and heating at 60 °C until the silica, hydrophobic agent, and DSO are perfectly mixed. The contacting process of the mixture with the substrate, which is prepared by the dip coating method.

Particle size distribution analysis

The particle size distribution of the synthesized silica was analyzed using ImageJ software. SEM micrographs were processed by setting the scale and applying thresholding to distinguish individual particles. The diameters of random particles were measured to obtain representative data. The resulting measurement data were then exported and plotted as a histogram using Veusz software to determine the average particle size and the distribution pattern.

RESULT AND DISCUSSION

Raw Material Preparation

Geothermal solid waste in the form of lumps is mechanically ground to obtain a smaller size. Screening with a sieve shaker with a size of 325 mesh aims to obtain a uniform particle size of raw materials. The raw materials that have been uniformly processed by the leaching method using 25% sulfuric acid aim to remove metal oxide compounds contained in the solid raw material sample and only leave silica compounds that are insoluble in acid compounds. The reaction of metal oxides that can dissolve in sulfuric acid follows reactions (1) and (2).

$$3H_2SO_{4(aq)} \rightarrow 6H^+ + 3SO_4^{2-} \quad (1)$$
$$6H+ + 3SO_4^{2-} + X_2O_3 \rightarrow X_2(SO_4)_3 + H_2O \quad (2)$$

The residue from the leaching process in the form of solids containing concentrated silica is dried to remove water content at a temperature of 105 °C. Visually, the raw material residue is obtained with a lighter color before undergoing the leaching process. This is in accordance with reaction (2) where sulfuric acid can dissolve metal oxides into sulfate compounds that are soluble in aquadest (Silviana et al., 2020).

Silica Synthesis

Silica synthesis is carried out by the sol-gel method, where the preparation goes through the sol phase stage, which produces sodium silicate sol and pH value adjustment with acid so that silica is produced in the gel phase. The reaction that occurs follows the reaction between silica and NaOH, which produces sodium silicate and water.

$$SiO_{2(s)} + 2NaOH_{(aq)} \rightarrow Na_2SiO_{3(aq)} + H_2O_{(l)} \quad (3)$$

After sodium silicate is formed, the addition of nitric acid aims to form a silica compound that has a silanol group, $Si(OH)_4$, and with drying, the $Si(OH)_4$ compound forms a white solid (Ismail et al., 2021). The reaction that occurs in the formation of $Si(OH)_4$ is written in reaction (4).

$$Na_2SiO_{3(aq)} + 2HNO_{3(aq)} + H_2O_{(l)} \rightarrow 2NaNO_{3(aq)} + Si(OH)_{4(l)} \quad (4)$$

In the synthesis process of silica, the sonication treatment aims to help homogenize the sol phase formed between silica and NaOH so that it reacts perfectly. The use of ethanol in the manufacture of silica aims to form silanol groups more quickly due to the presence of hydroxyl groups from ethanol and is followed by the condensation process of silanol groups into condensed silica compounds as stated in reaction (5) (Ismail et al., 2019).

$$Si(OH)_4 + Si(OH)_4 \rightarrow (HO)_3\text{-}Si\text{-}O\text{-}Si\text{-}(OH)_3 + H_2O \quad (5)$$

The centrifugation process aims to finalize the formed silica compound sediment so that it is separated from its filtrate. Dry silica (xerogel) still contains silanol groups that are needed for the preparation of hydrophobic materials.

Coating of Hydrophobic Material on Substrate

The first step in coating hydrophobic materials is to make silica, which is hydrophobic. Hydrophobic silica is made by mixing synthetic silica with an agent that forms hydrophobic properties, namely stearic acid. In this study, variations in the amount of stearic acid were carried out to see the effectiveness in forming hydrophobic properties in materials with silica as the raw material. In order to form a hydrophobic compound, an emulsifier is needed because silica tends to be hydrophilic and stearic acid is hydrophobic. To overcome this problem, dimethyl silicone oil (DSO) is used as an emulsifier. The interaction between the silica-DSO-stearic acid system is illustrated in Figure 1.

The interaction that occurs between the hydroxyl group on silica and the hydroxyl group on DSO is a hydrogen bond. Stearic acid added to the silica-DSO system has formed a covalent bond between the hydroxyl group of the carboxylic group of stearic acid and the hydroxyl group on DSO (X. Li et al., 2023). The interaction of the three components causes the silica/DSO/stearic acid system to be hydrophobic from the carbon chain in the alkyl group of the stearic acid compound and on the other side is hydrophilic which can be bound to the substrate because it contains abundant hydroxyl groups.

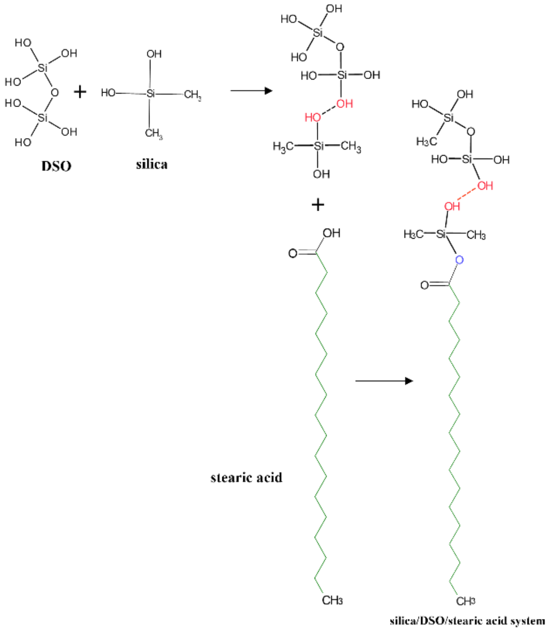

Figure 1. Interaction between silica, DSO, and stearic acid in a system of hydrophobic material

The hydrophobic material coating stage on the substrate is carried out by heating and dip coating methods. Heating at a temperature of 60 °C and stirring aims to make the hydrophobic silica system homogeneous and in a liquid phase. The dip coating and drying treatment at a temperature of 180 °C aims to ensure that the coating material adheres to the substrate evenly and perfectly (R. S. Sutar et al., 2019).

Characteristics of Silica and Hydrophobic Coatings

The initial characterization of silica is observing morphology and determining the particle size using the Scanning Electron Microscopy (SEM) method. The results obtained from SEM analysis show that the morphology of the silica particles is round, with agglomeration occurring in several parts. The particle sizes obtained from the SEM image approach show that silica particles have a size distribution below 1 μm, and the largest distribution is in the range of 200-250 nm, as shown in Figure 2.

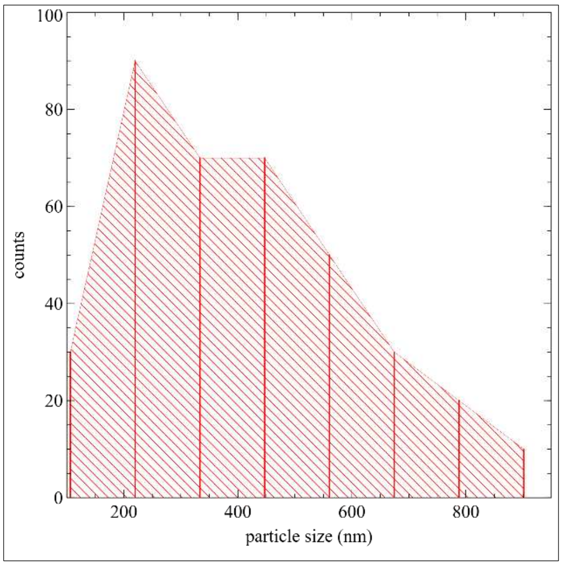

Figure 2. Silica size distribution after data processing with ImageJ and Veusz 3.6.2 software.

The distribution of particle sizes shows that the sizes of silica synthesized using the sol-gel method are of various sizes. This is because the silica form is still in the form of a silanol compound with the characteristic of having hydroxyl bonds on its surface. These hydroxyl bonds cause the formation of agglomerates that vary in size from one silica compound to another.

Characterization of the functional groups of the silica-DSO-stearic acid coating system was carried out by interpreting the spectra produced by FTIR analysis. The spectra of the synthesized silica are used as a comparison, as shown in Figure 3.

The FTIR spectrum of silica shows characteristic absorption at 470.63 cm^{-1}; 796.60 cm^{-1}; and 1095.57 cm^{-1} which shows characteristic absorption of Si-O-Si bend (Loganathan et al., 2013; Mujiyanti et al., 2020). Absorption at wave number 956.70 cm-1 shows the presence of the vibration of Si-OH, which is characteristic of the silanol group in silica compounds (Alattar, 2021; Cui et al., 2011). The characteristic of silica, which is still hydrophilic, is shown in absorption at wave number 1631.78 cm^{-1} as absorbed H_2O (K. M. Li et al., 2014). In addition, the characteristic absorption at wave number 3455.22 cm^{-1} shows the presence of a hydroxyl group (-OH), which strengthens the hydrophilic nature of silica used as raw material.

The FTIR spectrum of the silica-DSO-stearic acid system shows several characteristics indicating chemical interactions between silica, DSO, and stearic acid. Absorption at wave numbers 466.67 cm^{-1}; 800.46 cm^{-1}; and 1097.50 cm^{-1} indicates the characteristics of silica identity functional groups, namely Si-O-Si bends (Loganathan et al., 2013).

The covalent bonds found in the DSO compound are shown in the absorption wavenumber 686.66 cm$^{-1,}$ which is the absorption characteristic of Si-C (Cui et al., 2011). The alkyl groups in stearic acid are shown in absorption at various wavenumbers. The absorption that appears at 719.45 cm^{-1} shows the bend in the C-H group of alkanes (Mohrig et al., 2010). The presence of the C-H group of alkanes is reinforced by the emergence of new absorptions at wavenumbers 2848.86 cm^{-1}; 2916.37 cm^{-1}; and 2960 cm^{-1}. In addition, the absorption at 1469.76 cm^{-1} reinforcing the presence of C-H group absorption in alkane (Mujiyanti et al., 2020; Yuan et al., 2018).

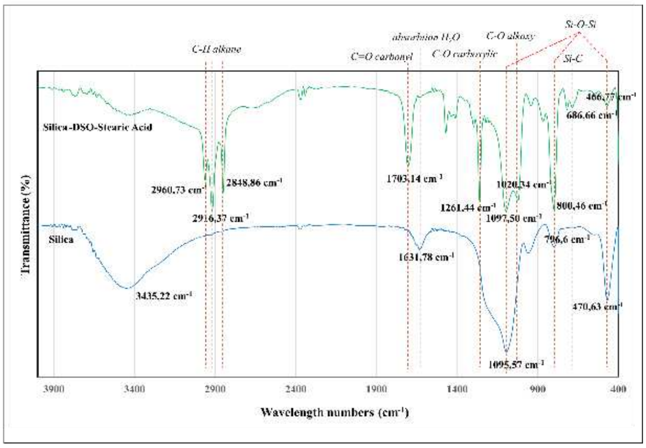

Figure 3. FTIR spectra of silica-DSO-stearic acid in the hydrophobic material system

The chemical interaction in the silica-DSO-stearic acid system is demonstrated by the presence of a new absorption formed at a sharp wavenumber at 1020.34 cm^{-1} as a C-O alkoxy group. This group is formed due to the breakdown of the hydroxyl group in stearic acid and covalently bonded with Si in DSO, and forms a Si-O-(C=O)- bond. The carbonyl group in stearic acid is stated in the new wave number absorption at 1703.14 cm^{-1}.

From several characterizations using FTIR spectra, it can be stated that the interaction between silica-DSO-stearic acid is indicated to occur as a covalent bond or a hydrogen bond (X. Li et al., 2023). The occurrence of covalent interaction is strengthened by the absorption of the hydroxyl group wave number, which is significantly reduced to 3435.22 cm^{-1} in the silica spectrum. This indicates that the hydroxyl group is reduced due to the formation of a sharper Si-O group at 800.46 cm^{-1}.

The inversion of the hydrophilic properties of the silica raw material used to become a hydrophobic material was initially indicated by the loss of absorption of 1631.78 cm$^{-1,}$ which is characteristic of the absorption of water (K. M. Li et al., 2014).

Visualization of the water contact angle on the substrate surface that has been coated with silica-DSO-stearic acid is a parameter that indicates the hydrophobicity of the material surface. The results of contact angle observations at various ratios of silica and stearic acid are stated in Figure 4.

The coating material showed the highest hydrophobicity symptoms on both glass and polyester fabric substrates with an optimal mass ratio of silica and stearic acid of 1:5. The hydrophobic phenomenon of the silica-DSO-stearic acid system coating is due to the presence of long chains of alkyl contained in the hydrophobic agent stearic acid facing the surface (X. Li et al., 2023). Superhydrophobic properties in observations were shown in coatings applied to the surface of polyester fabrics (Liu et al., 2021). This is due to the strong interaction between the hydroxyl groups on silica and the surface of polyester fabrics, which contain abundant carboxyl groups.

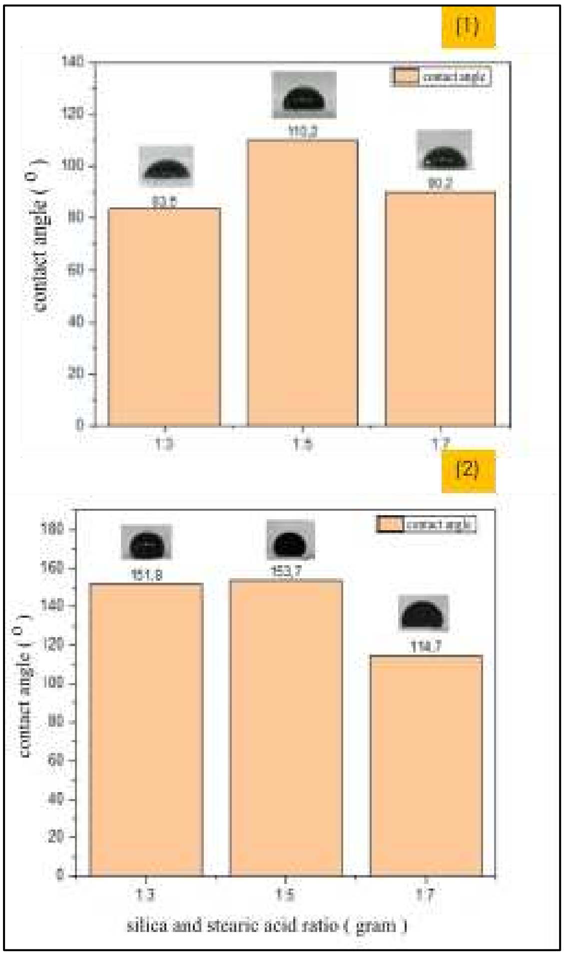

Figure 4. Contact angle of hydrophobic material on the glass substrate (1) and polyester fabrics (2) in various ratios between silica and stearic acid.

The experimental results demonstrated that the contact angles for silica-to-stearic acid ratios of 1:3 and 1:5 on polyester fabric substrates were nearly equivalent, showing only marginal differences. This phenomenon can be explained by both chemical synergetic effects and the physical morphology of the coating.

Chemically, the hydroxyl groups on the silica surface interact with the free oxygen-containing groups of the polyester fibers through hydrogen bonding and Van der Waals forces, ensuring strong adhesion of the primer layer. In this system, dimethyl silicone oil (DSO) effectively acts as a molecular bridge, facilitating the uniform attachment of stearic acid—the primary hydrophobic agent—onto the silica particles. At a 1:3 ratio, the concentration of stearic acid is already sufficient to saturate the available active sites on the silica surface, forming a dense and well-ordered hydrophobic layer. As the ratio increases to 1:5, the system reaches a chemical saturation point where the hydrophobic tails are closely packed, leaving no significant residual or unbound stearic acid.

The integration of silica particles into the micro-scale fibers creates hierarchical roughness. This structural arrangement promotes air pockets are trapped within the fabric's pores beneath the water droplet. Since the optimal surface coverage and hierarchical morphology are already effectively established at both 1:3 and 1:5 ratios, any additional stearic acid does not significantly lower the surface energy or alter the air-trapping capability, resulting in stabilized superhydrophobic performance.

The interactions that occur can be in the form of hydrogen bonds because, according to its function, the coating material does not react with the substrate material, which produces by-products. The stability of hydrophobic properties on the substrate is observed over time as presented in Figure 5.

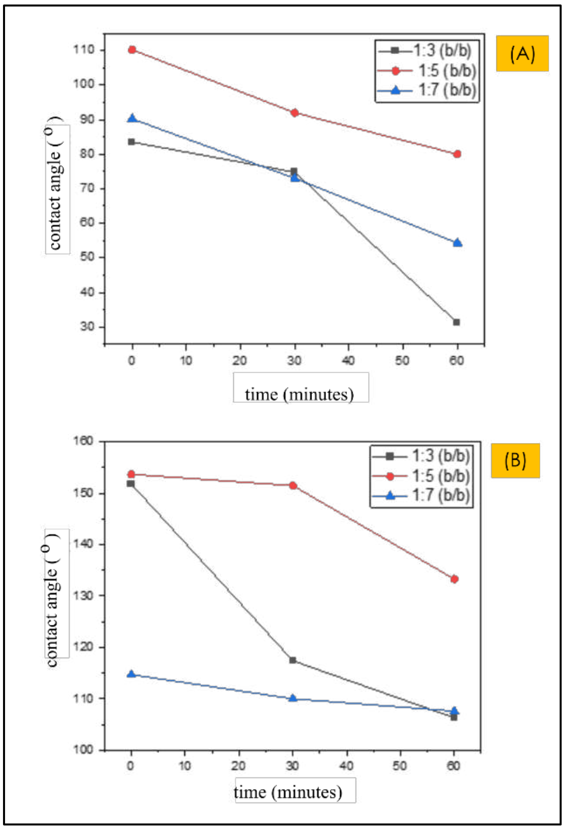

Figure 5. Contact angle stability as a function of time for hydrophobic materials on glass (A) and polyester fabric (B) at different silica-to-stearic acid ratios.

In observing the stability of hydrophobic properties, it was found that both substrates were able to maintain hydrophobic properties on a minute scale and continued to decrease as indicated by the contact angle value over time. This is because the interaction caused by the substrate and coating is only able to maintain hydrophobic properties on a certain time scale. The factor that causes the short interaction between the substrate and the coating is that the interaction that occurs between the two is still limited to physical interactions.

On polyester fabric substrates, hydrogen bonds can occur between hydroxyl groups on silanol and carboxyl on the substrate. On glass substrates the instability of hydrophobicity is caused by the adhesion force of the glass surface with the coating material. Therefore, coatings on glass substrates tend not to be able to maintain hydrophobic properties because the interaction between the glass surface and the coating material is limited to physical interactions.

A significant anomaly was observed in the 1:7 silica-to-stearic acid ratio, where the initial contact angle at minute 0 was markedly lower than that of other ratios, as illustrated in Figure 5B. This sharp decline in hydrophobic performance can be attributed to the excessive concentration of stearic acid. The overabundance condition of stearic acid molecules creates a steric hindrance effect that disrupts the effective interaction between the silica particles and the polyester substrate (Wang et al., 2025). This hindrance prevents the silica from anchoring securely to the fabric fibers, thereby inhibiting the formation of a stable hydrophobic network.

Furthermore, from a morphological perspective, the excess stearic acid acts as a redundant organic phase that submerges the silica particles. Instead of creating the necessary hierarchical roughness, the surplus acid fills the interstitial spaces between fibers and silica, resulting in a relatively smooth, waxy surface. This loss of surface roughness prevents the entrapment of air pockets, causing the system to fail in achieving the Cassie-Baxter state required for superhydrophobicity (Maghsoudi et al., 2023). Consequently, even at the initial contact (minute 0), the material exhibits a significantly lower contact angle as the water droplet interacts with a more uniform organic layer rather than a roughened, air-trapping interface.

CONCLUSION

Silica can be extracted from geothermal solid waste by the sol-gel method with a variety of sizes distributed at sizes below 1 μm. The silica-DSO-stearic acid-based coating is optimally hydrophobic at a silica and stearic acid ratio of 1:5. The waterproof coating shows superhydrophobic symptoms on the polyester fabric substrate with a contact angle value of 153.7 °.

The stability of the best hydrophobic coating with the contact angle measurement method against time is also shown in the silica and stearic acid ratio at a 1:5 ratio on the fabric substrate.

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