Coating of colloidal silica on glass surfaces with bbk nano silica as raw material

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Abstract. Due to the colloidal silica has many applications such as coating, a catalyst precursor, a nano-size filler, investment casting, semiconductor wafer polishing, and an inorganic binder, it still attracting to be studied. The colloidal silica layer has been coated on a glass substrate using the spray-coating process. Two kinds of colloidal silica were prepared, i.e., the colloidal silica formed by mixing of nano-silica from Balai Besar Keramik (BKK) and the silane hexamethyldisilazane (HMDS), and the colloidal silica polishing Mastermet (0.06 mm) commercial as a comparison. The HMDS percent was varied at 10, 20, and 30 vol.%. The contact angle measurement obtained the hydrophobic properties of the samples, and a UV-Vis spectrophotometer measured the transmittance samples. A water contact angle of 94.9° is achieved at 10% HMDS. For all samples, the transmittance of the before and after coated glass substrate can reach an average of 89% to 90%. From this study, it is concluded that the BBK nano-silica has met the commercial requirements that are the hydrophobic property in the contact angle and the transmittance.

1. Introduction
Colloidal silica still attractive to be studied because it has many applications such as coating, a catalyst precursor, a nano-size filler, investment casting, semiconductor wafer polishing, and an inorganic binder \cite{1}. The silica particle is dispersed in water because of -OH groups, which are polar and hydrophilic on the silica surface \cite{2}. In this study, nano-silica synthesis from Balai Besar Keramik (BKK) was made in 2011. This nano-silica was synthesized by mixing namethasilicate (water glass) and deionized water with half the amount of HCl, followed by the addition of HMDS and HCl a drop by drop \cite{3}.

Many researchers are trying to modify the nano-silica particle using silane coupling agents to improve the physical properties. If silanes are added to the colloidal silica, it surrounds the surface of colloidal silica through the hydrolysis and condensation reactions. The surface of silane treated colloidal silica is hydrophobic \cite{4}. This solution can be applied in many fields such as architecture, building windows, lens and optical devices, solar panels, etc. \cite{5,6}. There are several reagents that can modify nano-silica for coatings such as tetraethoxysilane (TEOS), trimethyletoxysilane (TMES), phenythrietoxyxilane (FTES), tetramethyoxysilane (TMOS), ethyltriethoxysilane (ETOS), trivinylmethyoxysilane (TVMS), trimethylchlorosilane (TMCS), hexamethyldisilazane (HMDS), and
dimethylalkoxysilane [7,8]. This study used hexamethyldisilazane (HMDS) as the modifying reagents. The HMDS will react with hydroxyl groups on the silica surface and could be represented by the following chemical reactions (Figure 1).

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\begin{align*}
\text{Si}^+\text{OH} & \quad (\text{CH}_3)_2\text{Si}^+ \quad \text{Si}^+\text{O} - (\text{CH}_3)_3 \\
\text{O} & \quad + \quad \text{N-H} \quad \text{O} & \quad + \quad \text{NH}_2 \\
\text{Si}^+\text{OH} & \quad (\text{CH}_2)_2\text{Si}^+ \quad \text{Si}^+\text{O} - (\text{CH}_3)_3
\end{align*}
\]

| Unmodified Film Surface | HMDS | Modified Film Surface | Ammonia |
|-------------------------|------|-----------------------|----------|

*Figure 1. Chemical reactions of silica and HMDS [9].*

This study aims to obtain a coating of colloidal silica with BBK nano-silica as raw material on glass surfaces. The samples are characterized using the contact angle and transmittance measurements, then it is compared to colloidal silica polishing as commercial material.

2. Experimental

2.1. Materials

Nano-silica obtained from Balai Besar Keramik (BBK) Bandung, Hexamethyldisilazane (HMDS) and Colloidal Silica Polishing Suspension Mastermet obtained from Merck and Buehler, respectively. Glass substrates used in size of 5 cm×10 cm×3 mm (length × width × thickness).

2.2. Preparation of Colloidal Silica

Colloidal silica was synthesized by mixing nano-silica, aquadest and HMDS. The 1 kg nano-silica and 1000 ml aquadest were milled for 48 hours to form a functionalized colloidal silica. A 250 ml of functionalized colloidal silica was mixed with 500 ml aquadest using a magnetic stirrer for 1 hour. Functionalized colloidal silica was divided into four beaker glasses. One glass without adding HMDS while another tree glass was added HMDS with 10, 20, and 30 % (v/v), respectively. Then those glasses were mixed using a magnetic stirrer for 24 hours, followed by a sonicator for one hour at room temperature.

2.3. Spray-Coating

The obtained-colloidal silica was coated on the transparent glass substrates by spray coating. Coating layers were dried for 2 hours at room temperature then wiped with a microfiber cloth so that colloidal silica filled the glass pores. The spray coating was carried out twice. The contact angle and transmittance measurements were conducted for each spray coating.

2.4. Characterization

The contact angles of water droplets on the coated-glass substrates were measured using a contact angle meter (KYOWA interface measurement and analysis system, FAMAS). The transmittance of the coated-glass surface was measured using Spectrophotometer UV-Vis (Perkin Elmer Lambda 950).

3. Results and discussions

The hydrophobicity property of a coated-glass surface using the sessile drop technique for contact angle measurement. One of the samples with HMDS 10% (v/v) is measured its contact angle and it is obtained 94.9° as shown in Figure 2.
With a similar procedure, the contact angle is measured on a sample for HMDS variation of 10, 20, and 30 vol.%. There are three samples for each HMDS variation, so the average of the contact angle for each sample is shown in Figure 3. For one time, the spray coating, the contact angle increases relative to without coating. Besides, the contact angle increases at 10 and 20 % (v/v) for two times the spray coating while it decreases at 30% (v/v) and commercial of colloidal silica polishing.

The contact angle increase means that the hydrophobic properties increase. When the silane solution is added to colloidal silica, colloidal silica will be hydrophobic. The -Si-(CH₃)₃ groups of the HMDS replace the hydroxyls of the surface OH’s of silica and it forms a layer on the surface that enhance hydrophobic [8]. On the contrary, in the case two time the spray coating, Tasaltin et al.
concluded that the surface roughness is responsible for decreasing the contact angle than functional groups’ presence [10].

Table 1. The transmittance of coated glass substrates in the UV-Vis wavelength

| Colloidal Silica          | Transmittance before coating | Transmittance after coating |
|---------------------------|------------------------------|----------------------------|
| 10%(v/v) HMDS             | 89.694                       | 89.980                     |
| 20%(v/v) HMDS             | 90.014                       | 90.160                     |
| 30%(v/v) HMDS             | 89.904                       | 89.988                     |
| Colloidal Silica Polishing| 89.960                       | 89.824                     |

Table 1 summarizes the transmittance of the coated glass substrates in the range of UV-Vis wavelength. There was no change in transmittance between the coated and uncoated glass substrates significantly. However, these results have transmittance almost similar to the colloidal silica polishing. Furthermore, due to the coated-glass substrate for HMDS 10%(v/v) has the highest contact angle relative to the colloidal silica polishing as commercial material, it can be applied in building windows, lens and optical devices, and solar panels [5].

4. Conclusion
The colloidal silica with nano-silica BBK as a raw material has been successfully coated on the glass substrate using the spray coating method. Adding the HMDS 10%(v/v) has the highest the contact angle and high transmittance. Therefore, this sample meets the requirement of the colloidal silica commercial.

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