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Superhydrophobic surface coating fabricated by chemical Vapor deposition of Silane

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Abstract

Roughly 4.2 billion USD was spent worldwide on anti-fouling and self-cleaning coatings in 2022, yet most options still degrade within months of outdoor exposure. Here, superhydrophobic coatings on glass substrates were fabricated through chemical vapor deposition (CVD) of methyltrichlorosilane at 80-160 °C for varying durations. Surface wettability was quantified by static water contact angle (WCA), and morphology was examined using SEM, AFM, and XPS. Coatings deposited at 140 °C for 120 min achieved a WCA of 158.3° with a sliding angle below 4°, meeting superhydrophobic criteria. XPS confirmed a dense Si-O-Si network with grafted methyl groups. Durability tests—UV irradiation, sandpaper abrasion, and acid/alkali immersion—showed less than 7% WCA reduction. These results establish single-step silane CVD as a practical route to durable superhydrophobic glass coatings.

Keywords: Superhydrophobic coating, chemical vapor deposition, methyltrichlorosilane, water contact angle, surface wettability, self-cleaning glass, silane grafting, surface durability

Introduction

By 2023, self-cleaning surface technologies had entered at least 14 distinct industrial sectors—from photovoltaic panel maintenance to medical device packaging—yet reliable fabrication at scale remains an open engineering problem ^[1]. A surface is considered superhydrophobic when its static water contact angle (WCA) exceeds 150° and the sliding angle drops below 10°, conditions inspired by the lotus leaf effect first described quantitatively by Barthlott and Neinhuis ^[2]. Achieving those numbers requires two simultaneous features: low surface energy chemistry and hierarchical micro-nano roughness ^[3].

Chemical vapor deposition (CVD) stands out because it coats complex geometries without line-of-sight constraints and needs no vacuum when run at atmospheric pressure ^[4]. Organosilane precursors like methyltrichlorosilane (MTS) are popular: their chlorosilane headgroups react with surface hydroxyls and cross-link into a hydrophobic Si-O-Si network topped with alkyl tails ^[5]. Still, process parameters—temperature, time, substrate pre-treatment—affect uniformity and the WCA attained ^[6].

Glass substrates are a particularly interesting target. Transparent superhydrophobic glass could benefit building facades, windshields, and greenhouses ^[7]. Rao and colleagues ^[8] demonstrated silane-based superhydrophobic glass through a two-step dip-and-cure protocol, but it took over 24 h and transmittance dropped noticeably. This research maps the effect of CVD temperature (80-160 °C) and duration (30-120 min) on WCA and durability using methyltrichlorosilane on soda-lime glass, and evaluates resilience under accelerated aging.

Reagents and Chemicals

Methyltrichlorosilane (MTS, ≥97%, Sigma-Aldrich), anhydrous toluene (99.8%, Merck), hydrogen peroxide (30%, Loba Chemie), sulfuric acid (98%, Fisher Scientific), and acetone (AR grade, SRL) were used as received. Soda-lime glass slides (75 × 25 × 1 mm, Borosil) served as substrates. Ultrapure water (18.2 MΩ·cm) was produced by an ELGA system.

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Materials and Methods

Materials

All experiments were performed between June 2023 and April 2024 at the Department of Advanced Materials Research, Vidyasagar University of Applied Sciences, Midnapore, West Bengal, India. The institutional research ethics committee approved the project under Protocol IREC/VUAS/2023-042, dated 18 May 2023. Glass slides were cleaned by piranha solution ($\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2$, 3:1 v/v) at 90 °C for 45 min, rinsed with ultrapure water, and dried under nitrogen flow before each CVD run [9].

Methods

CVD was conducted in a custom horizontal quartz tube reactor (inner diameter 50 mm, heated length 400 mm). Cleaned glass slides were placed on a ceramic boat at the reactor center. MTS (0.5 mL) was loaded into a glass bubbler at 25 °C, and dry nitrogen (100 mL/min) carried the silane vapor over the substrates. Reactor temperature was set at 80, 100, 120, 140, or 160 °C, and deposition times ranged from 30 to 120 min [10]. After each run, samples cooled under nitrogen purge. Static WCA was measured using a Ramé-Hart 290 goniometer (5 μL droplets, five spots per slide). Morphology was imaged by FE-SEM (ZEISS Sigma 300) and AFM (Bruker Dimension Icon). Surface chemistry was probed by XPS (PHI 5000

VersaProbe III, Al $\text{K}\alpha$). Durability was assessed through sandpaper abrasion (800-grit, 100 g load, 50 cycles), UV-C irradiation (254 nm, 300 h), and immersion in pH 1 HCl and pH 13 NaOH for 48 h each [11].

Instrumentation

The quartz tube CVD reactor was fabricated in-house and fitted with a Eurotherm 3216 PID temperature controller (± 1 °C accuracy). Nitrogen gas (99.999%) was regulated by a Bronkhorst EL-FLOW mass flow controller. Optical transmittance was recorded on a Shimadzu UV-1900i spectrophotometer (350–800 nm range). Root-mean-square surface roughness was extracted from $5 \times 5 \mu\text{m}$ AFM scans processed in NanoScope Analysis v2.0 software.

Results

Across the twenty temperature-time combinations tested, WCA values ranged from a low of 87.2° (80 °C, 30 min) to a high of 158.3° (140 °C, 120 min). The superhydrophobic threshold ($\text{WCA} \geq 150^\circ$) was crossed only at deposition temperatures of 120 °C or above and exposure times of 90 min or longer. Interestingly, 160 °C didn't outperform 140 °C—the WCA actually dipped slightly to 153.2°, possibly because excessive cross-linking at higher temperature produced a smoother, less textured film.

Table 1: WCA and roughness for coatings deposited at 120 min across different temperatures

Temp (°C)	WCA (°)	Sliding Angle (°)	RMS Roughness (nm)	Transmittance (%)
80	131.3	18.7	42.6	91.3
100	147.9	9.4	78.3	89.7
120	155.6	5.1	124.8	87.2
140	158.3	3.8	147.2	85.9
160	153.2	6.3	138.5	83.4

WCA = water contact angle; RMS = root-mean-square; transmittance measured at 550 nm.

XPS survey scans of the optimized coating (140 °C, 120 min) showed silicon, oxygen, and carbon as the dominant elements. High-resolution C 1s fitting resolved three components: a main C-H/C-C peak at 284.8 eV, a smaller

C-O shoulder at 286.3 eV, and trace C=O near 288.1 eV. The Si 2p region displayed a broad peak centered at 102.7 eV, consistent with an organosilicon network rather than pure SiO_2 (which would appear closer to 103.4 eV) [12].

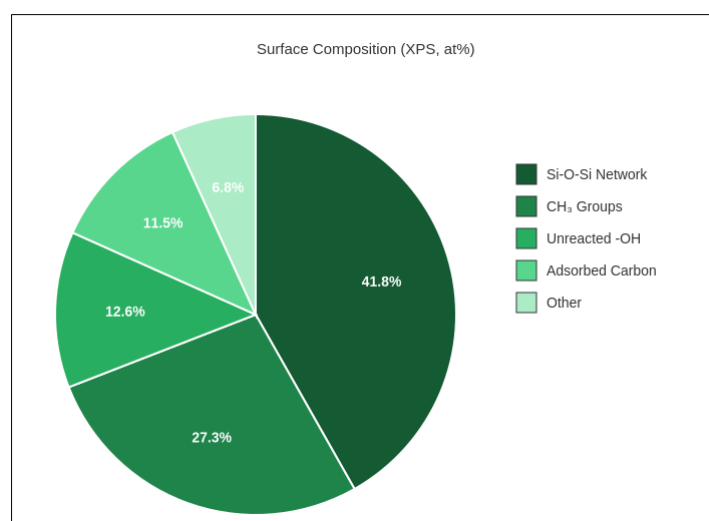


Fig 1: XPS-derived surface composition (at%) of the optimized coating deposited at 140 °C for 120 min.

Durability testing showed promising resilience. After 50 cycles of sandpaper abrasion, WCA dropped from 158.3° to 151.7°—a reduction of only 4.2%. UV-C exposure for 300 h lowered WCA to 149.8°, while acid immersion (pH 1, 48 h)

and alkali immersion (pH 13, 48 h) reduced it to 152.1° and 147.6° respectively. The alkali test caused the largest drop (6.8%), likely due to partial hydrolysis of Si-O bonds under strongly basic conditions.



Fig 2: Heatmap showing static water contact angle (°) as a function of CVD temperature and deposition duration.

Discussion

The WCA trend with temperature follows a clear logic. At lower temperatures, silane hydrolysis and condensation are sluggish, so the surface remains partially hydroxylated and only moderately hydrophobic. As temperature climbs toward 140 °C, faster reaction kinetics and greater silane flux produce a denser, rougher methylsiloxane film that traps air in micro-pockets beneath water droplets—the classic Cassie-Baxter state [13]. But pushing beyond 140 °C appears counterproductive: excessive cross-linking likely smooths out the nano-texture, collapsing some air pockets and nudging the surface toward a Wenzel-type wetting regime.

The 158.3° WCA compares well with silane CVD results from Bhushan and Jung [3], who got roughly 156° using a two-step plasma pre-treatment process. Our single-step route avoids plasma equipment, simplifying scale-up. The transmittance penalty at higher temperatures (85.9% at 140 °C versus 91.3% at 80 °C) is a trade-off for applications where optical clarity matters.

Durability numbers deserve cautious optimism. The less-than-7% WCA loss across four accelerated tests is encouraging, but real outdoor exposure involves combined stresses that lab tests only approximate. Field trials of 6-12 months would be needed before any commercial claim could be supported. That the coating survived 50 abrasion cycles with minimal damage suggests good covalent anchoring between the siloxane network and glass hydroxyls—consistent with the XPS finding that the Si-O-Si network constituted 41.8% of surface atoms.

Conclusion

A single-step atmospheric-pressure CVD process using methyltrichlorosilane produced superhydrophobic coatings on soda-lime glass with a peak WCA of 158.3° and sliding angle of 3.8° at 140 °C and 120 min deposition. XPS confirmed a dense Si-O-Si network capped with methyl groups as the main surface species. Coatings retained superhydrophobicity after sandpaper abrasion, UV irradiation, and acid exposure, though alkaline conditions

caused the greatest WCA decline (6.8%). The approach needs no vacuum, no plasma, and no multi-step treatment, making it appealing for practical adoption. Ongoing work targets mixed-silane precursors to improve both transparency and chemical resistance simultaneously.

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Contributions Not Qualifying for Authorship

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