EPFL
École polytechnique fédérale de Lausanne

CHEMINA
Laboratory of Chemical Nanotechnology

Surface Chemistry
Lesson 8

MSE 304

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November 7th 2025

Plan of the Course: Fundamentals, Characterization, and Applications

1: Intro to Surfaces & Interfaces

2: Surfaces in the Real World - Adsorption

3: Surface Energetics & Interfacial Phenomena

4: Atomic Structure of Real Surfaces

5: Solid-Solid Interfaces

6: From Ideal Planes to Real Materials (Recap)

7: Characterization of Surfaces & Interfaces

8: Surface Chemistry

9: Surface Chemistry and Patterning

10: Characterization of Molecular Assembly

11: Biological Processes at Surfaces

12: Electronic Properties of Surfaces

13: Biosensor Fundamentals

14: Biosensing applications

Recap from Lesson 7

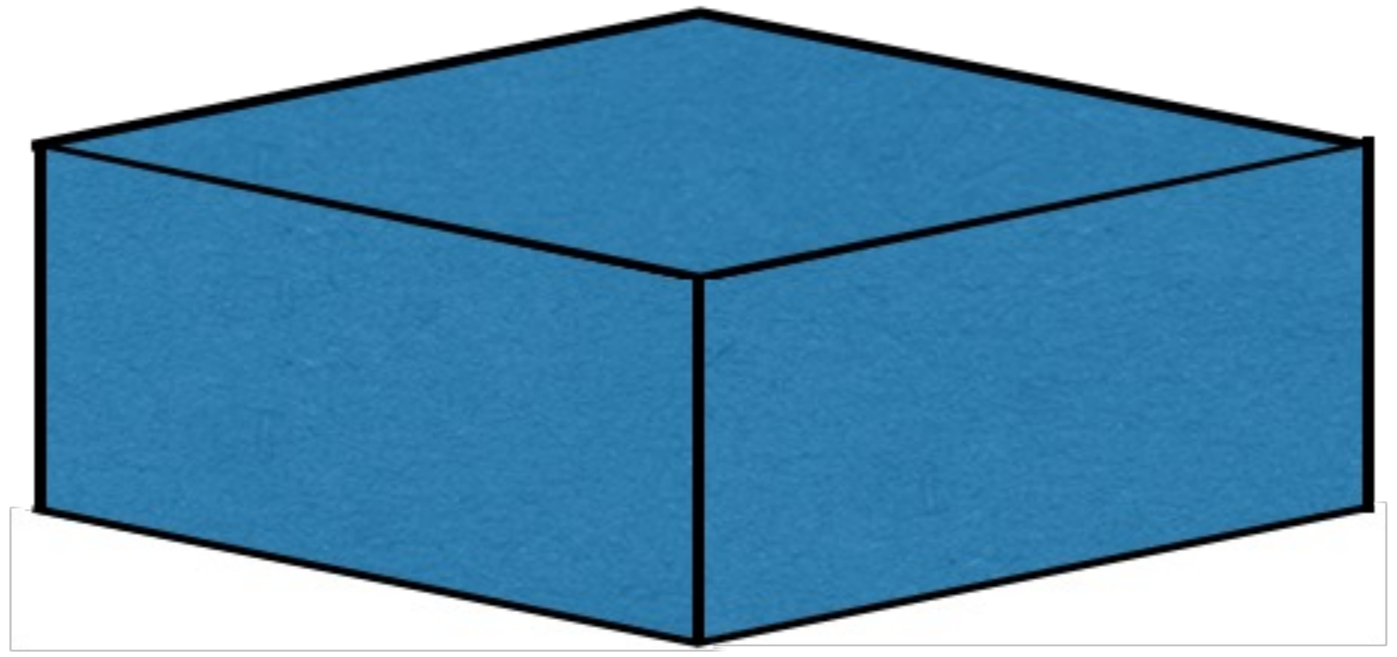
- We can visualize surfaces using electron microscopy techniques (TEM/SEM)
- SEM add-ons like EBDS and EDX characterize grain boundaries/elements respectively
- Overcome challenges of studying surfaces using ions and low-energy electrons (secondary ion mass spectrometry and x-ray photoelectron spectroscopy)
- XPS relies on the photoelectric effect to be a surface-sensitive technique
- Work function is a material property but is influenced on surface properties

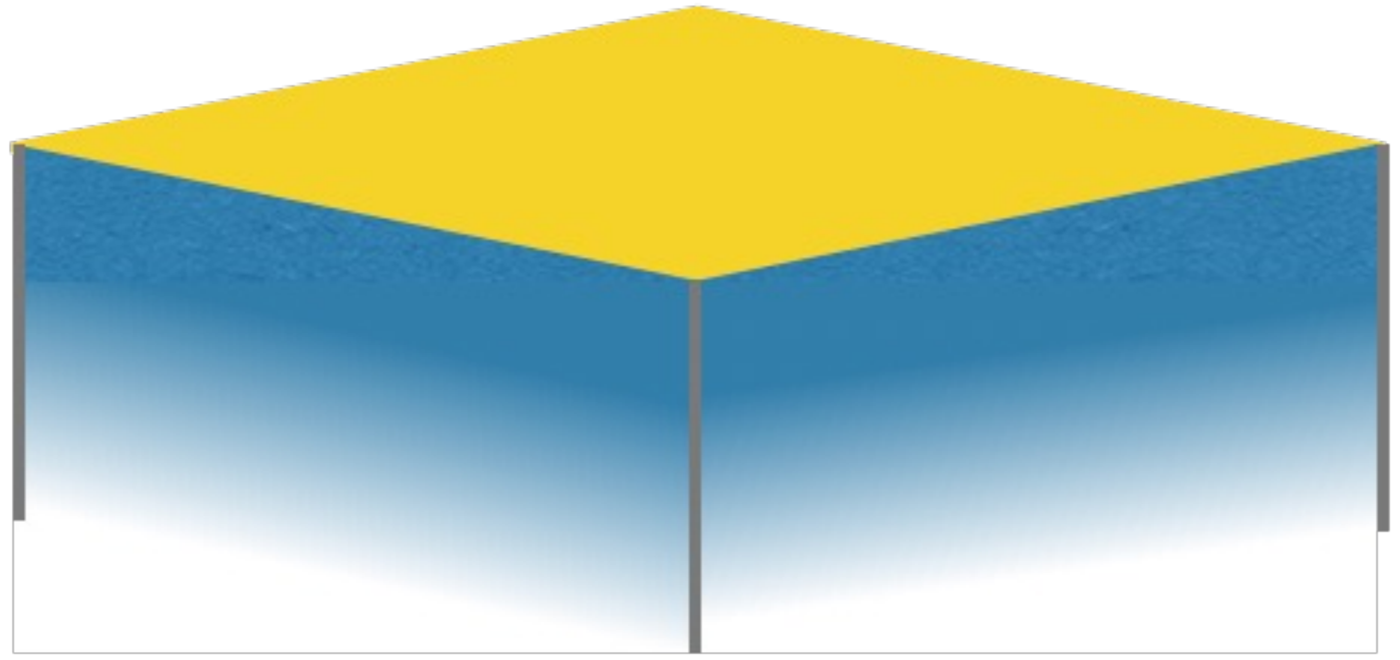
Outline of Lesson 8

- How to functionalize surfaces using self-assembly
- Factors governing self-assembly
- Various surface binding chemistries (thiol, silane, carboxyl)
- Challenges and opportunities of various surface chemistries
- How do we characterize these self-assembled monolayers?
- Recap: Hydrophobicity/hydrophilicity, intermolecular forces, pKa values, techniques

Surface Chemistry – Why Functionalize Surfaces?

The goal is to combine ideal bulk properties with ideal surface properties





Bulk Properties

Reflect behavior of atoms/molecules within interior of material

Porosity

Thermal conductivity

Electrical conductivity

Density

Elasticity

Tensile strength

Magnetism

Surface Properties

Properties unique to atoms on material's surface where they are less coordinated and experience different forces

Wettability

Biocompatibility

Corrosion resistance

Roughness

Lubricity/ease-of-cleaning

Catalytic activity

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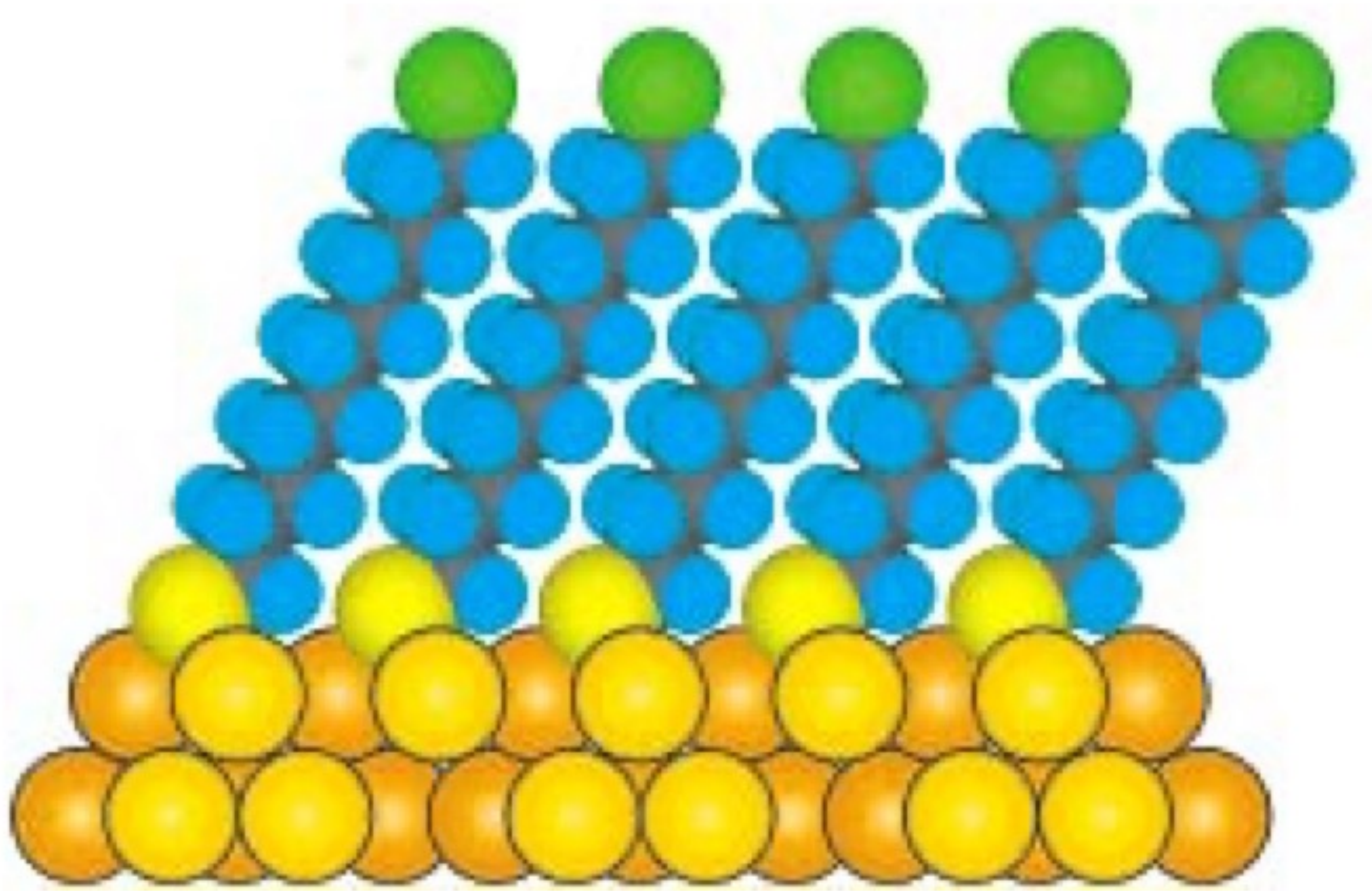
How to Functionalize Surfaces? Self-Assembled Monolayers

Spontaneously adsorbed, single layer of species with a high degree of lateral organization

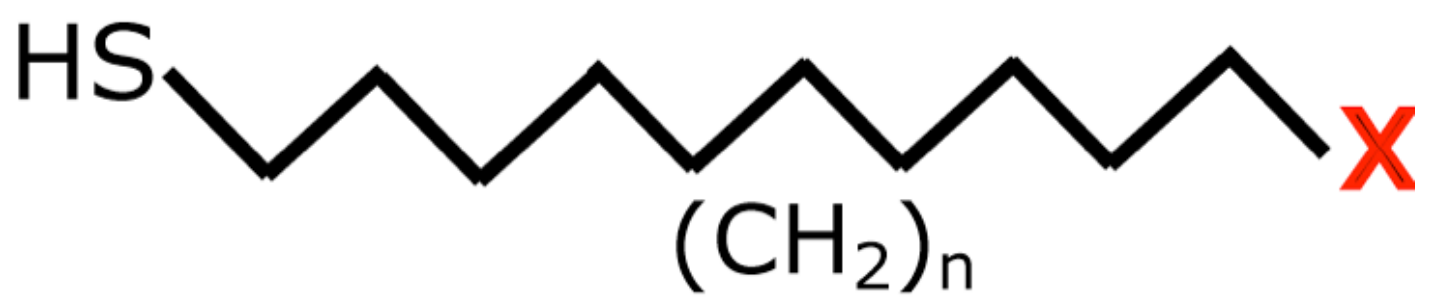


Functional group: mainly determines surface properties

Hydrocarbon chain: interchain van der Waals interactions

Head/anchoring group: interacts with substrate (chemisorption)

Examples of alkanethiols that spontaneously self-assemble on surfaces:



X = tail group/functional group

CH_3, OH, COOH, NH_2, *etc.*

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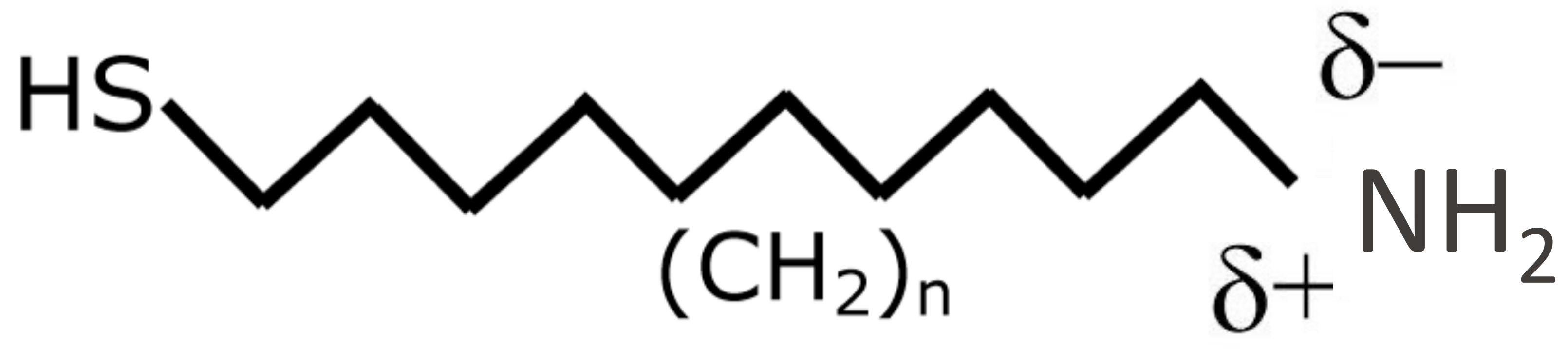
Hydrophilic *vs.* Hydrophobic Terminal Groups

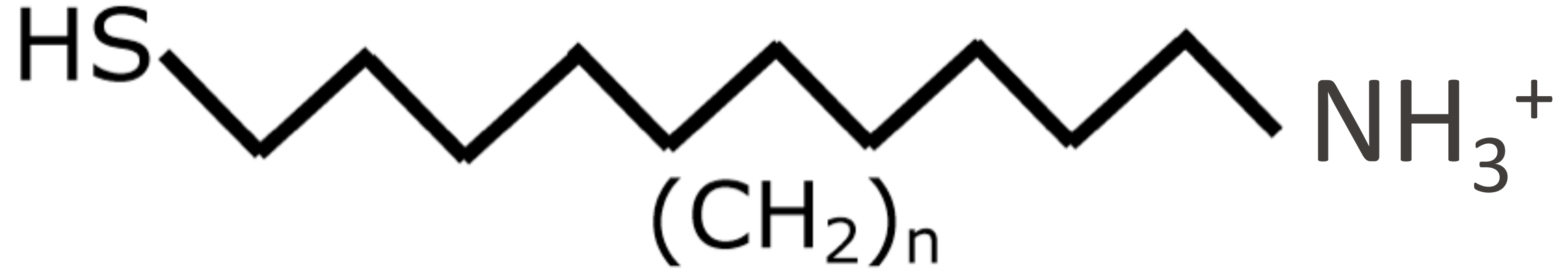
Hydrophilic terminal groups

- OH (hydroxyl)
- COOH (carboxyl)
- NH_2 (amine)

Polar

Hydrogen-bonds or ionic interactions with H_2O



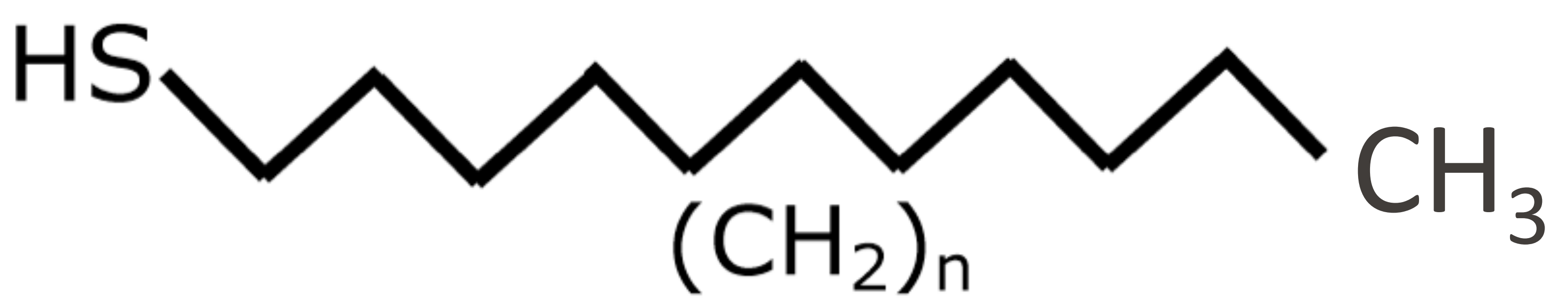


Hydrophobic terminal groups

- CH_3 (methyl)
- CF_3 (triflyoromethyl)
- C_6H_5 (phenyl group)

Non-polar

Minimal interactions with water due to low polarity



Which form of the amine-group is more hydrophilic?

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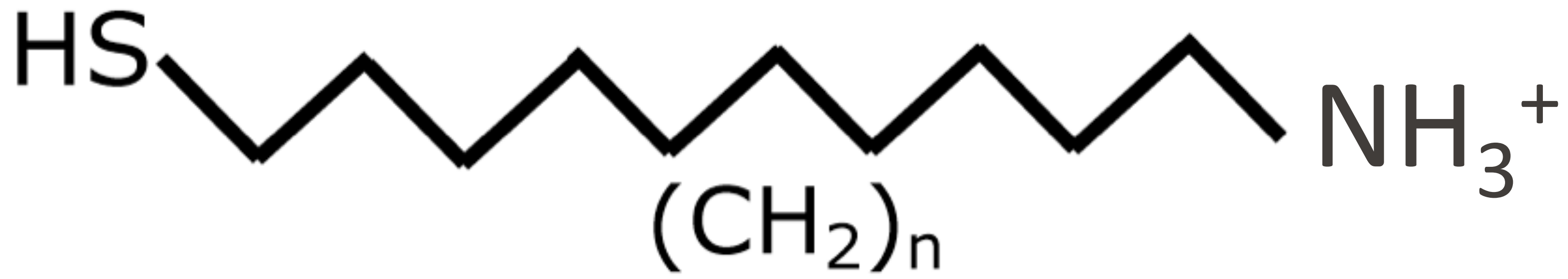
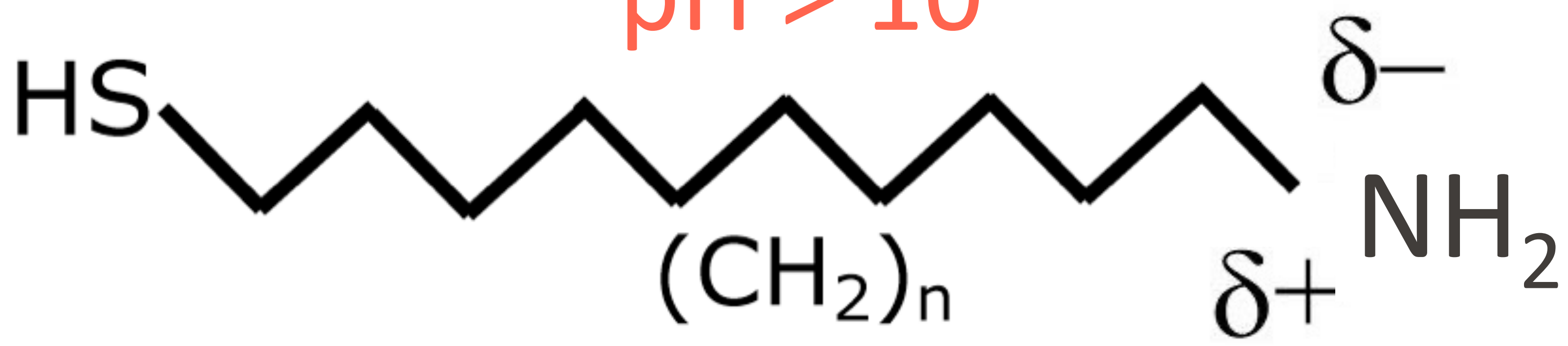
Recall the Strength of Intermolecular Interactions/Forces

pH < 10



pKa ~ 10

pH > 10

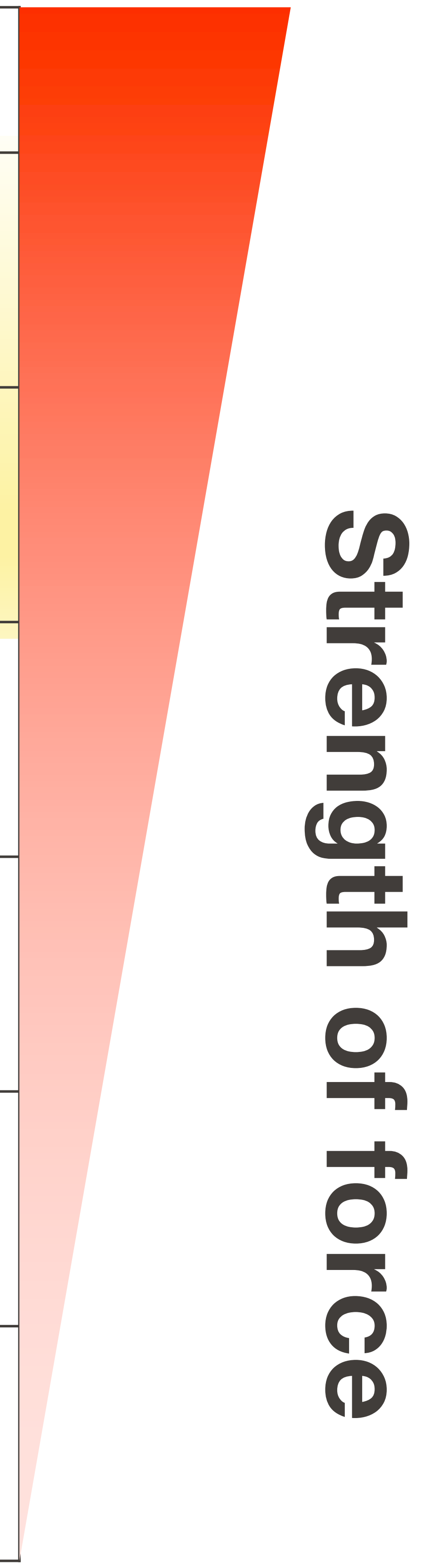


Acid dissociation

$$HA \rightleftharpoons H^{+} + A^{-}$$

$$K_a = \frac{[A^-][H^+]}{[HA]}$$

Intermolecular Forces	Formed by attraction between:
Ion-dipole	Ion + polar molecule
Hydrogen bond	Molecules with H, N, O, F atoms
Dipole-dipole	Two polar molecules
Ion-induced dipole	Ion + nonpolar molecule
Dipole-induced dipole	Polar + Nonpolar molecule
Van der Waals forces	Two nonpolar molecules



Henderson-Hasselbach equation

$$pH = pK_a + log\frac{[A^-]}{[HA]}$$

pH < pKa ➔ protonated
pH > pKa ➔ deprotonated

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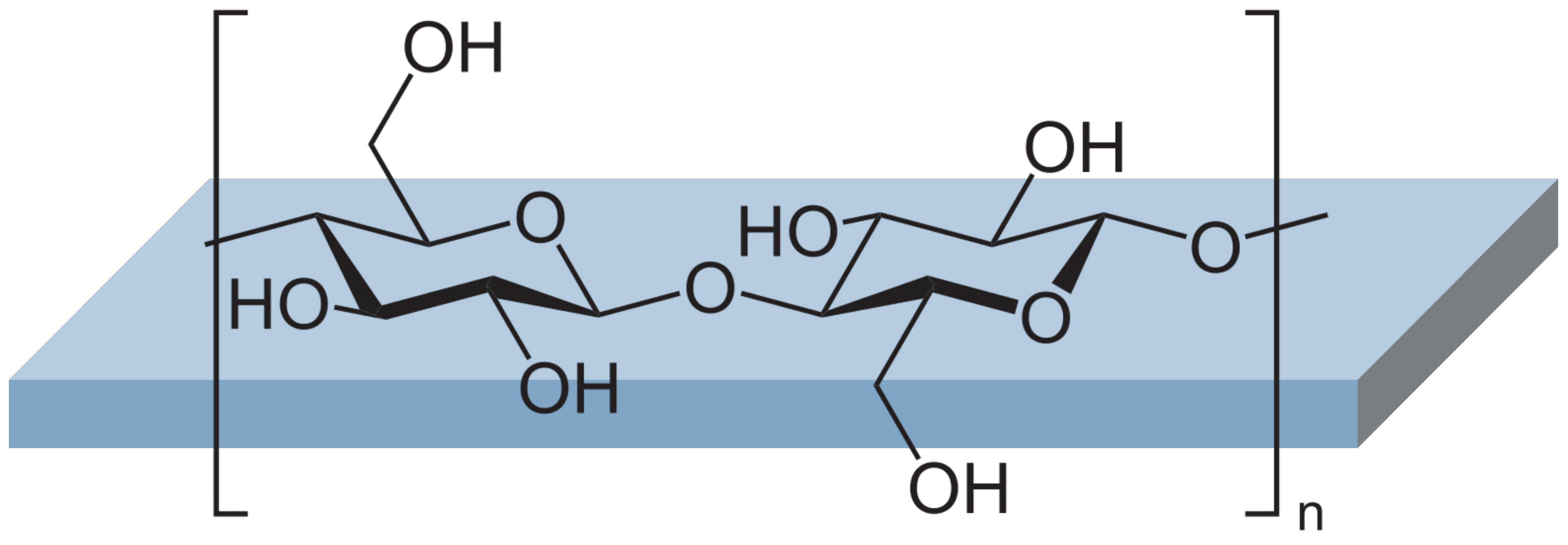
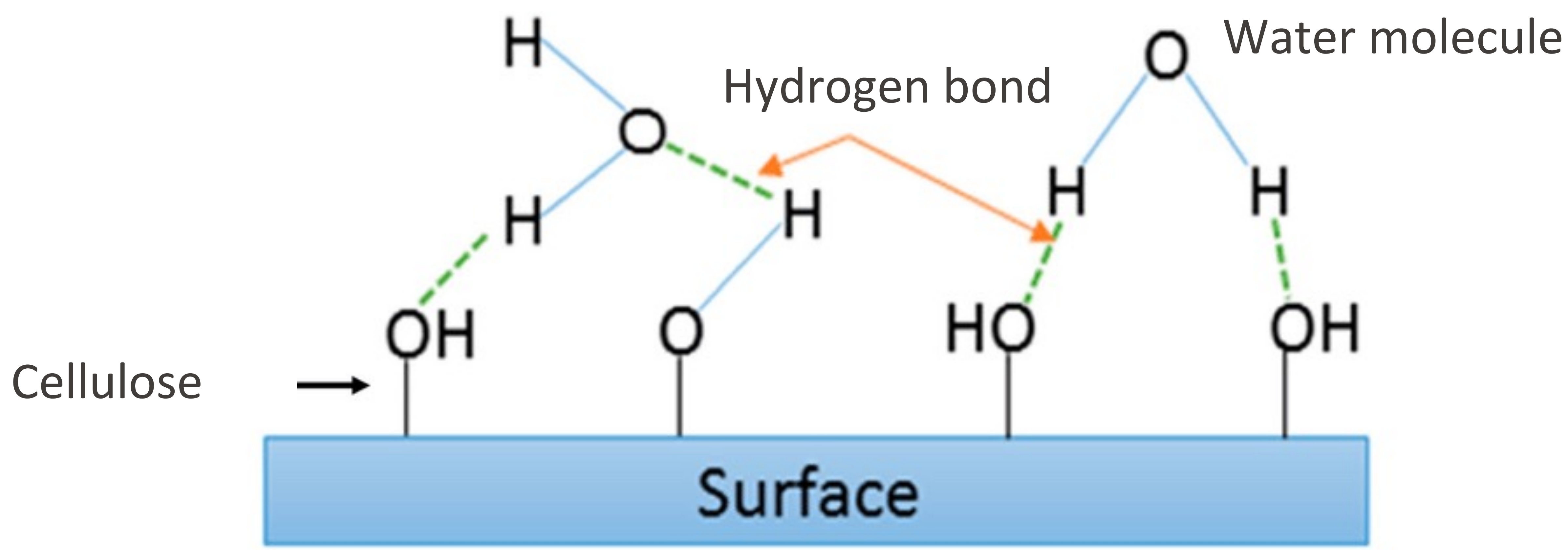
Hydrophilic *vs.* Hydrophobic Terminal Groups

Hydrophilic terminal groups

- OH (hydroxyl)



Cellulose on a surface



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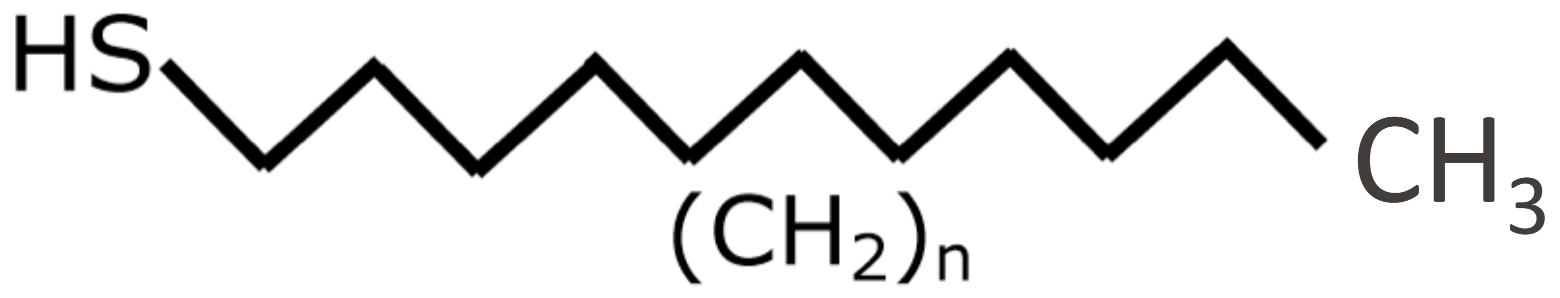
Non-polar

Minimal interactions with water due to low polarity



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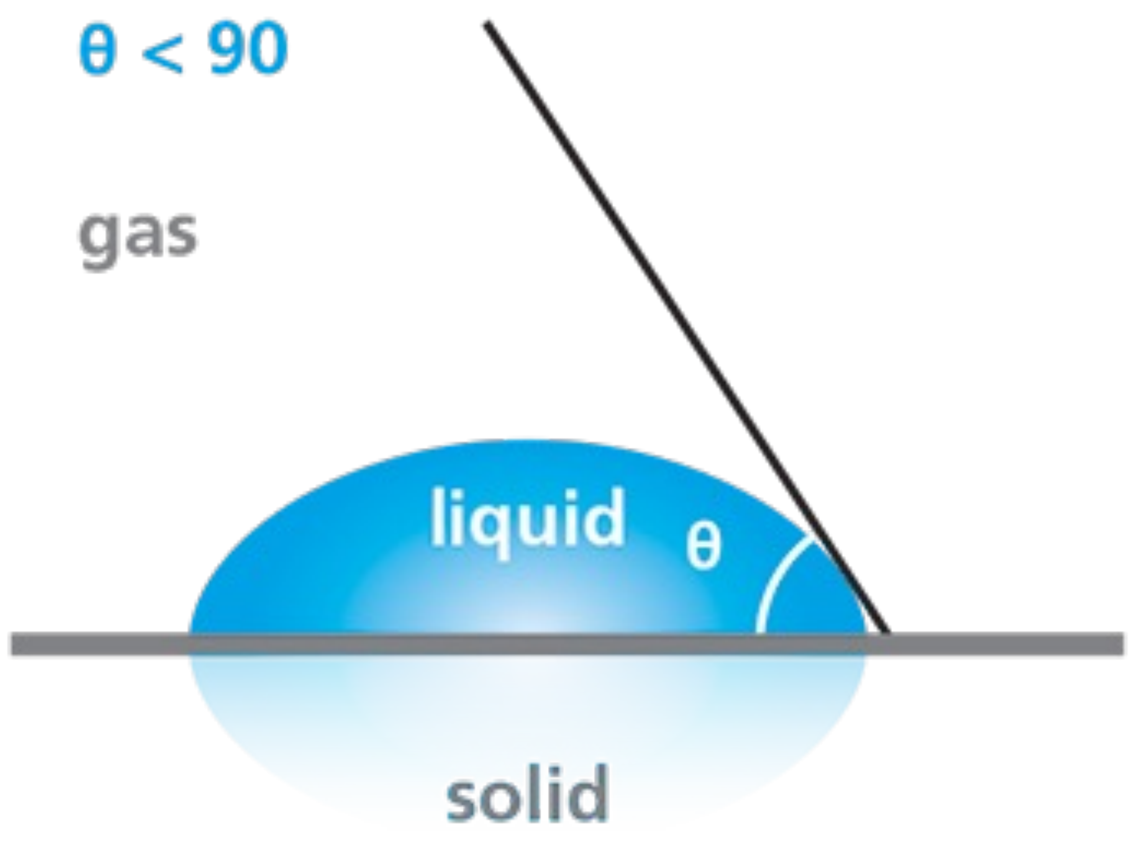

Hydrophobic terminal groups

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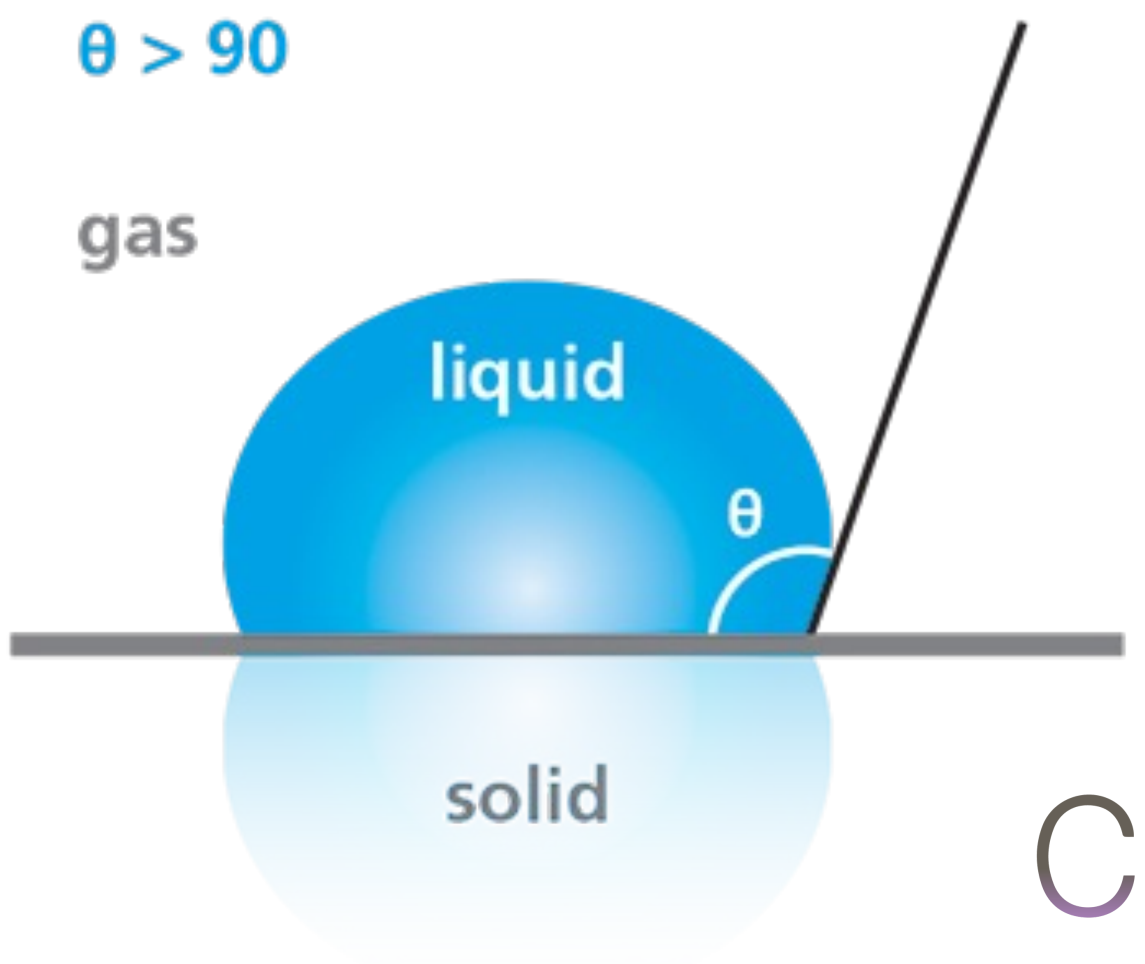
Contact angle



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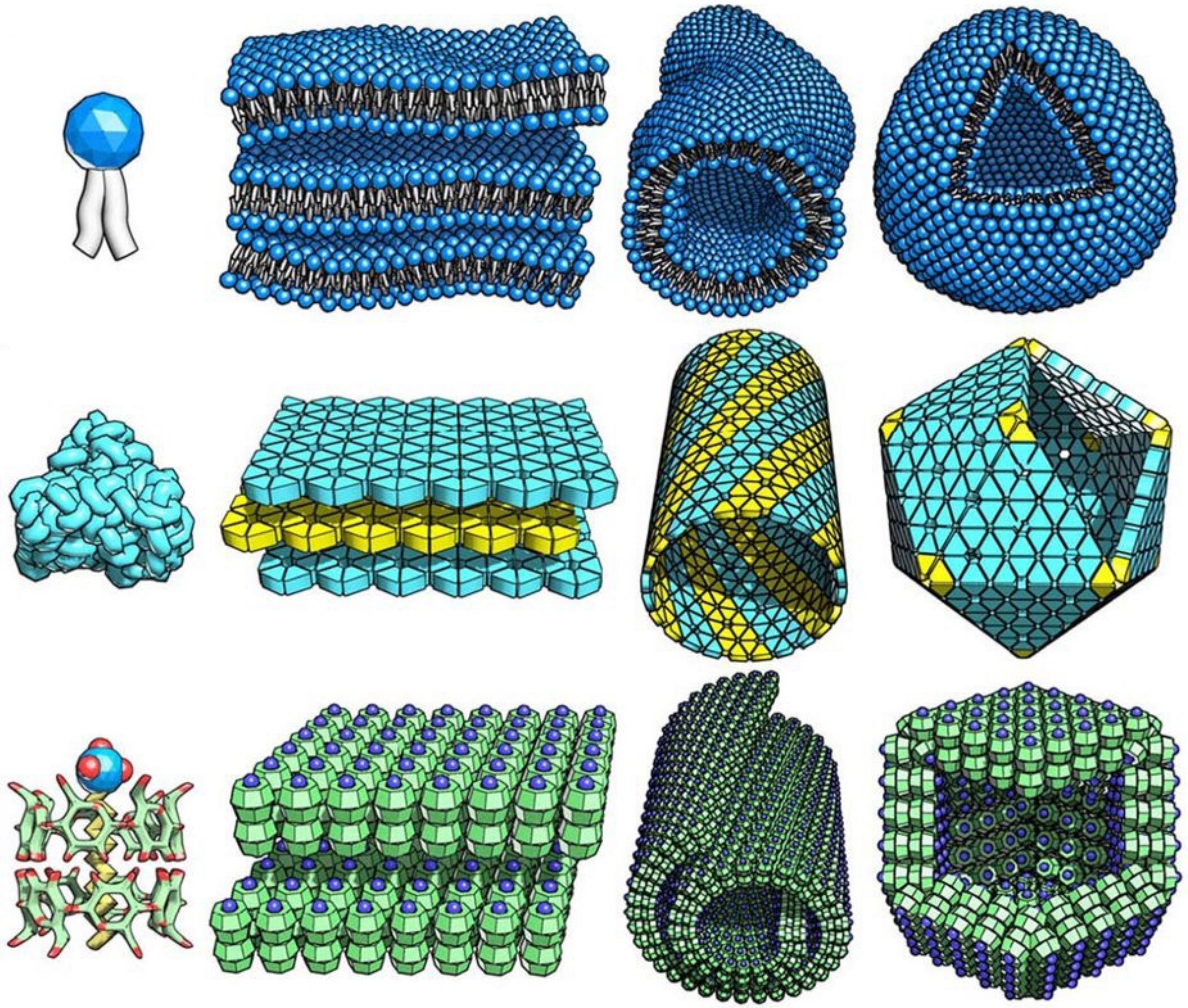
Self-Assembled Monolayers – What is Molecular Self-Assembly?

Self-assembly: process in which a disordered system of pre-existing components forms an organized structure or pattern due to specific interactions among the components themselves, without external direction

Lipids

Proteins

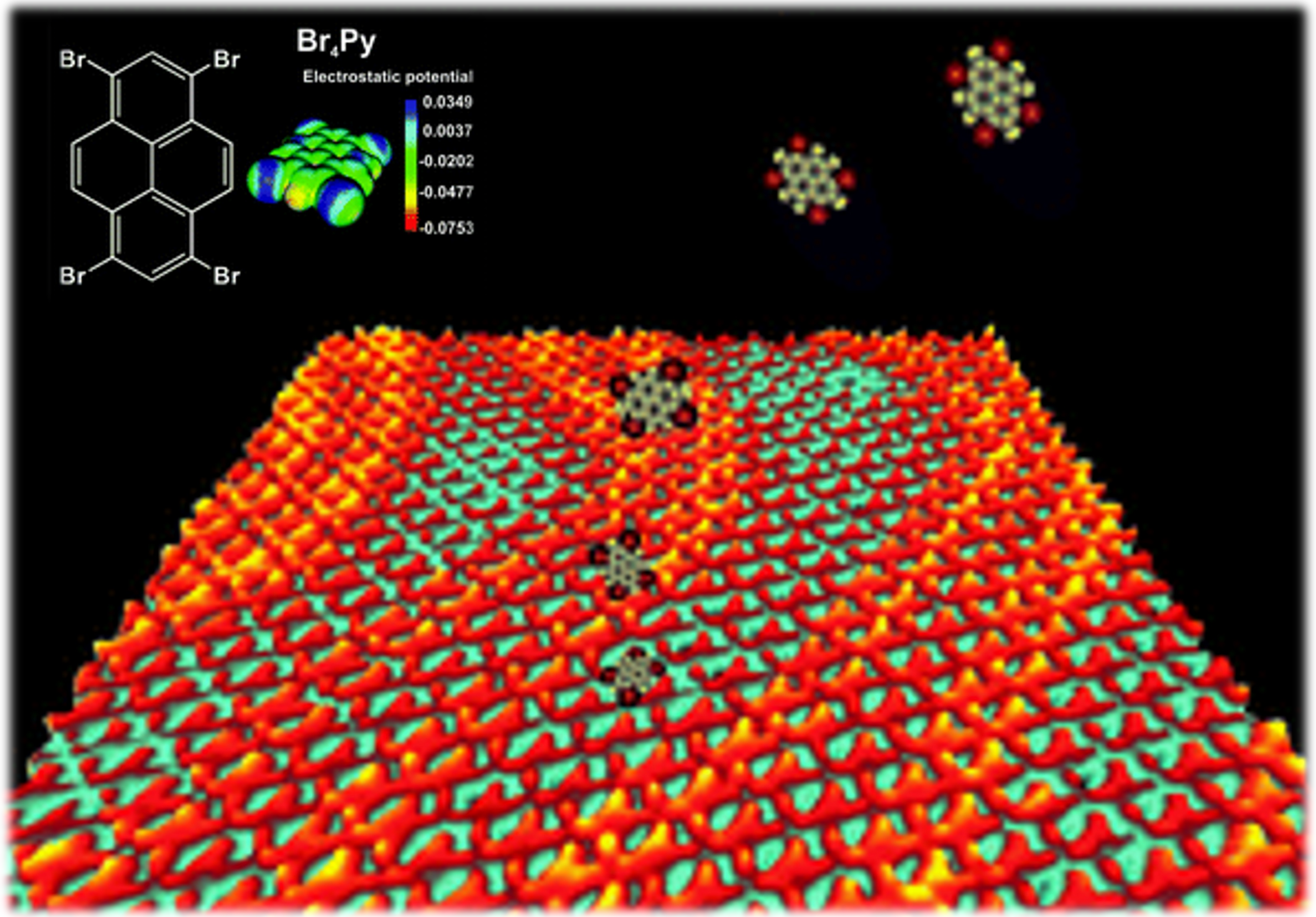
Surfactants



The ordered state forms as a system approaches equilibrium, reducing its free energy

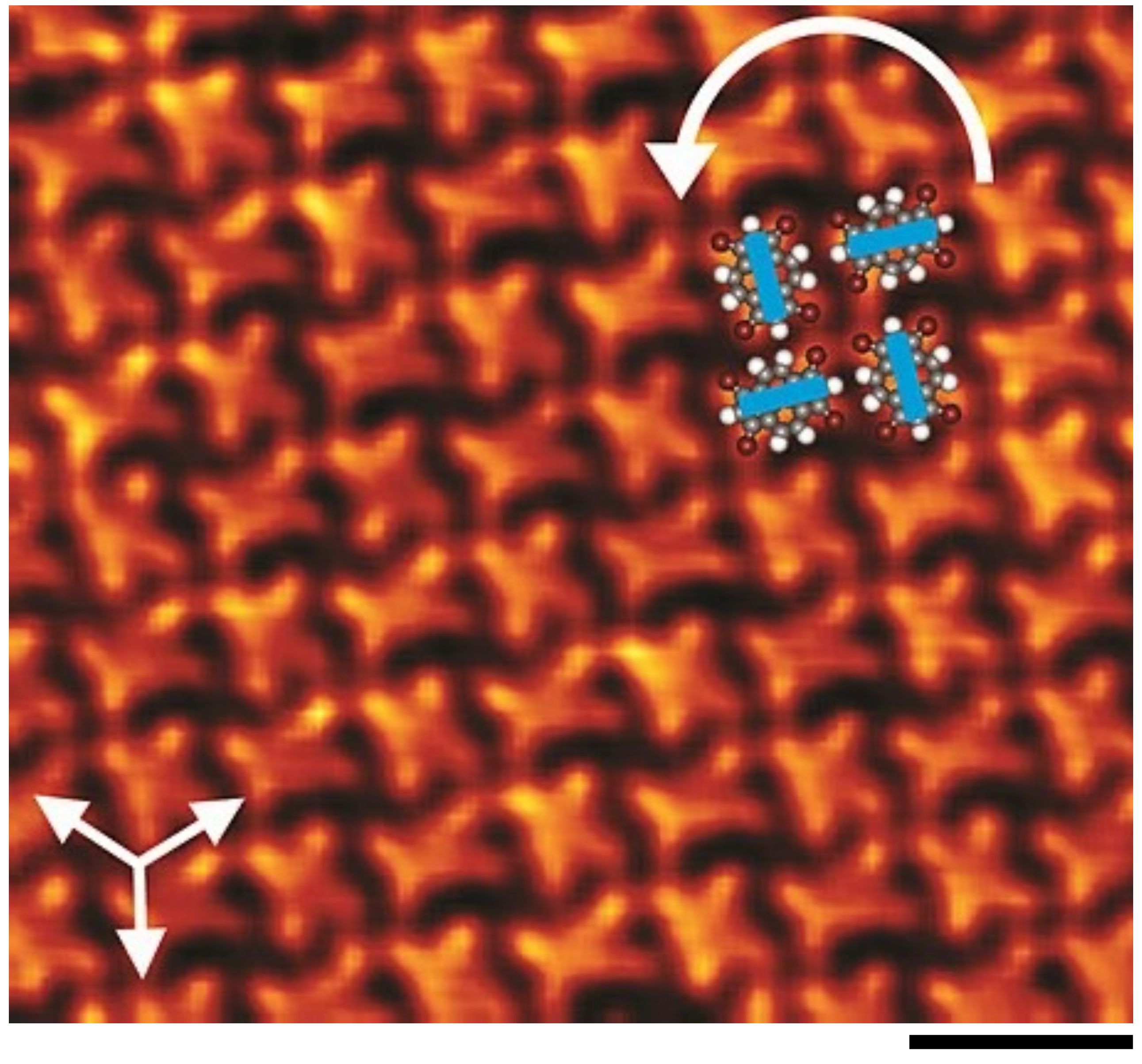
Source: Wikipedia

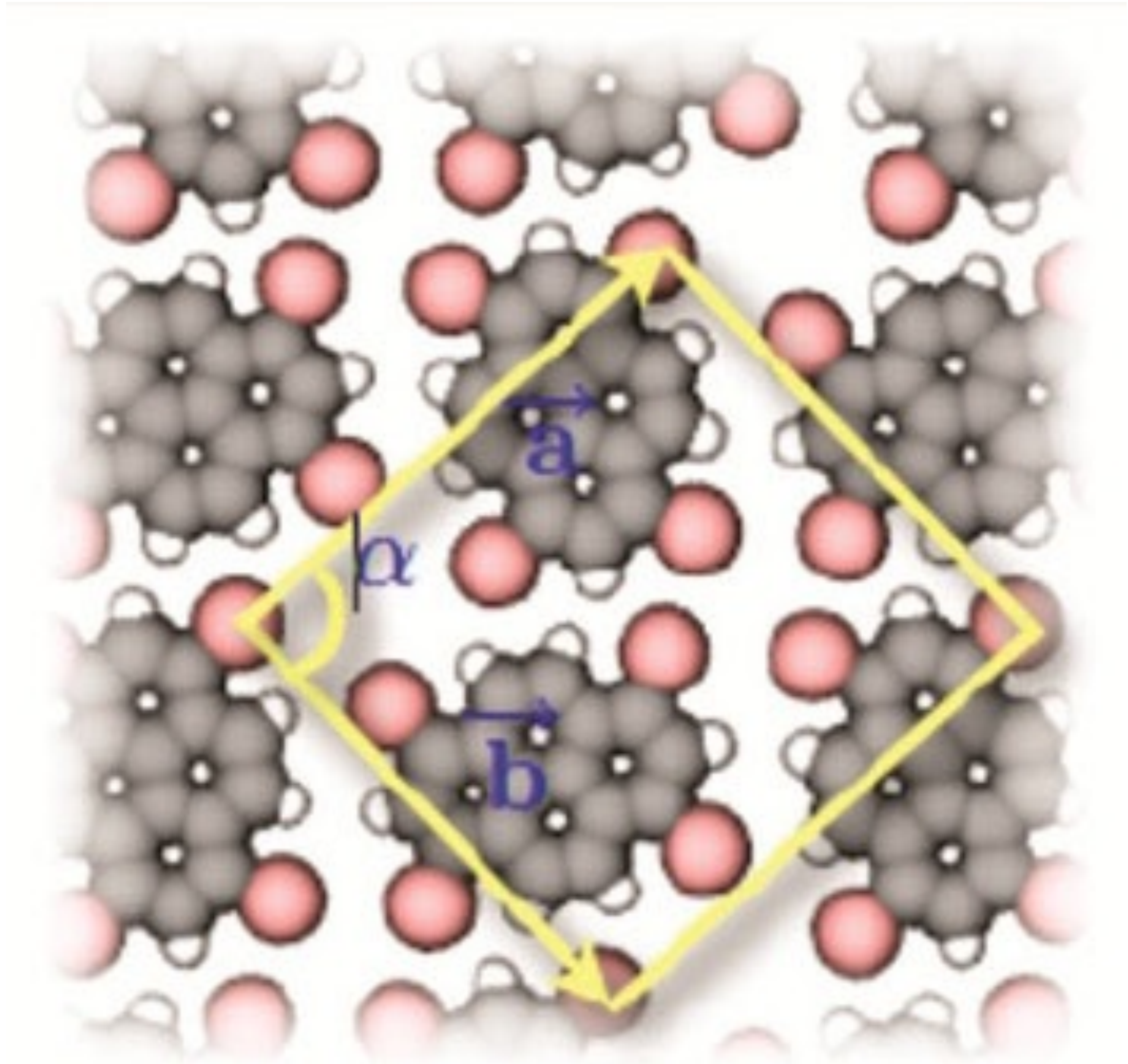
Molecular Self-Assembly of Br_4^- pyrene molecules on Au(111)



Self-assembly is the spontaneous and reversible organization of molecular units into ordered structures by non-covalent interactions – nanostructure builds itself

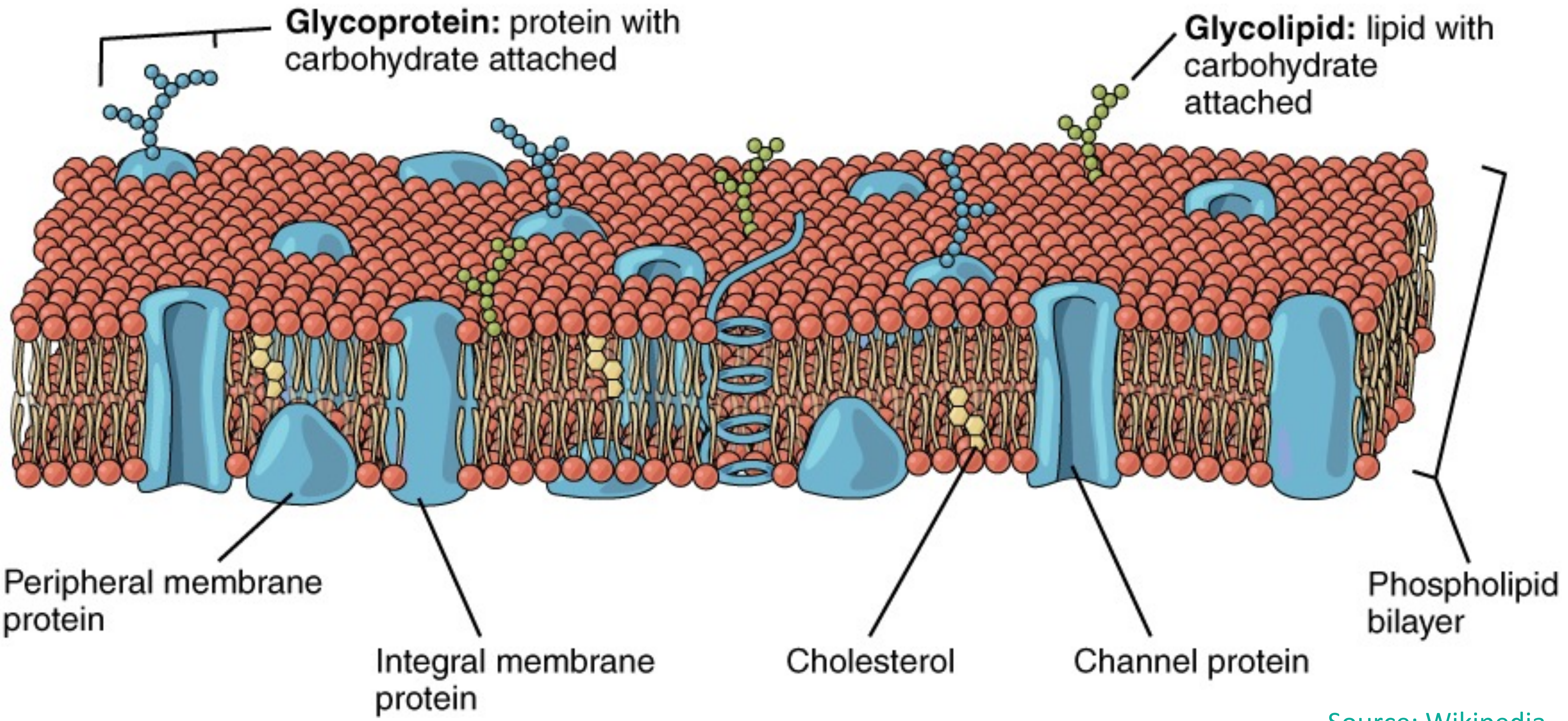
Scanning tunneling microscopy (STM) images





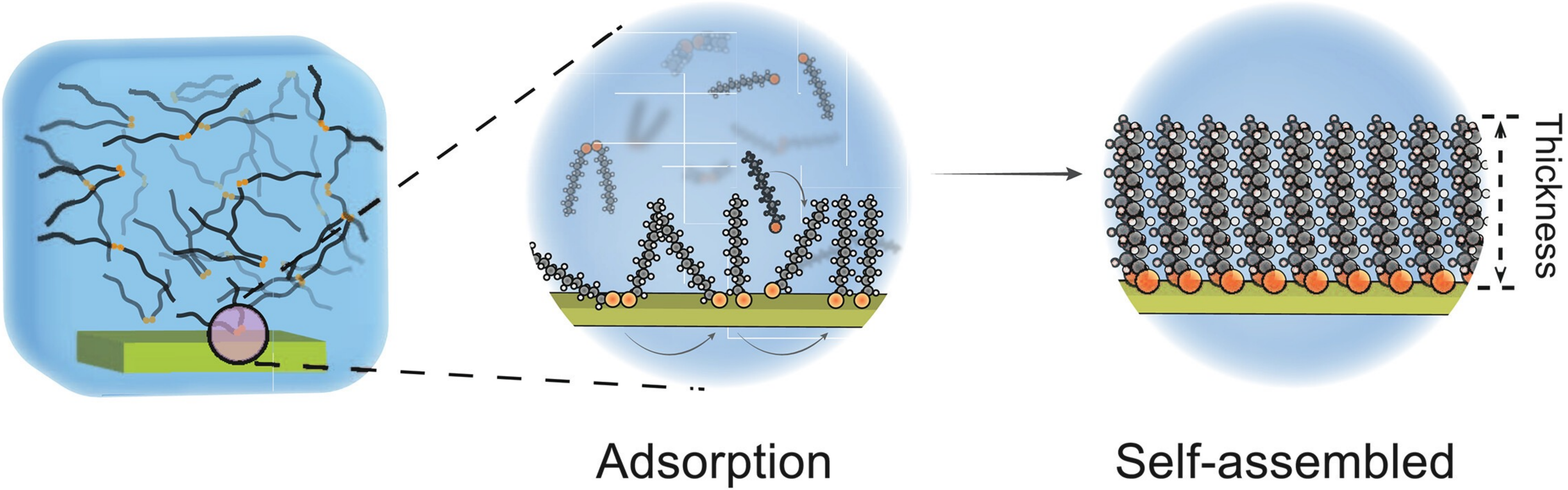
Pham *et al.* | Chem Commun. | 2014

Example of Spontaneous Self-Assembly in Nature



Source: Wikipedia

Self-Assembled Monolayers Form by Themselves



Chen et al. | Sci. Adv. | 2023

Technically simple “dip and rinse” process

No external driving force needed

Large choice of anchor groups, substrates, and functional groups

Easily modified and patterned

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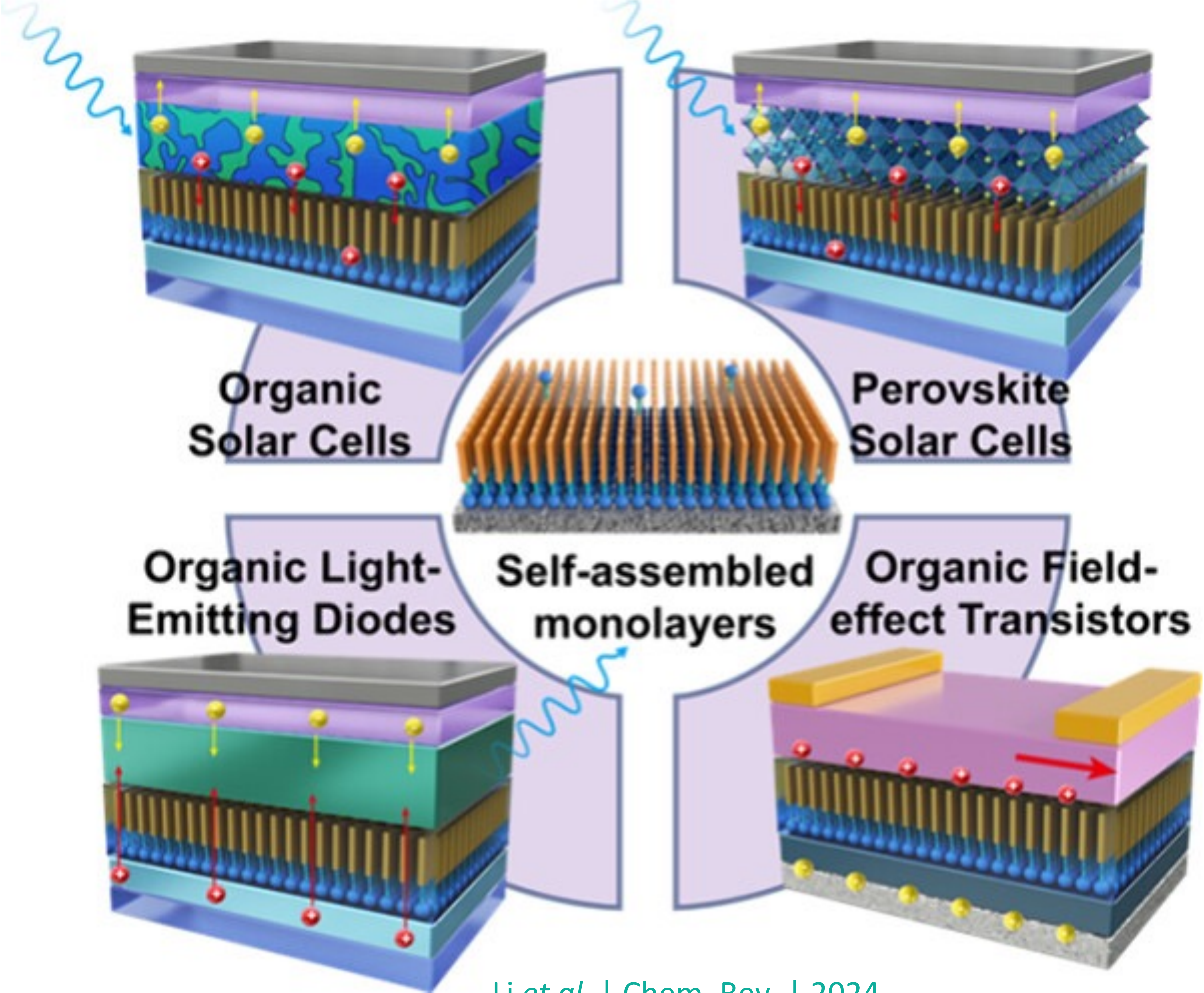
Application of Self-Assembled Monolayers: Electronic Devices



Li *et al.* | Chem. Rev. | 2024

Transparency
Diversity
Stability
Sensitivity
Selectivity
Surface passivation ability

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Factors Governing Self Assembly: Thermodynamics

Self-assembly (like a SAM forming on a surface) is driven by the system trying to lower its Gibbs free energy

–
Spontaneous

$$\Delta G = \Delta H - T\Delta S$$

Energy from forming or breaking bonds

↓

Molecules chemisorb to surface ➔ release anergy

↓

ΔH more negative

Atomic disorder

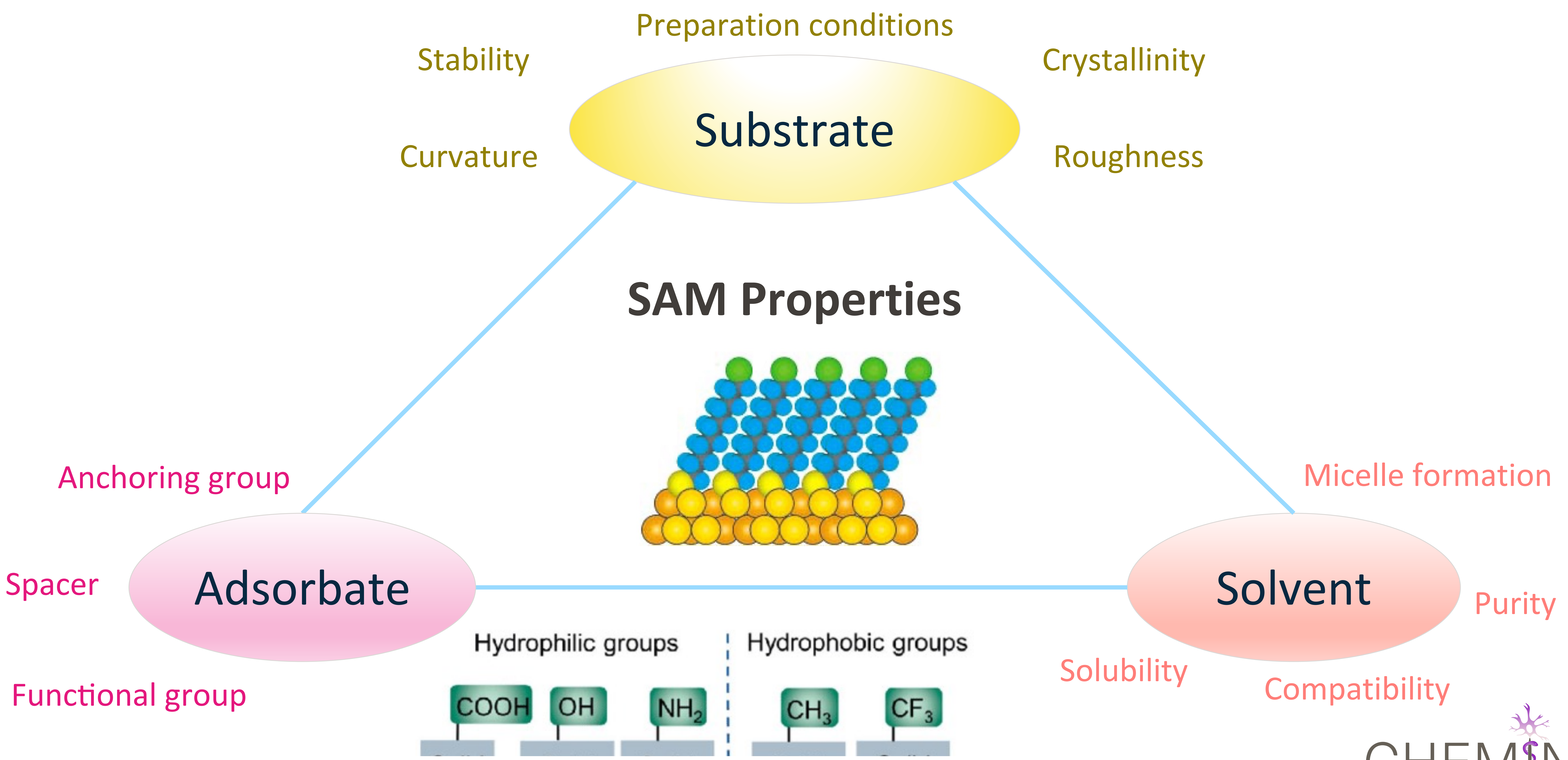
↓

Molecules line up into ordered monolayer

↓

ΔS more negative

Factors Governing Self Assembly



Key Takeaways

- We functionalize surfaces to modulate surface properties
- We can functionalize surfaces using self-assembled monolayers (SAMs)
- Molecular self assembly allows assembly from individual components
- Thermodynamics drive self assembly
- The Adsorbate, substrate, and solvent drive self assembly

Overview of Surface Binding Chemistries

Adsorbate	Substrate
Thiols: R-SH	**Au**, **Ag**, Cu, Pt, Pd, Ru, Hg, AuAg, AuCu,Ni, Ir, Zn, ZnSe, CdSe, CdS, CdTe, Ge
Disulfides: R-S-S-R'	**Au**, **Ag**, Pd
Silanes: R-$SiCl_3$, R-$Si(OR')_3$ R-$Si(CH_3)_2Cl$	**SiO_2**, TiO_2, Au
Phosph(on)ates: R-PO_4H_2 R-PO_3H_2	**Al_2O_3, TiO_2,** Nb_2O_5, Ta_2O_3, ZrO_2

Adsorbate	Substrate
Ammonium : R-NH_3^+ R-$N(CH_3)_3^+$ R_2-$N(CH_3)_2^+$	**Mica**
Carboxylic acid: R-COO^-/R-COOH	**Al_2O_3**, Ni, Ag, Ti/TiO_2
Alkyne: R-C≡CH	**Si(111):H**
Alcohols: R-OH	**Si:H**

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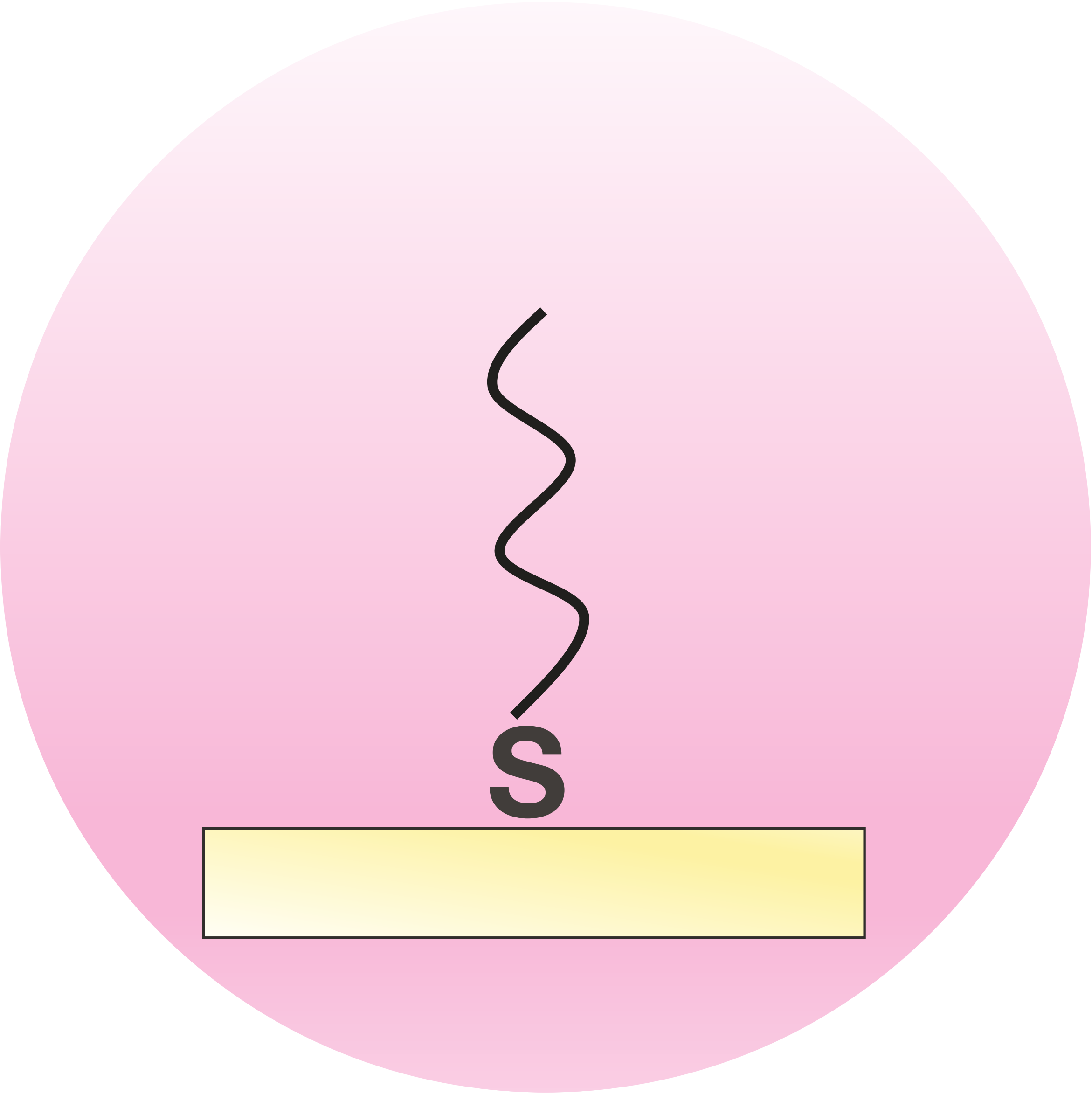
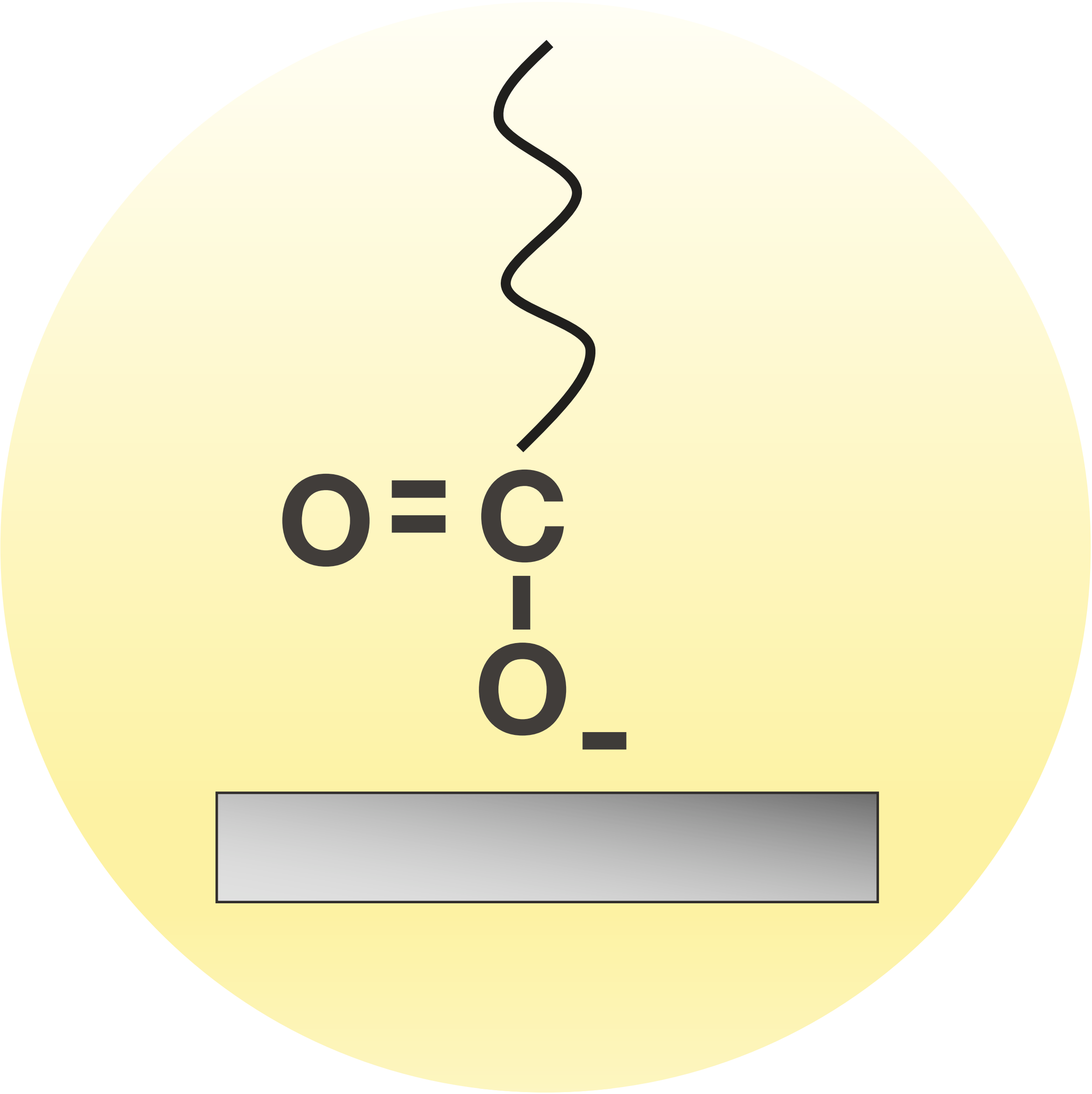
What We Will Cover in Class

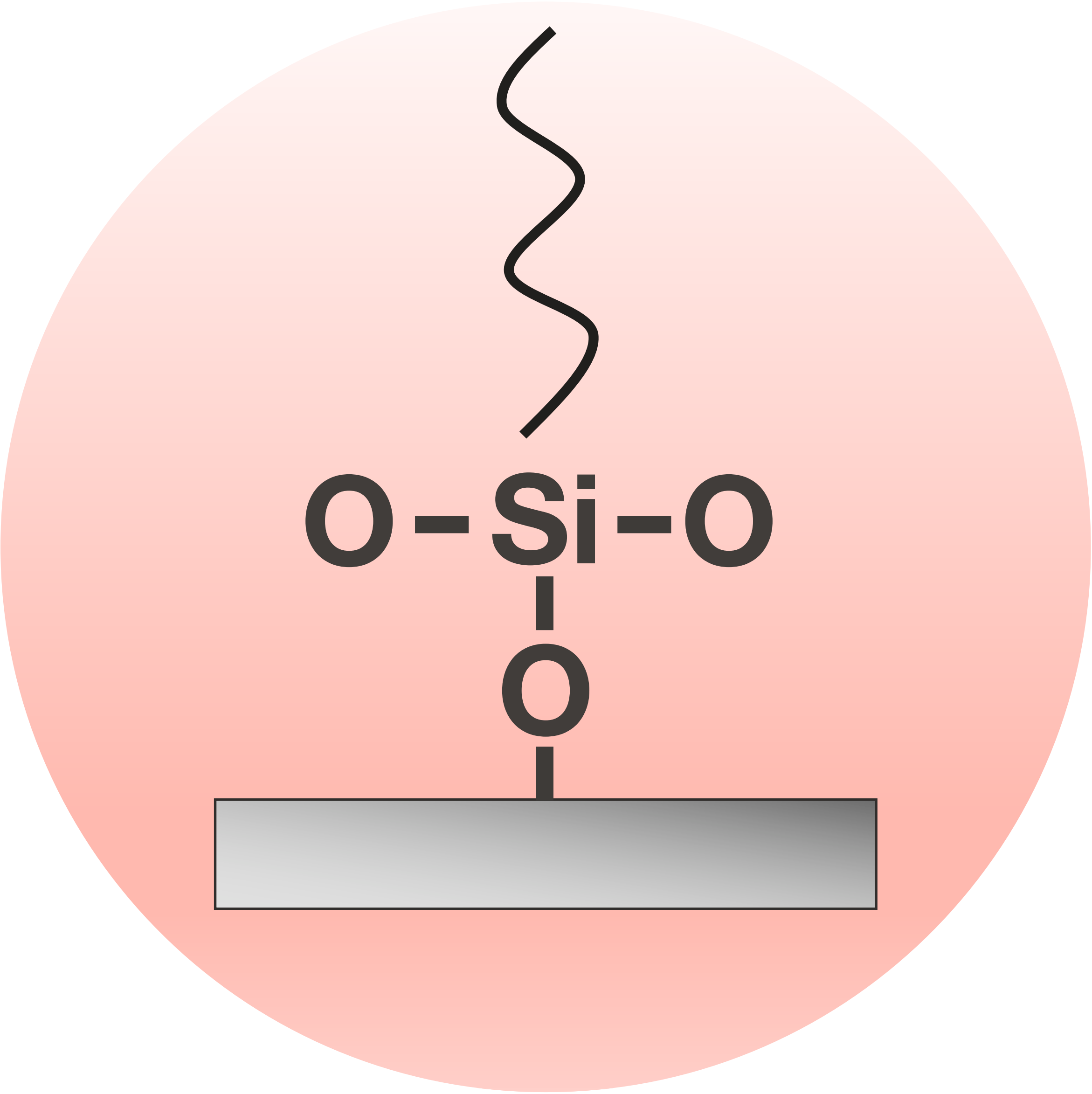
Surface chemistry



Thiol-based chemistry
Metallic surfaces



Carboxylic acids
Metal oxide surfaces

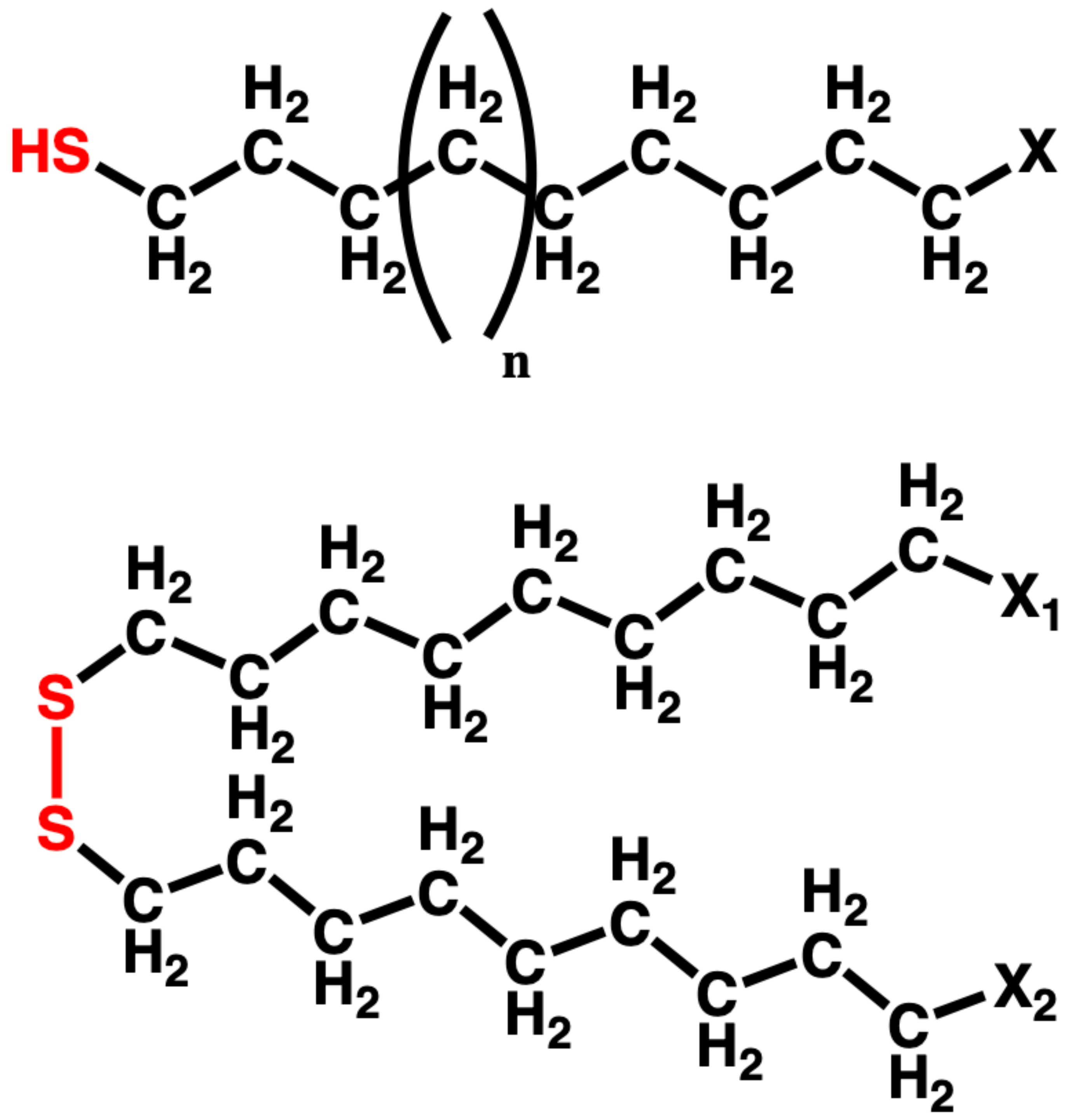


Silane, Phosphonates
Metal oxide surfaces

Alkanethiol Molecules

Substrate

Au
Ag
Hg
Cu
Pt
Pd



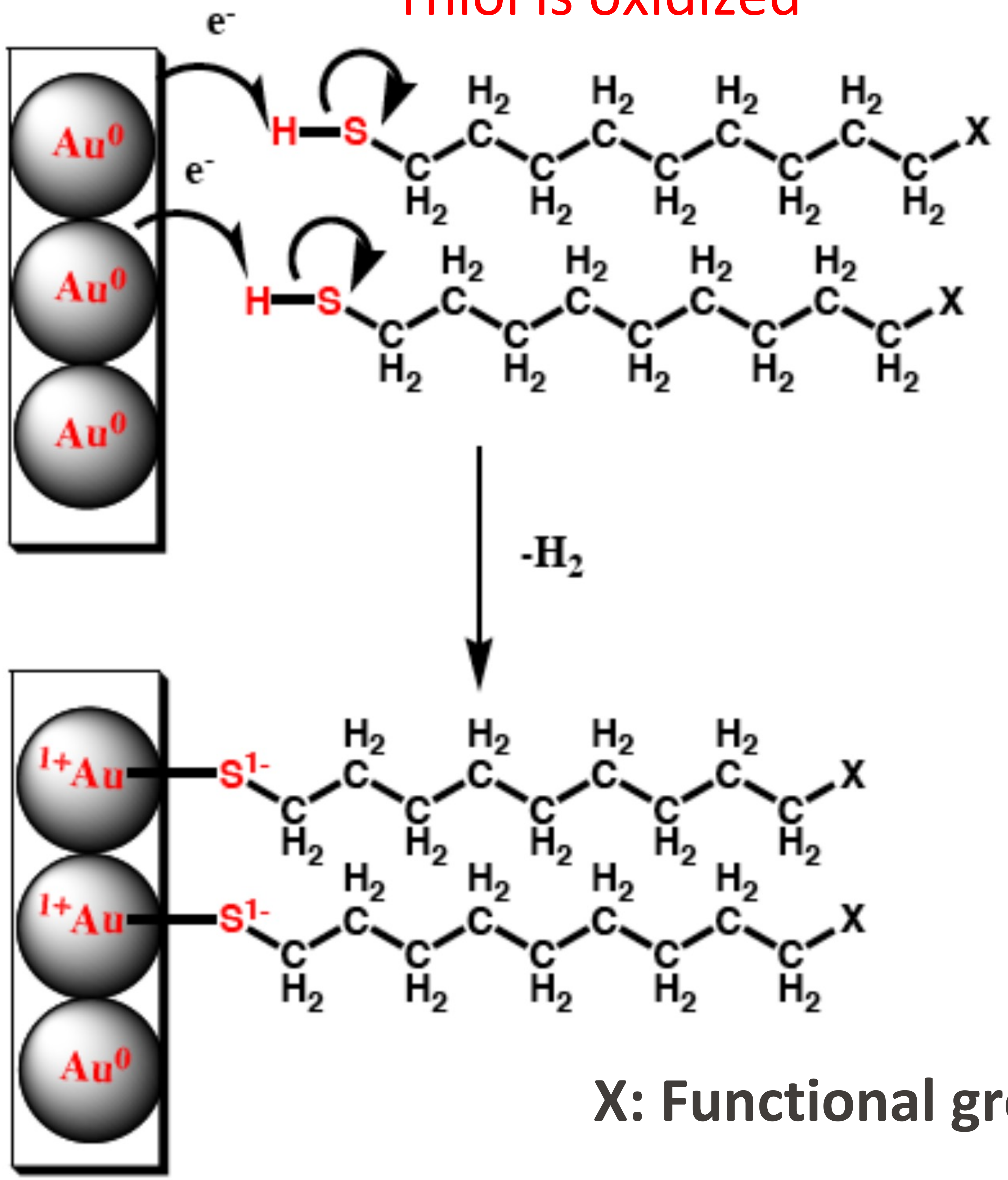
X: Functional group

Alkane	Alkanethiol	Formula
methane	methanethiol	$CH_3–SH$
ethane	ethanethiol	$CH_3–CH_2–SH$
propane	propanethiol	$CH_3–CH_2–CH_2–SH$
hexane	hexanethiol	$CH_3–(CH_2)_4–CH_2–SH$

Alkanethiols Chemisorption Mechanism

Thiol is oxidized

Gold surface is reduced



Au-S bond stabilizes the system by decreasing the ΔG

X: Functional group

*Identical mechanism for thiols and disulfides

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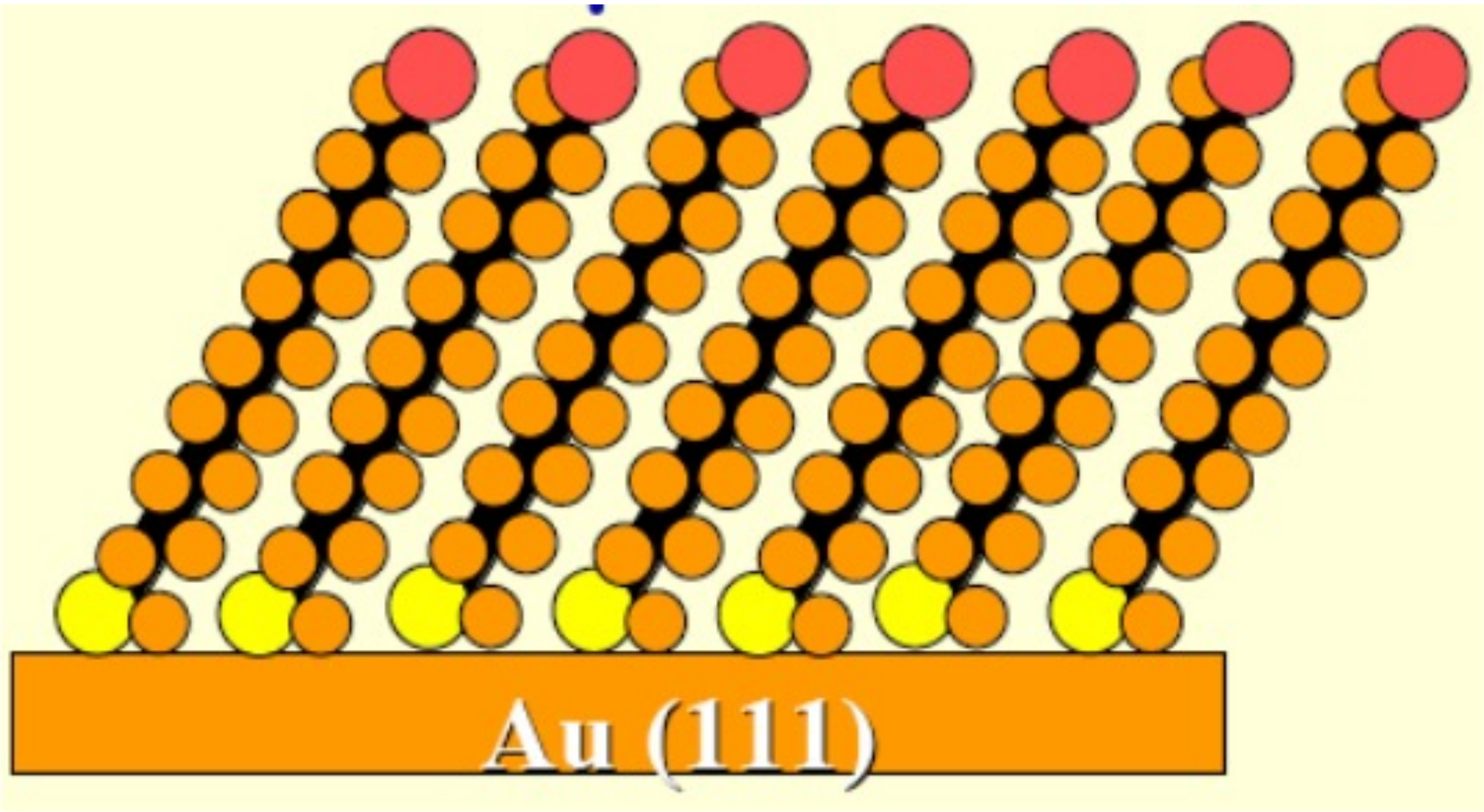
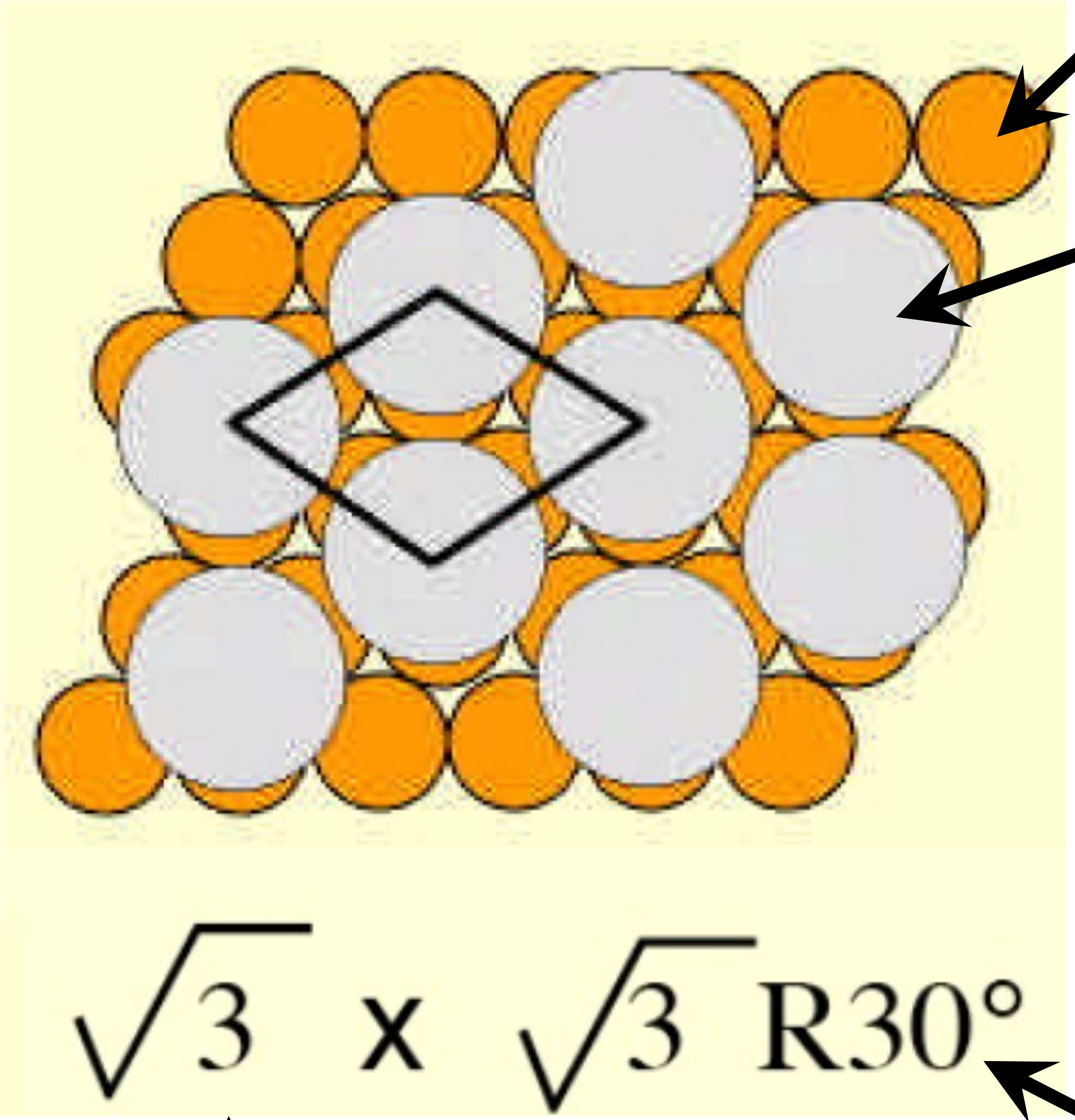
Alkanethiols Adopt Specific Arrangements on Surfaces



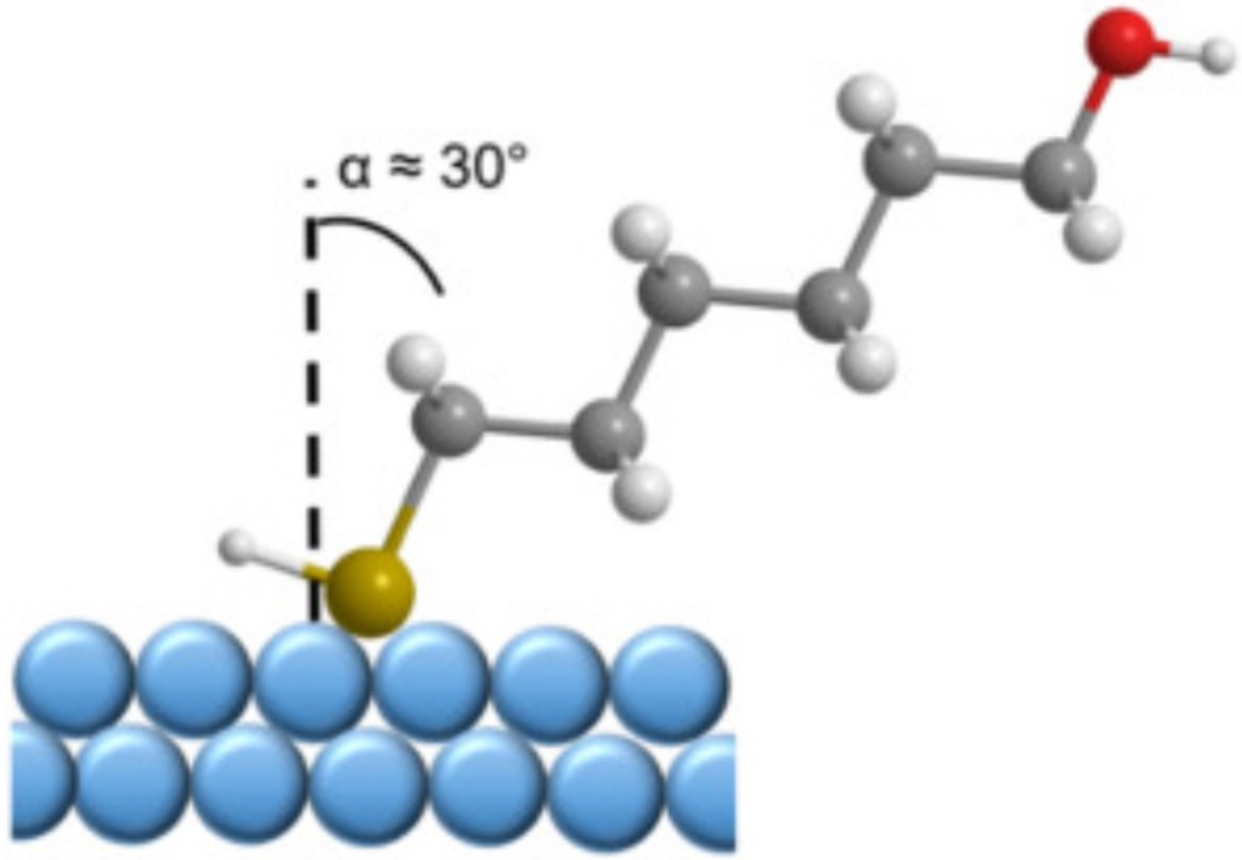
Face-centered cubic (FCC)
Close-packed hexagonal lattice
d_{Au} ~0.29 nm

Alkanethiols form covalent bond with the gold atoms and adopt a superlattice structure

Au



Alkanethiol



Rotation
Alignment of adsorbed lattice relative to gold lattice

Every 3rd Au atom is a binding site

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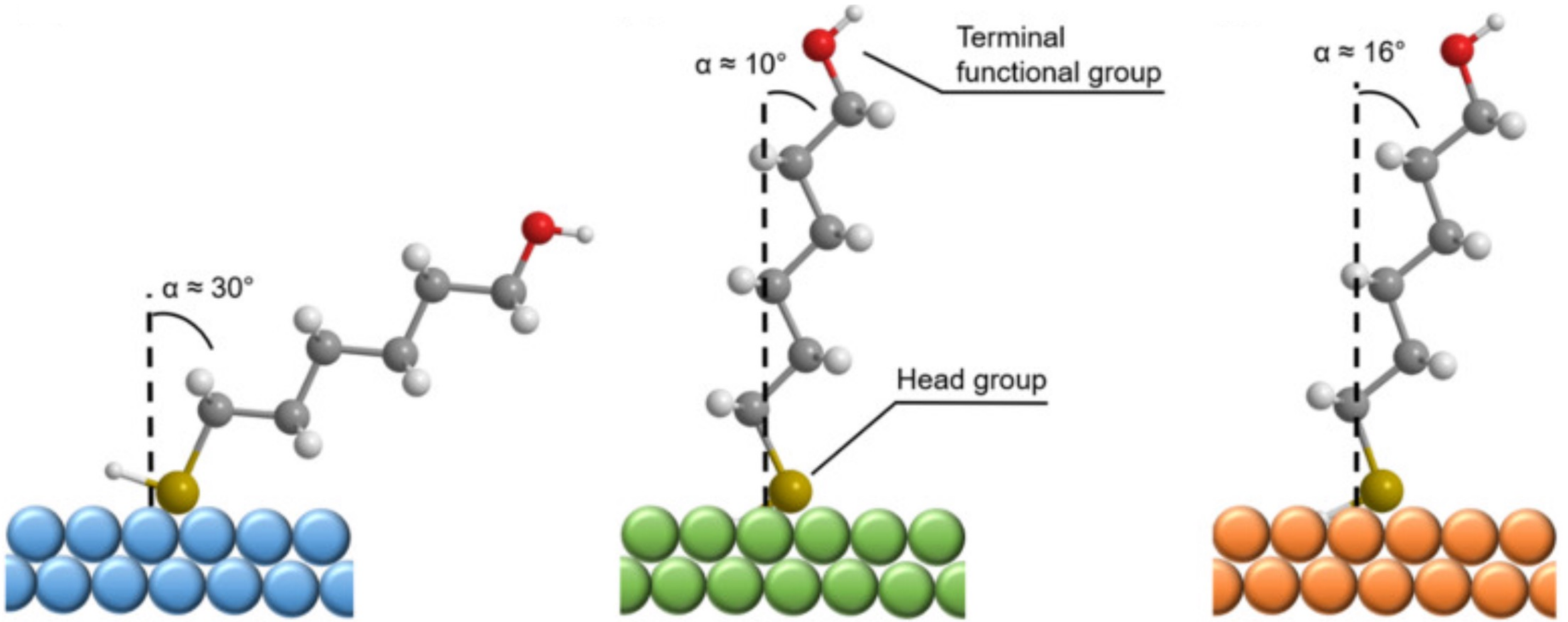
Alkanethiols – Effect of Substrate Material

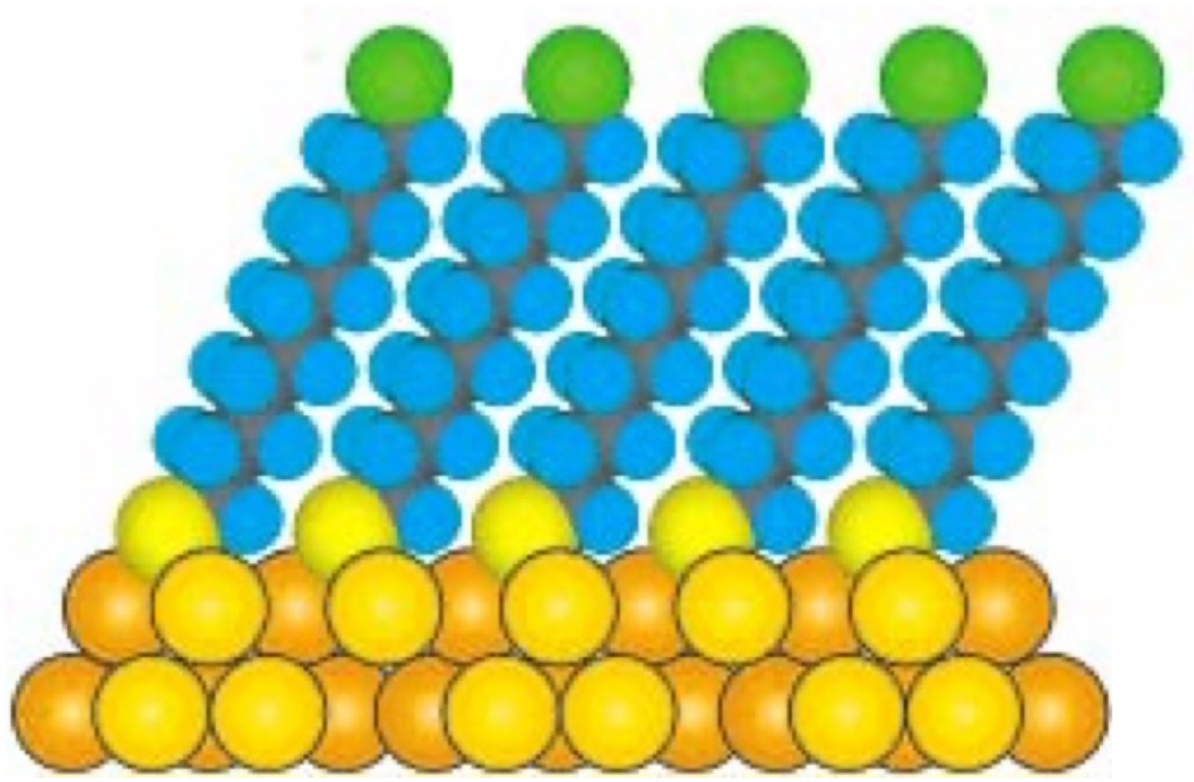


Au substrate **Ag substrate** **Pd substrate**

Lattice mismatch between the gold substrate and the natural packing density of alkanethiols, requiring higher tilt to achieve stable van der Waals interactions

Güvener *et al.* | Fundamentals of Sensor Technology | **2023**

Alkanethiols – Effect of Spacer Group



Functional group

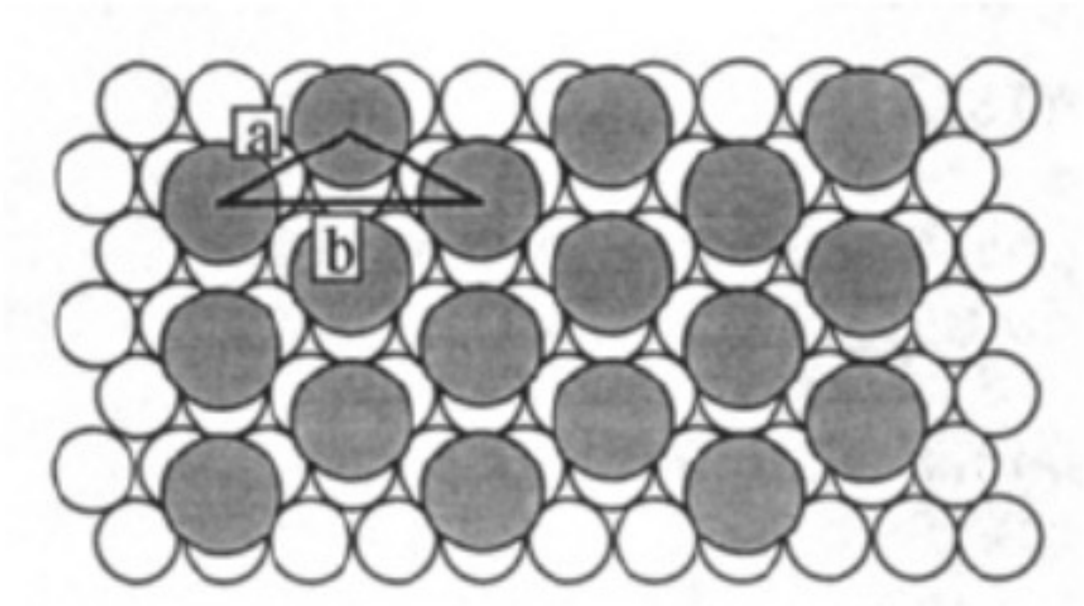
Spacer group

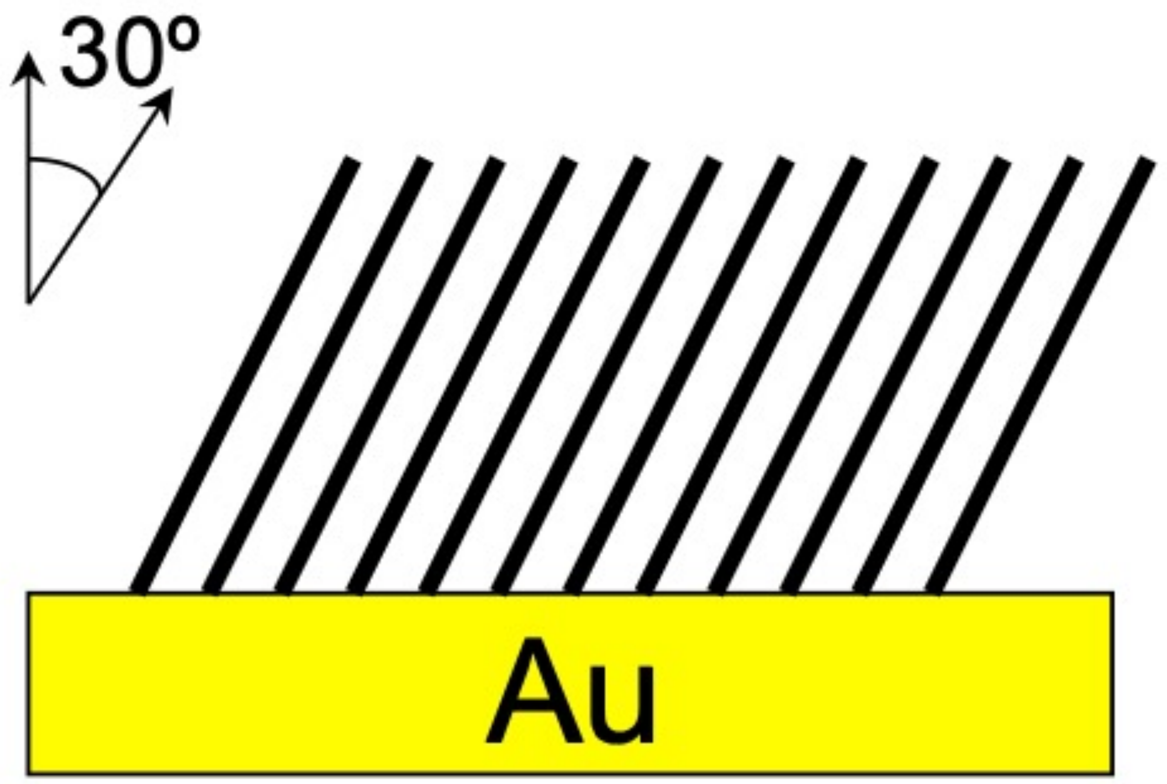
Anchor group

Hydrocarbon *vs.* fluorinated spacer group

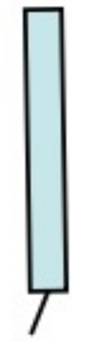
$CH_3(CH_2)_9SH$

Chain diameter: 4.3Å



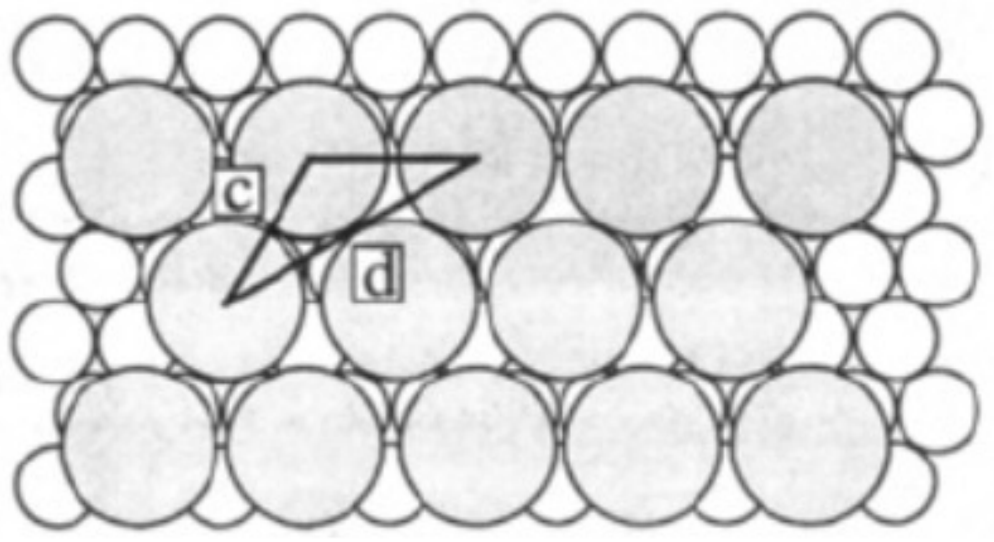


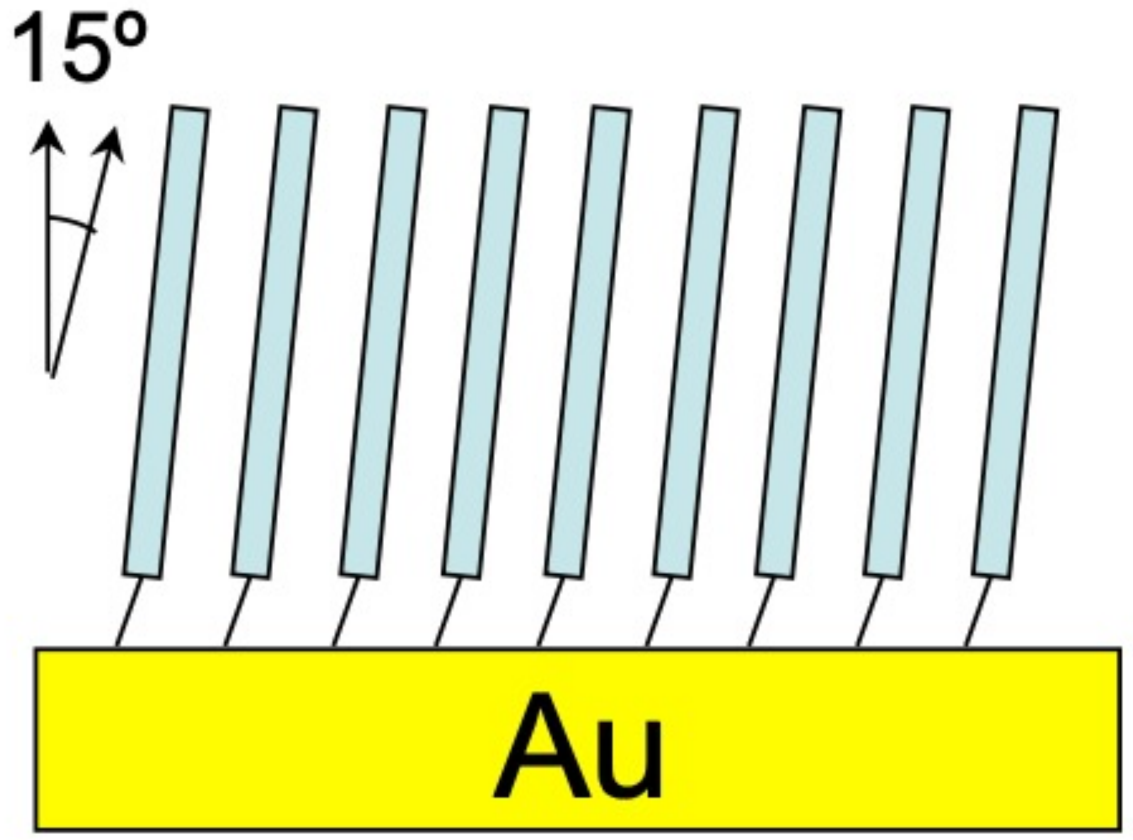
Different packing due to different sizes of the chains



$CF_3(CF_2)_7(CH_2)SH$

Chain diameter: 5.7Å

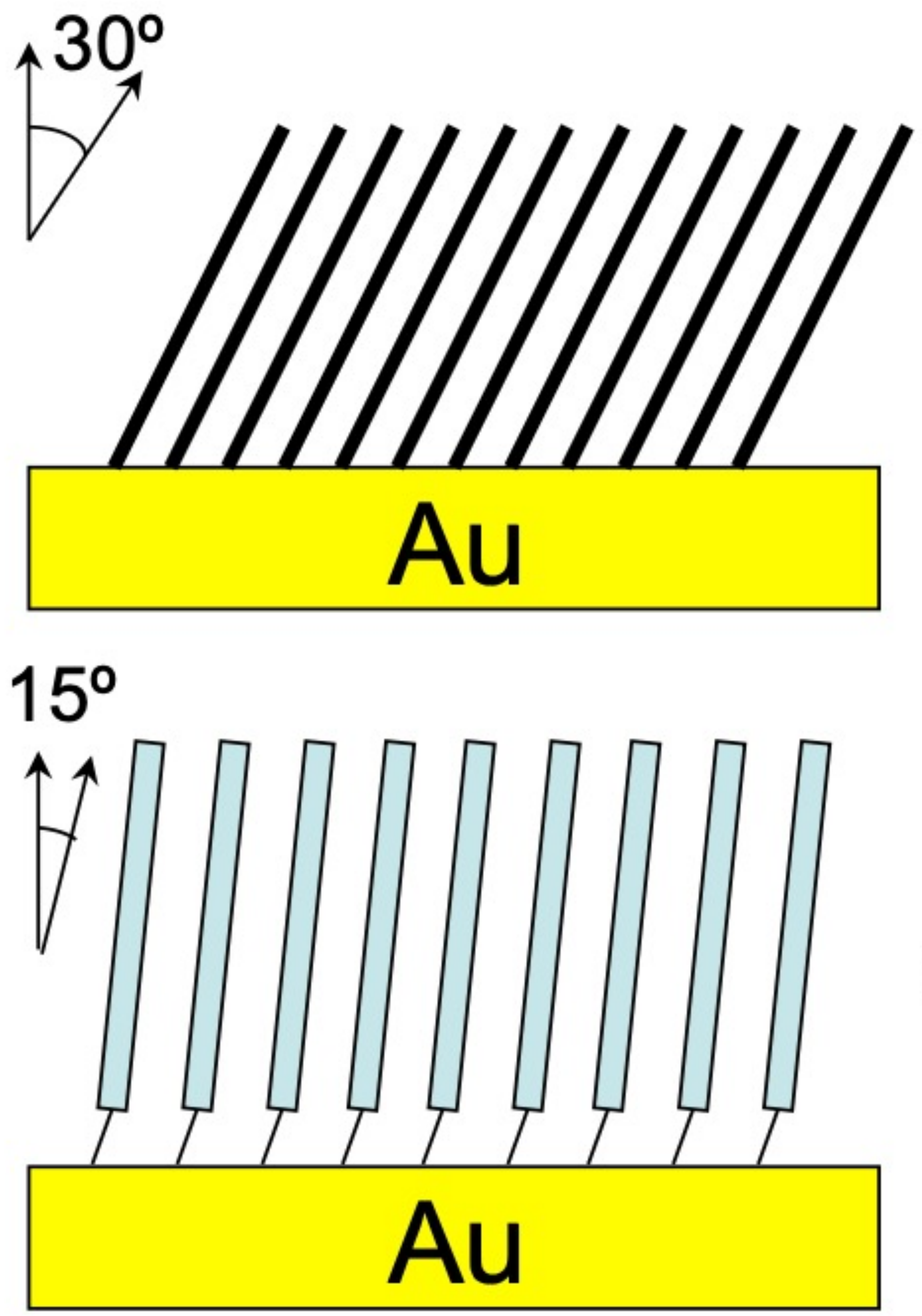




Alves & Porter | *Langmuir* | 1993

Why Packing Density/Geometry/Interactions Matter

Different packing density ➔ Different molecular interactions ➔ Different macroscopic behavior



$CH_3(CH_2)_9SH$

$CF_3(CF_2)_7(CH_2)SH$

Surface energy - exposes different chemistry at interface

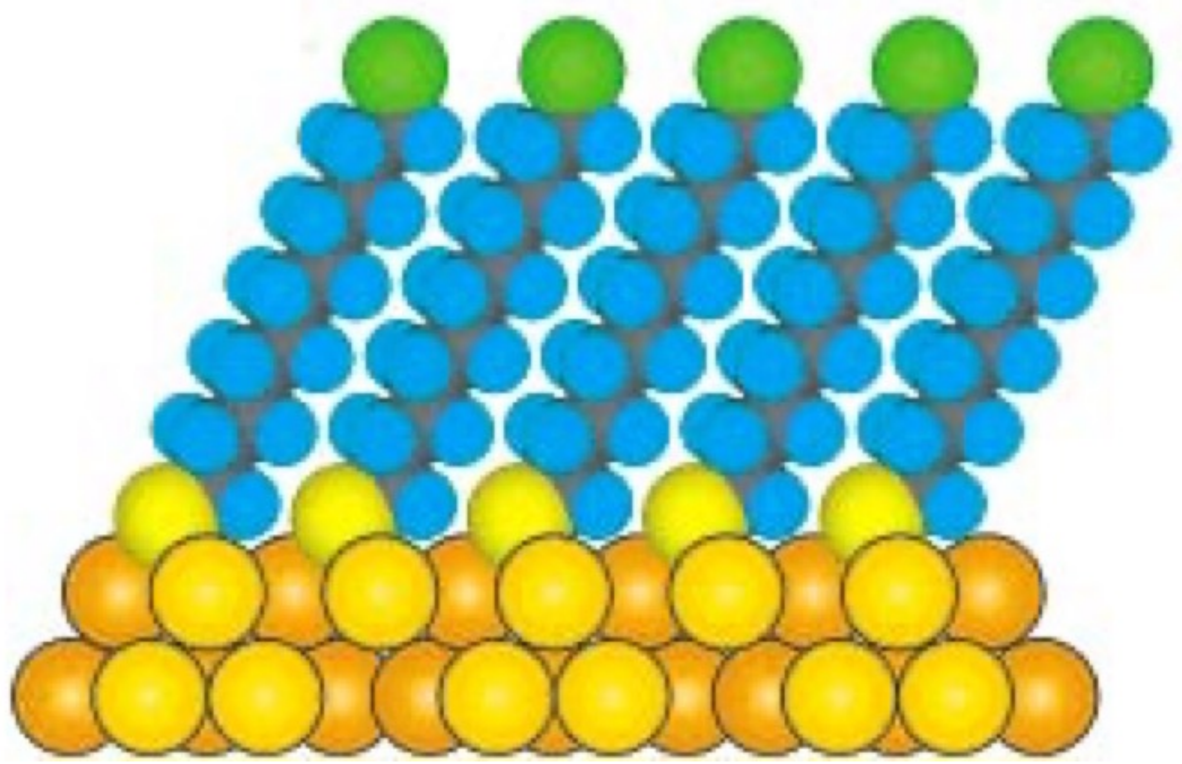
SAM stability based on lateral interactions

Barrier properties - electron transfer, permeability, *etc.*

Thickness of the monolayer

Alves & Porter | *Langmuir* | 1993

The Pros and Cons of Alkanethiols



Metal surface *(e.g.,* Au*)*

Selective bonds formed with noble metal surfaces

Many different functional end groups commercially available

Easy to prepare (Au chemically inert)

Very well studied (order and packing well known)

Patterning is possible

Mobile – instability over time under ambient conditions

Sensitivity to oxidation leads to low stability

Do not chemisorb on most technologically important materials

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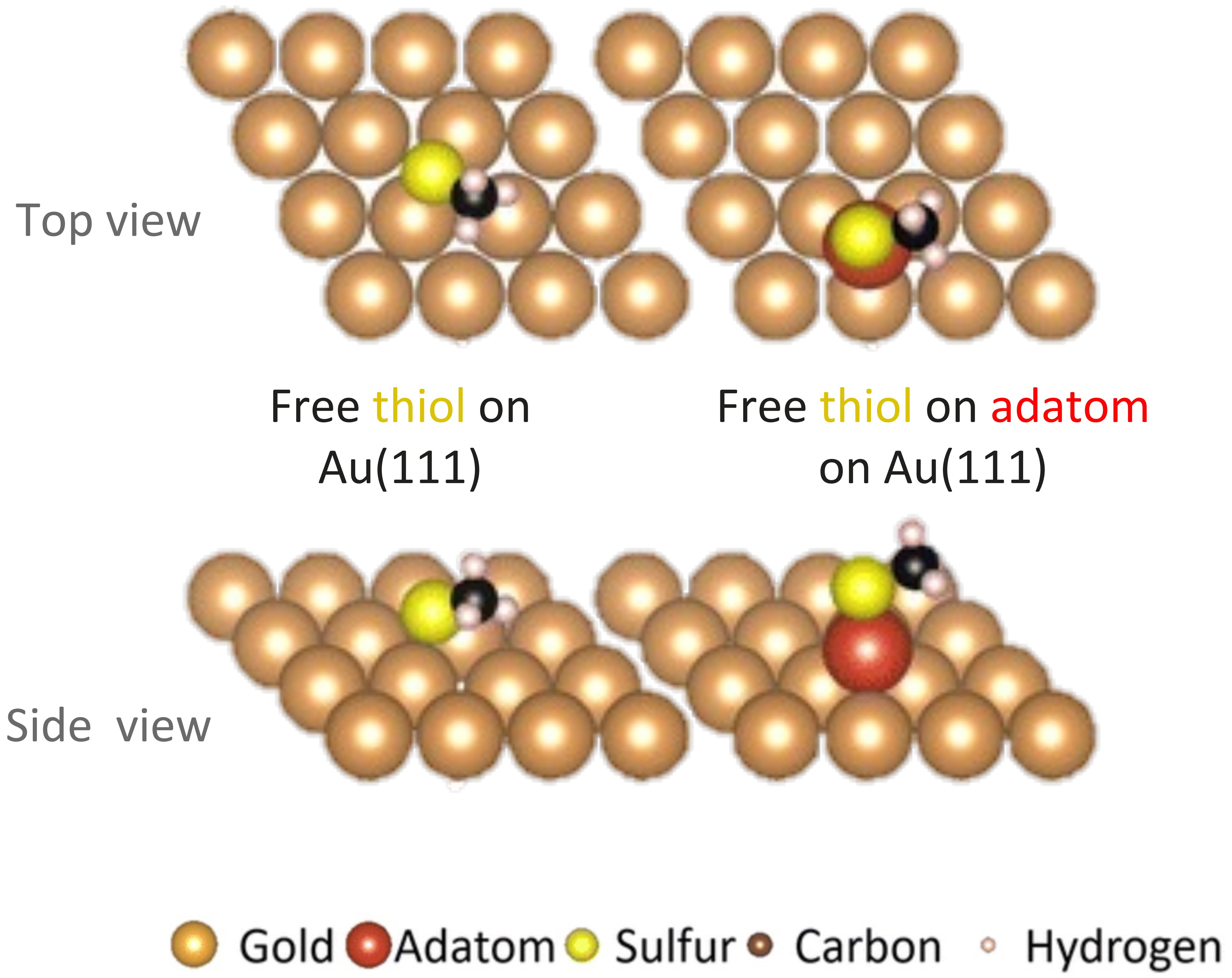
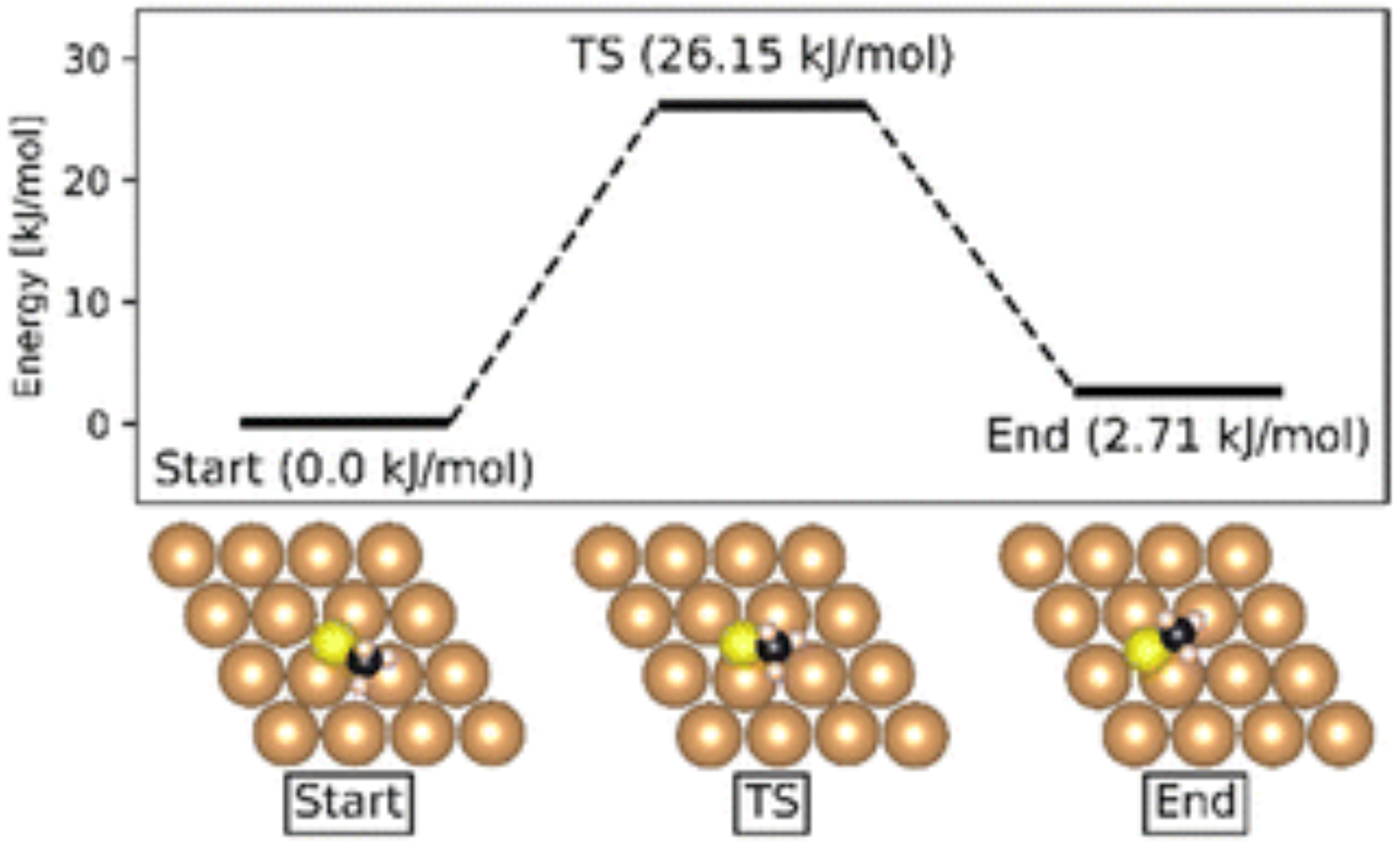
Mobility of Thiols on Au

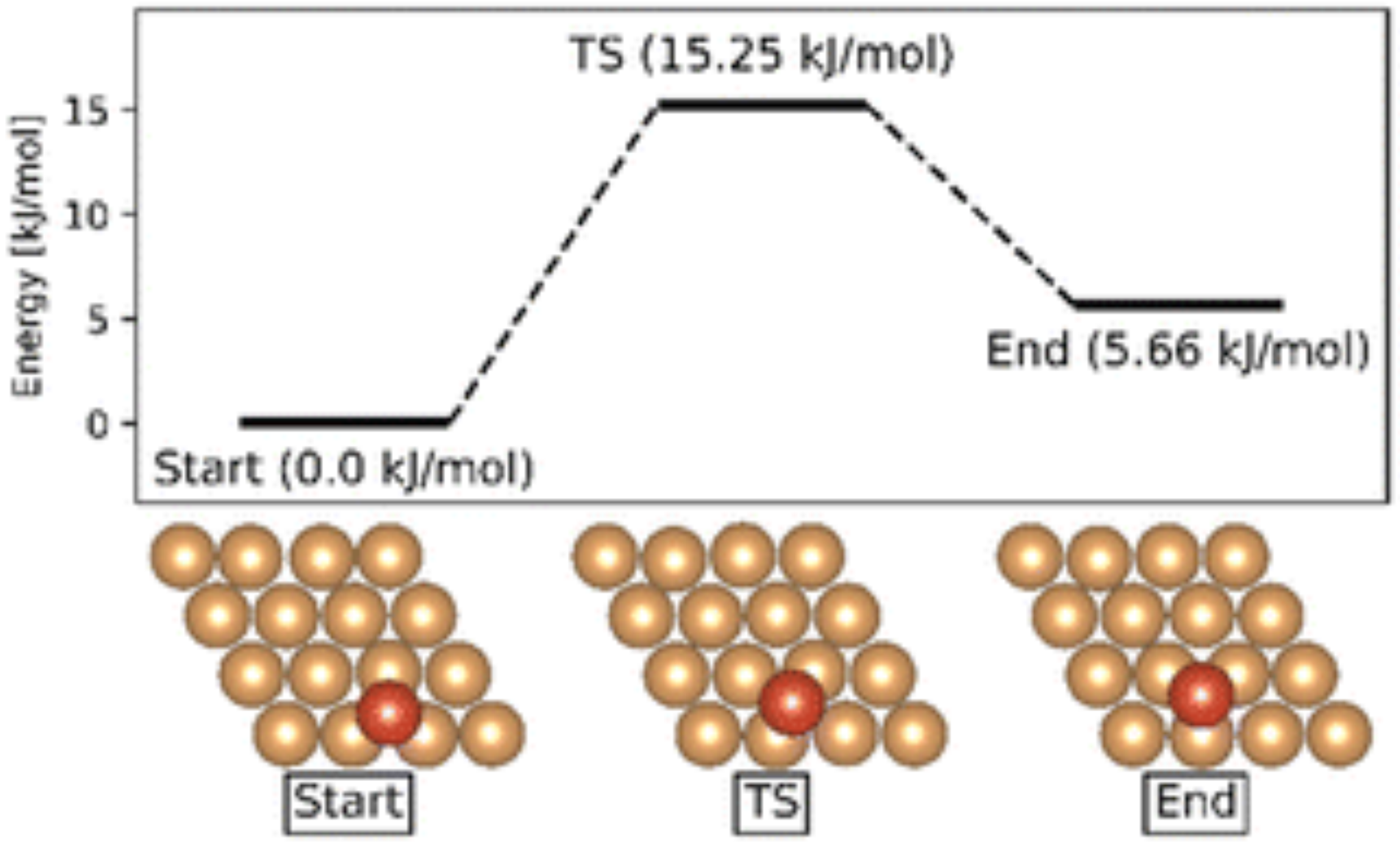
When thiol approaches, surface rearranges
➔ induces formation of adatoms



Barrier to movement for free thiol on Au(111)

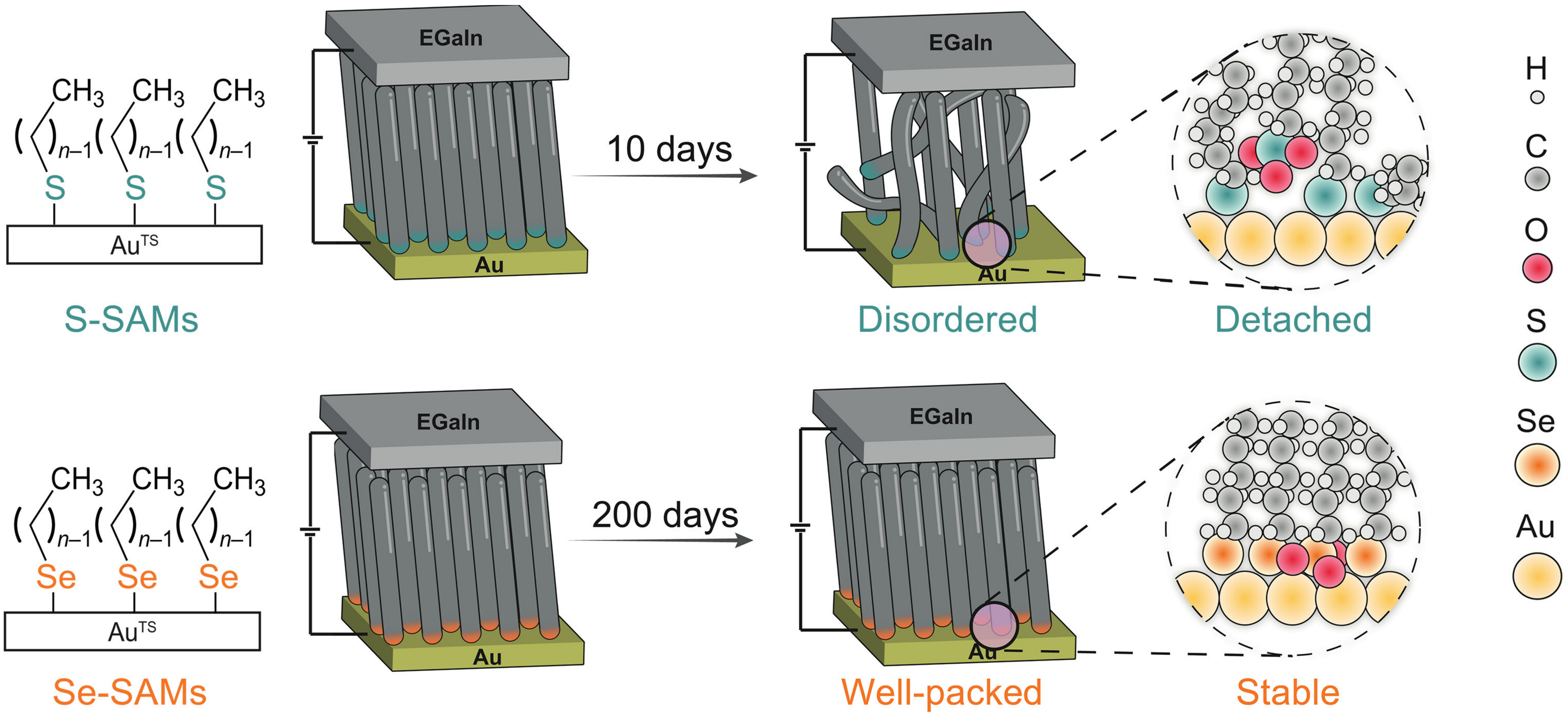


Barrier to movement for adatom on Au(111)



Duijnstee *et al.* | *Phys. Chem. Chem. Phys*| 2025

Leads to Disordered Thiolated SAMs on Au Under Ambient Conditions



Chen *et al.* | *Science Advances* | 2023

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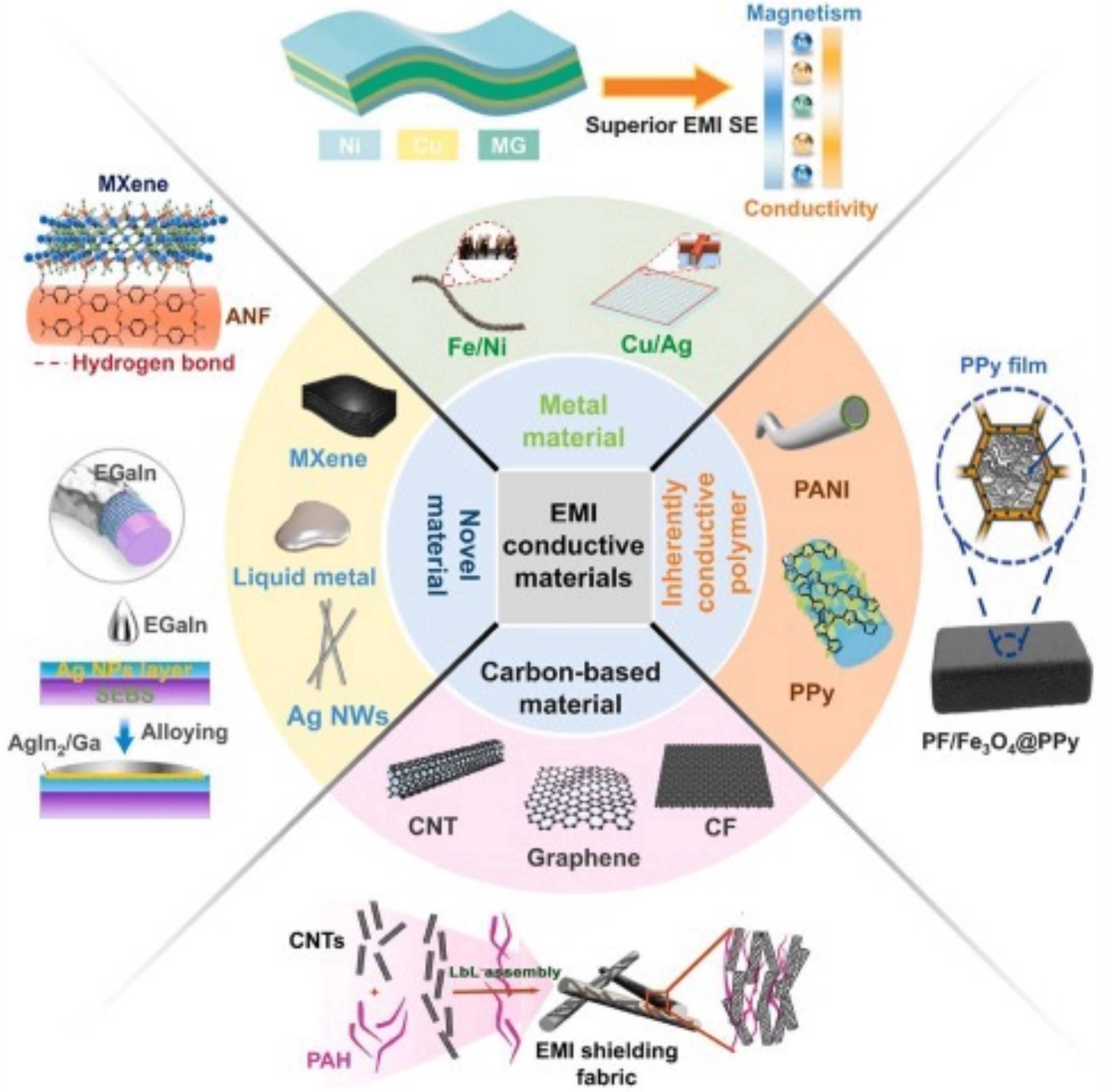
Thiols Do Not Chemisorb To Technologically Important Materials



Shi *et al.* | *Materials Today*| 2025

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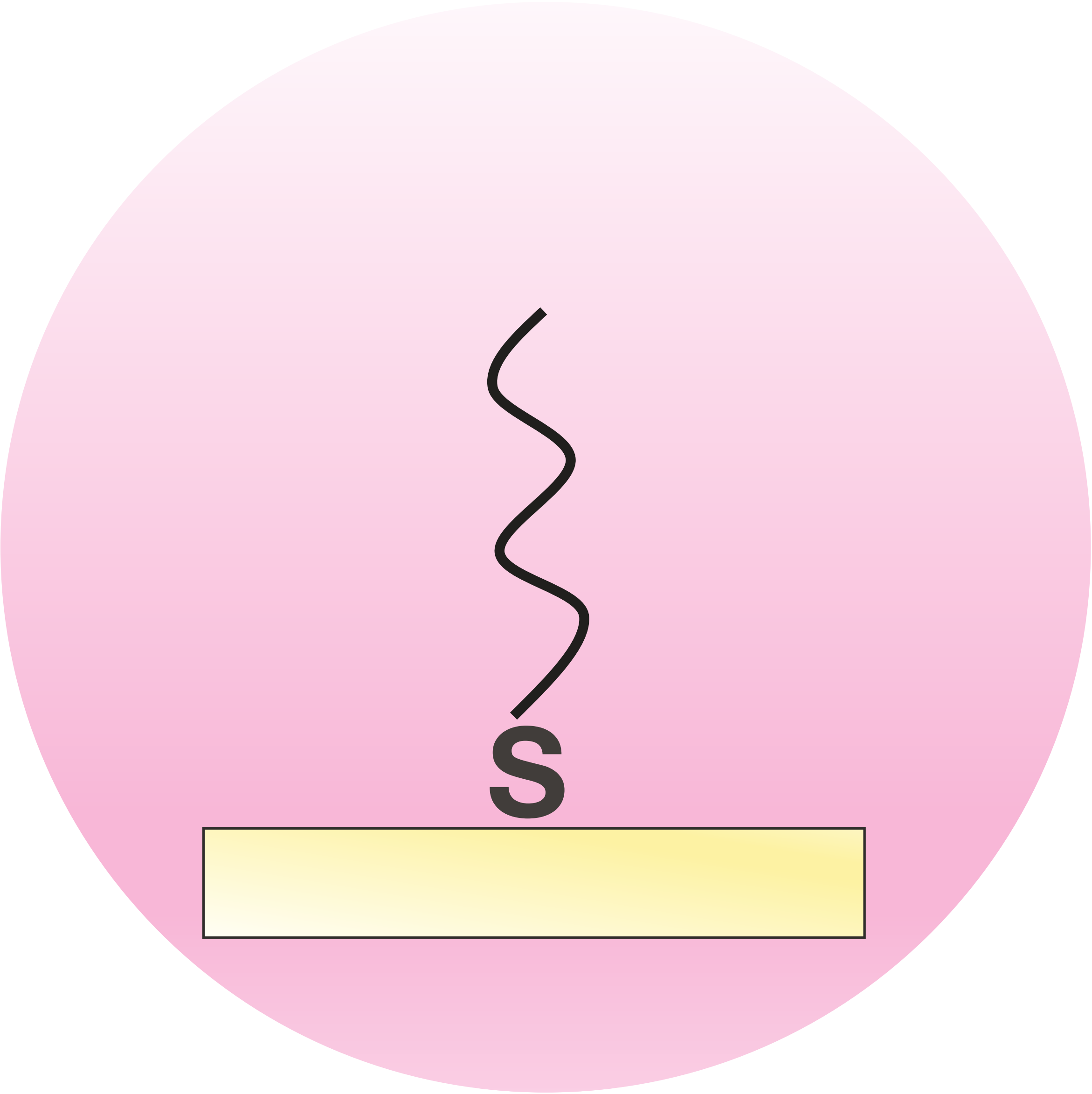
Key Takeaways

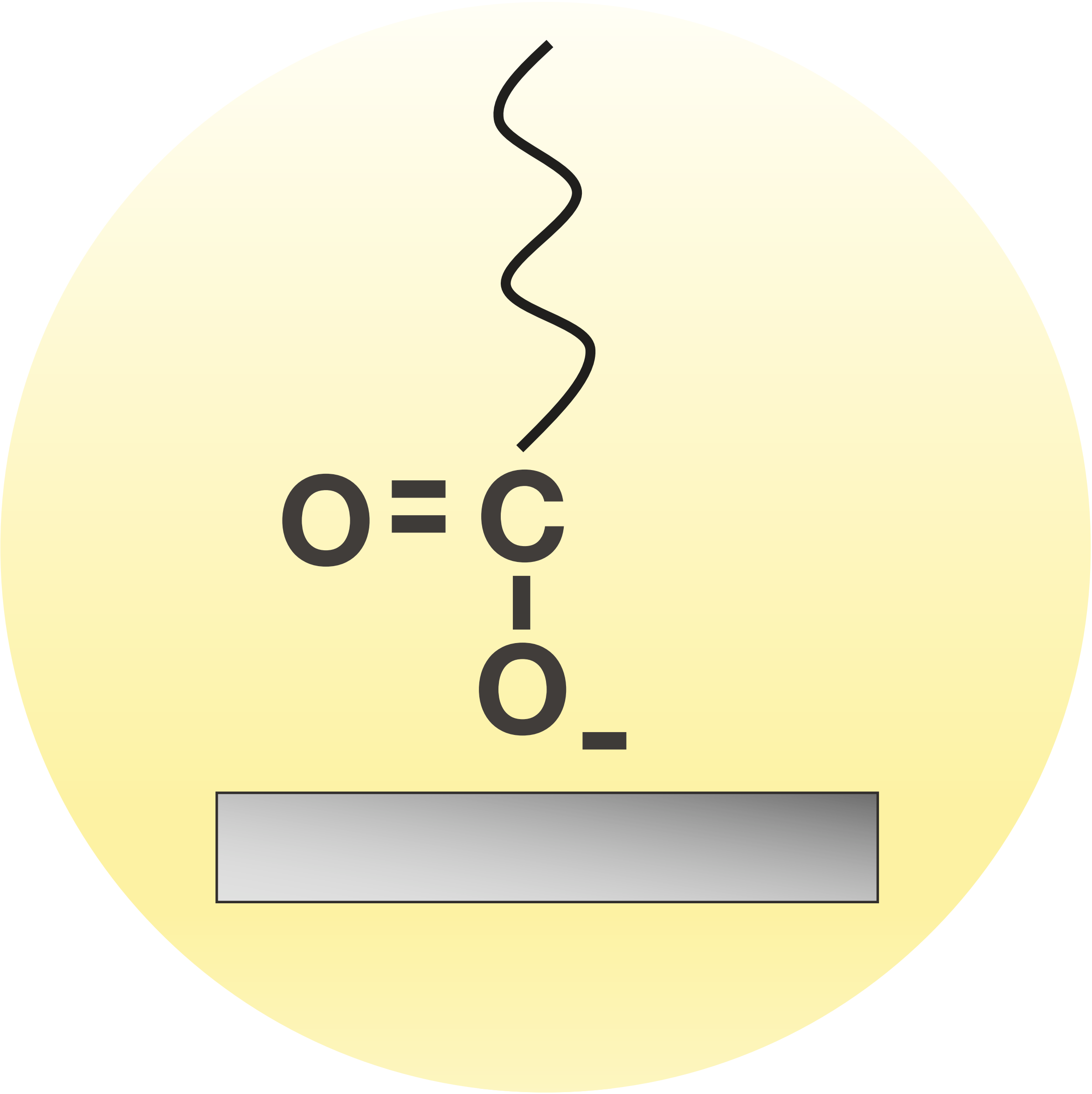
- We can functionalize metallic substrates with alkanethiols
- Alkanethiols adopt specific arrangements on surfaces based on lattice
- The substrate material and spacer groups can influence alkanethiol packing
- Alkanethiols are widely used but also show stability challenges
- Alternative chemistries can tackle remaining stability issues

Moving Beyond Alkanethiols

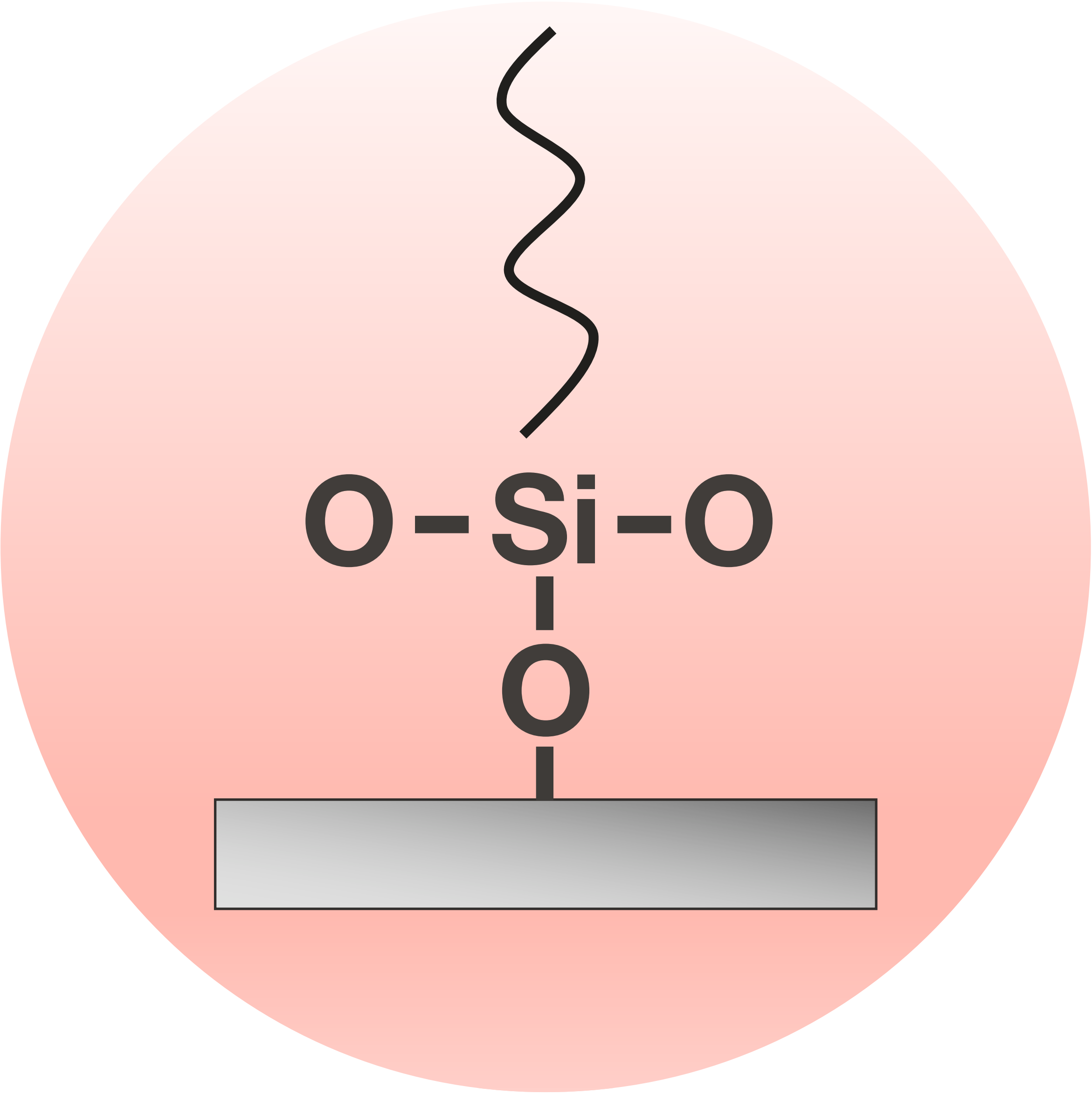
Surface chemistry



Thiol-based chemistry
Metallic surfaces

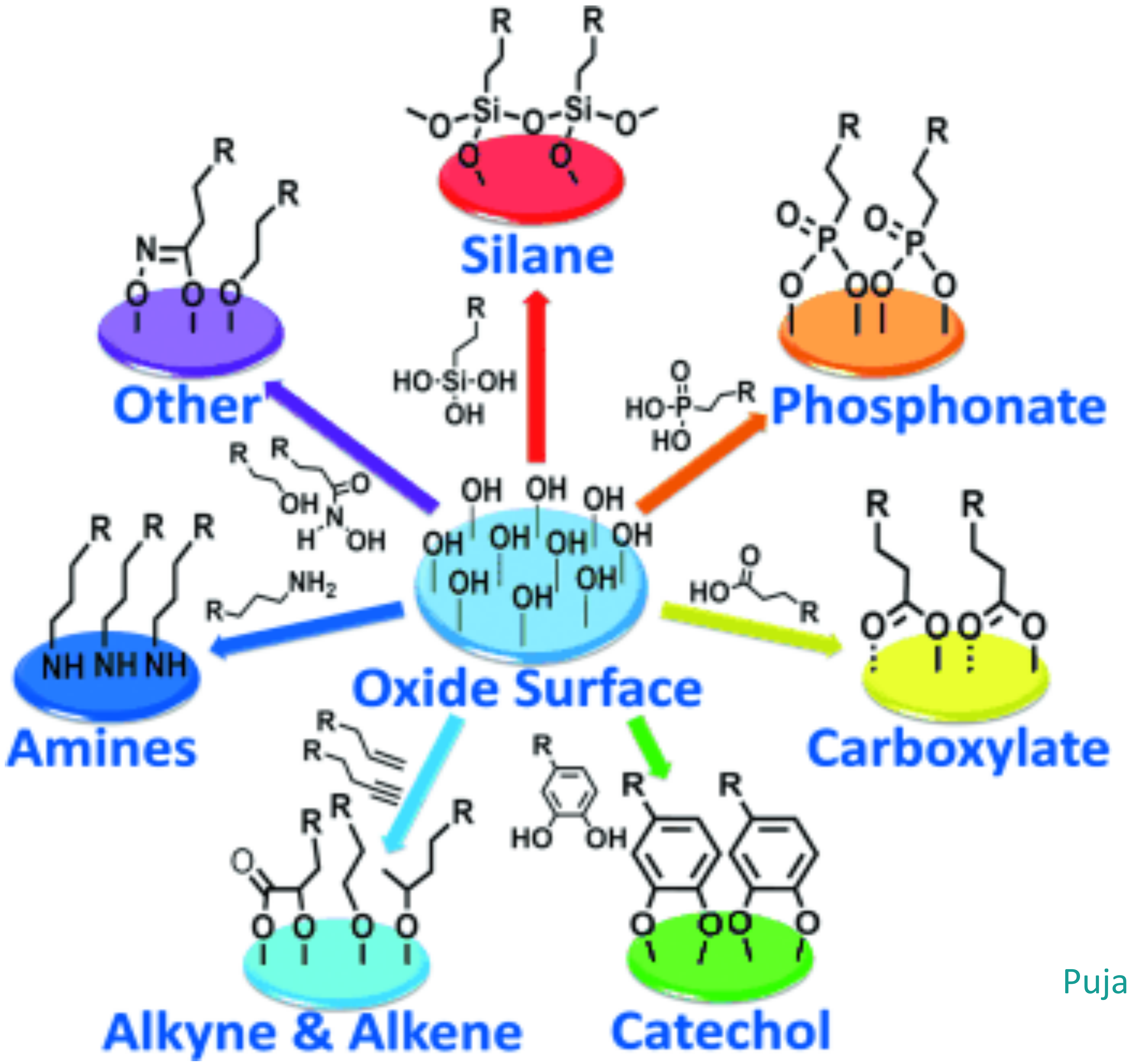


Carboxylic acids
Metal oxide surfaces



Silane, Phosphonates
Metal oxide surfaces

Covalent Surface Modification on Oxide Surfaces



Pujari *et al.* | *Angewandte Chemie*| 2014

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Silane Chemistry for Surfaces with –OH Groups (Metal Oxides)

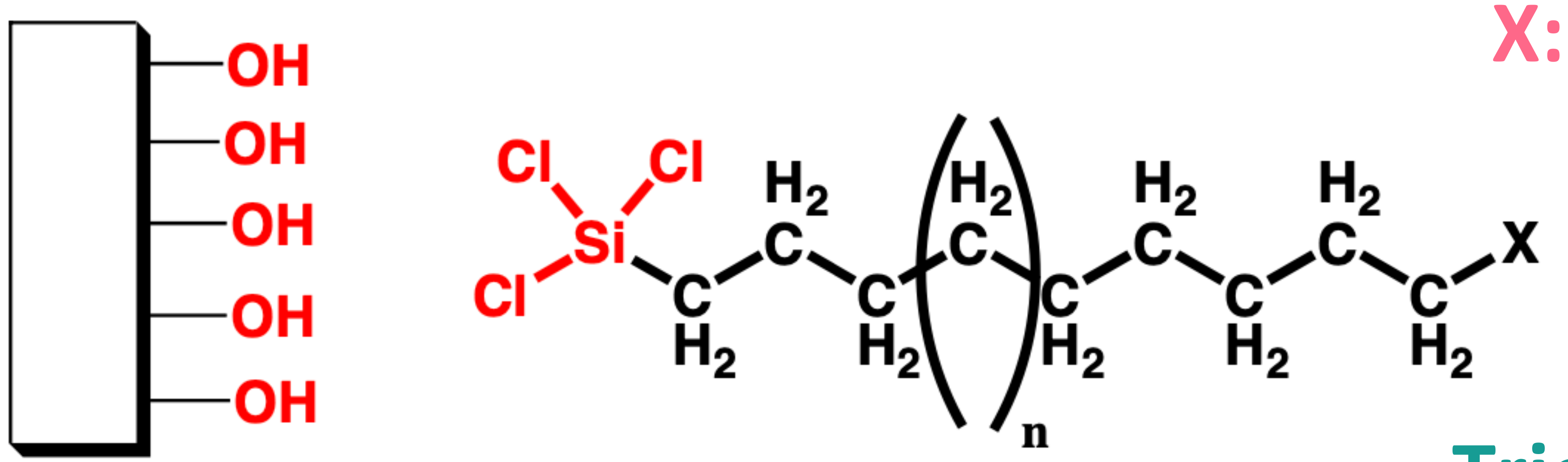
Applications of silane chemistry

Water repellant coatings

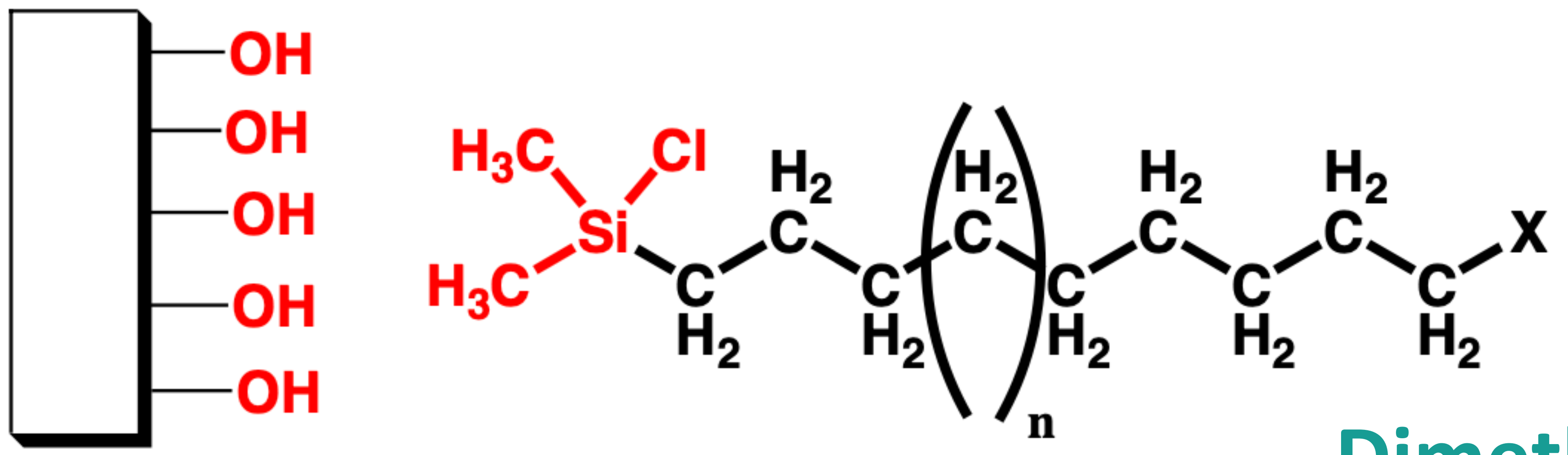
Adhesives, primers, paints

Antifog

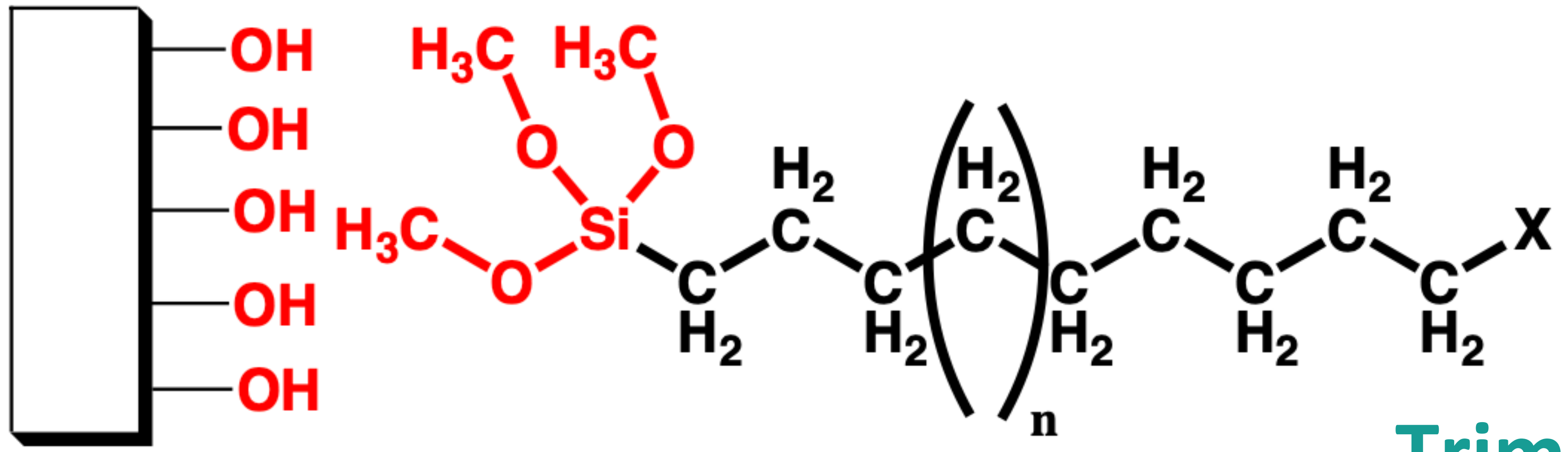
X: Functional group



Trichlorosilane



Dimethylchlorosilane



Trimethoxysilane

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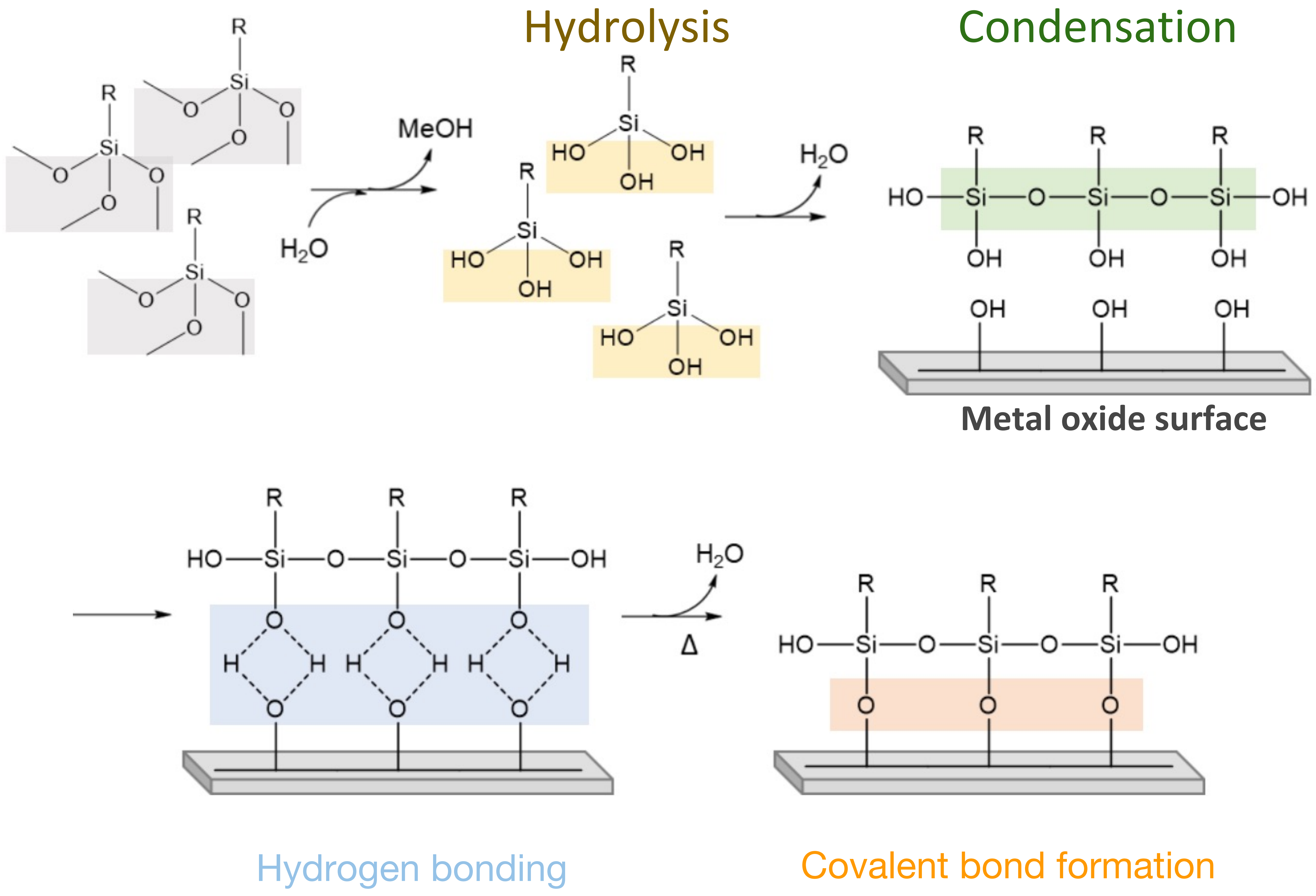
Alkylsilane Chemisorption Mechanism



Arkles | *CHEMTECH* | 1977

*The precise mechanism of assembly is unknown

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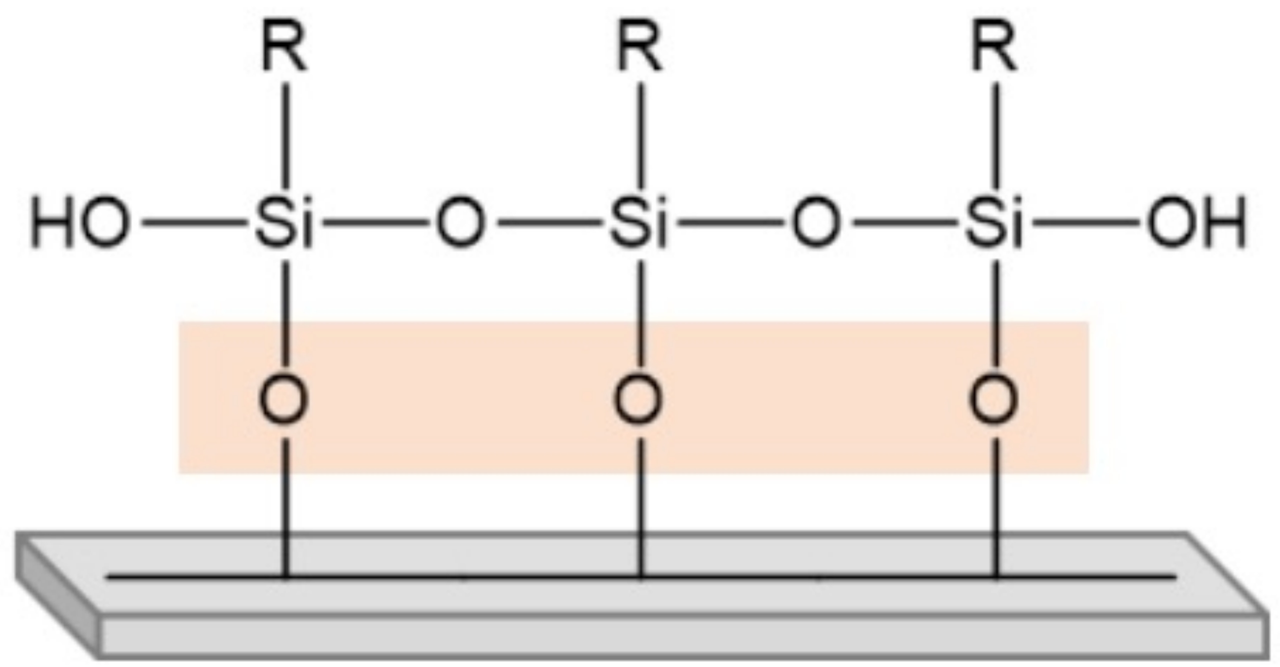
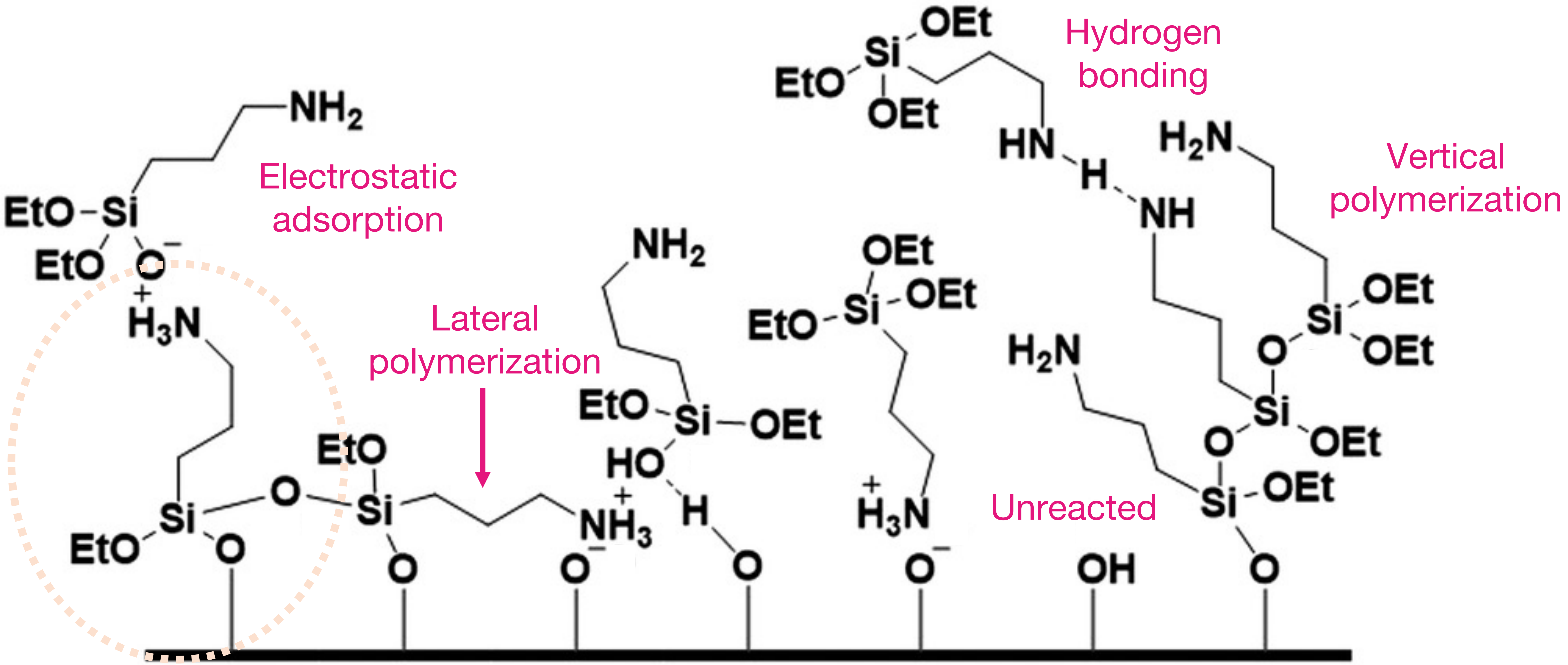
Challenges of Homogenous and Reproducible Silane Chemistry

Expectation ☺



Metal oxide surface

Reality ☹



Layer characteristics are heavily influenced by solvent, water content, environmental humidity, pH, silane concentration, reaction time, temperature, etc.

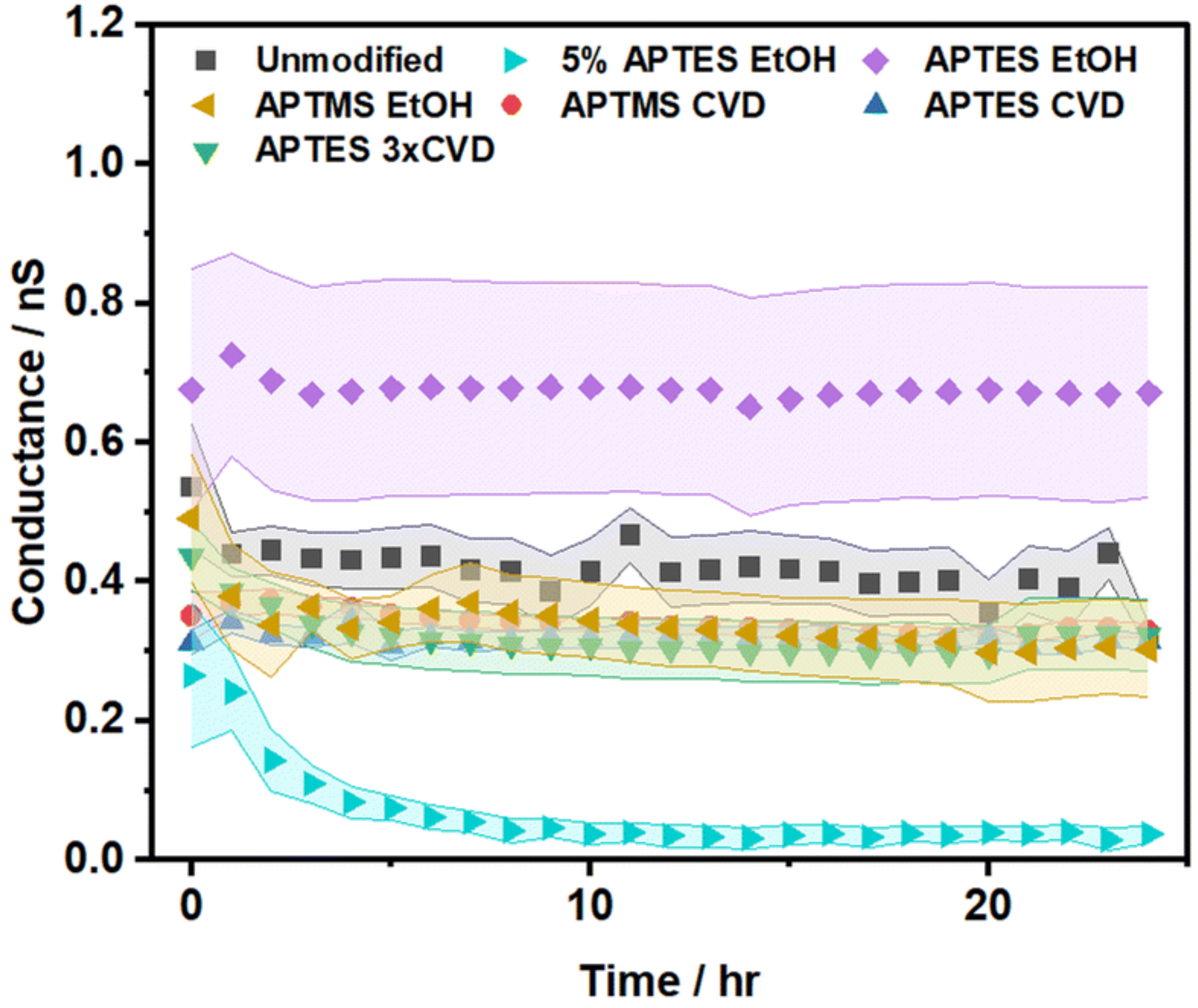
Duleba *et al.*, | *Phys. Chem. Chem. Phys.* | **2024**

Achieving Reproducible Silane Layers in the Lab

Modifying the surface of quartz surfaces

Pre-treatment	Silane	Concentration	Solvent	Reaction	Post-treatment
Piranha	APTES	5%	Ethanol	2 h	120 °C for 1 h
Piranha	APTES	5%	Ethanol	1 h	120 °C for 1 h
	APTES	1%	Ethanol	2 h	110 °C for 1 h (vacuum)
	APTES	1%	Ethanol		120 °C for 2 h
Piranha (0.5 h)	APTES	0–1.1%,	Ethanol	0–4 h	110 °C for 0.5 h
	APTMS	1%	Acetone	4 °C for 12 h	120 °C for 1 h
Boiling H_2O_2 (0.5 h)	APTMS	11%	Acetone	Overnight	120 °C for 1 h
	APTMS	Vapour-phase	Vapour-phase	40 °C for 1 h	
	TESPG	2.5%	Ethanol	Overnight	N_2 dry
Piranha (24 h)	DHITES	0.74 mM	Ethanol	Overnight	
	DHITES	17%	Ethanol	Overnight	
Piranha (24 h)	ATMS	1 mM	Ethanol	Overnight	
Piranha (2 h)	TESP-SA	1%	Isopropanol	1 h	60 °C for 1.5 h (vacuum)
Piranha	TESBA	5%	Ethanol, acetate buffer (pH 4.7)	20 h	
	MPTES	0.01%	Ethanol	15 min	60 °C for 2–3 h
Boiling H_2O_2 (20 min)	MPTMS	10%	Ethanol	4 h	120 °C

Solution deposition yields higher variability



Duleba *et al.*, | *Phys. Chem. Chem. Phys.* | **2024**

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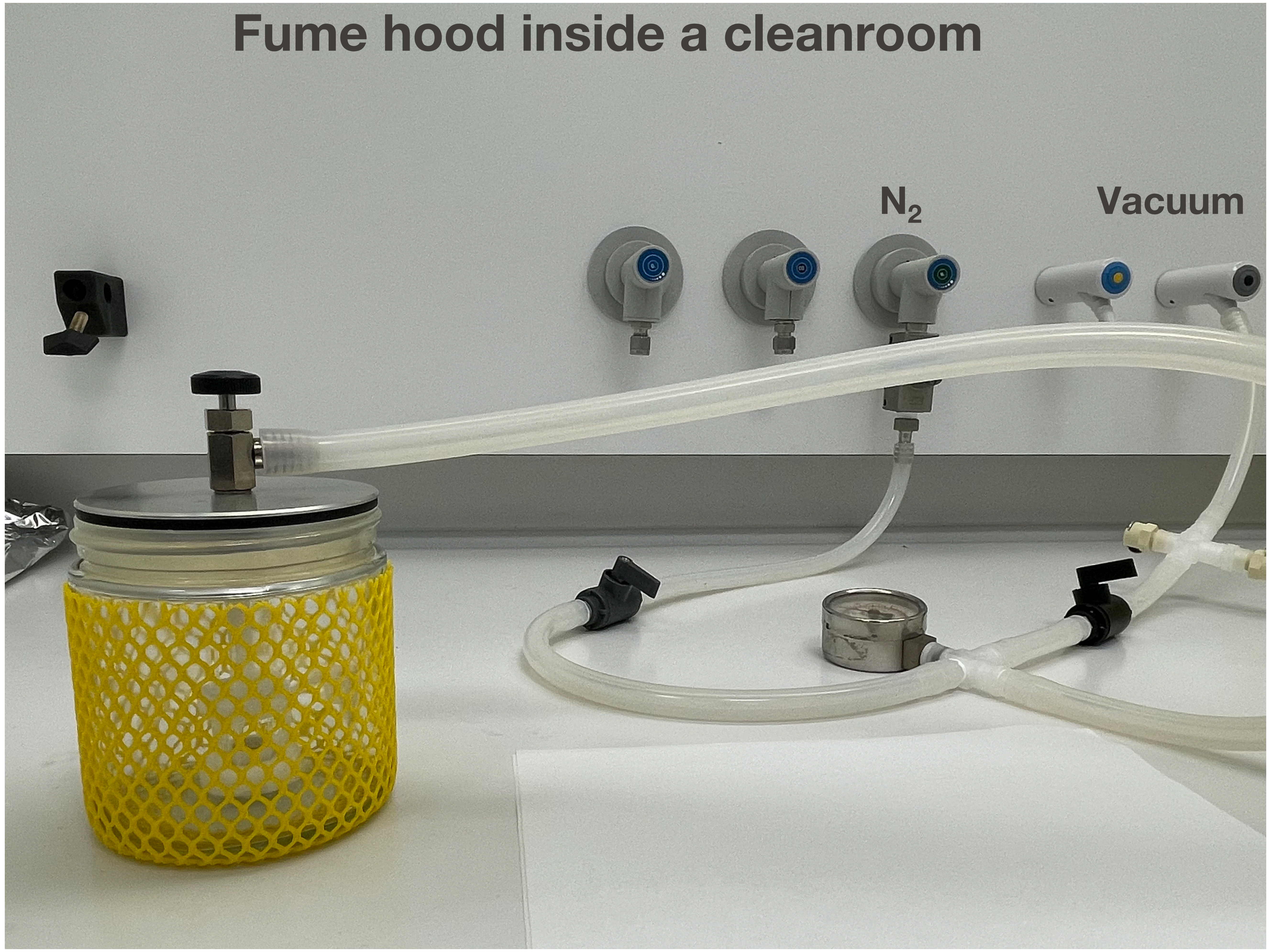
How We Conduct Silane Chemistry in our Lab

Lianxin Xu



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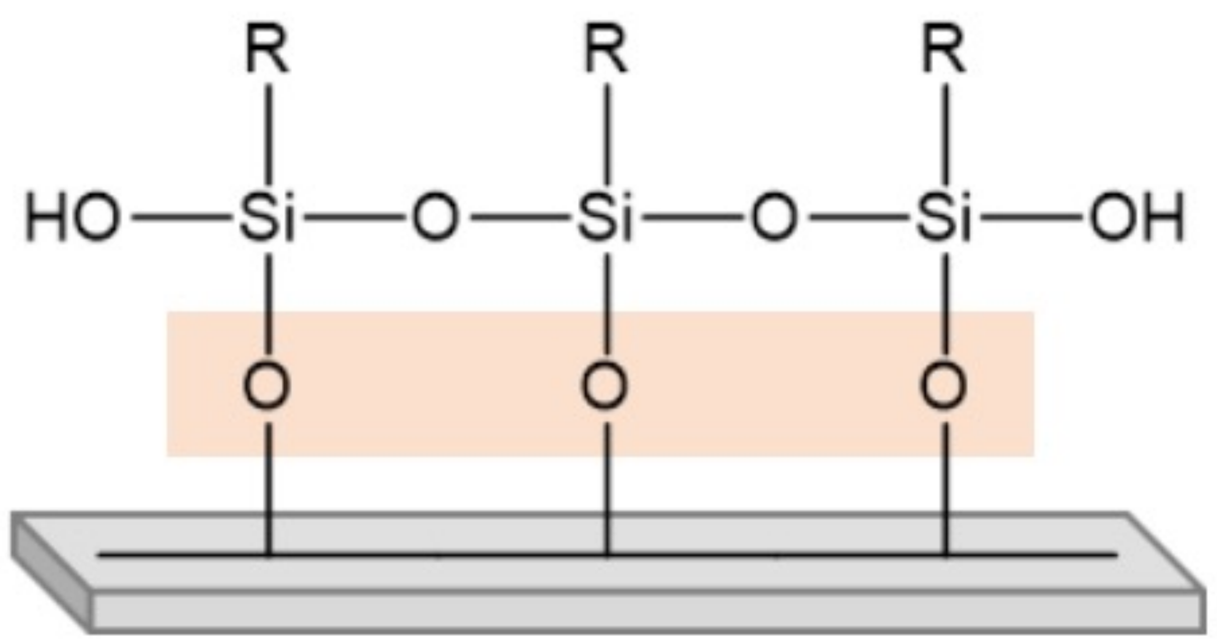
The Pros and Cons of Alkylsilanes



Metal oxide surface (-OH)

Only requirement for substrate is presence of –OH groups

Many functionalized silanes commercially available

High stability due to polysiloxane formation

Limitation in functional groups due to possible reactivity with head/anchor group

Reproducible sample prep can be difficult

Multilayer formation is possible (and common)

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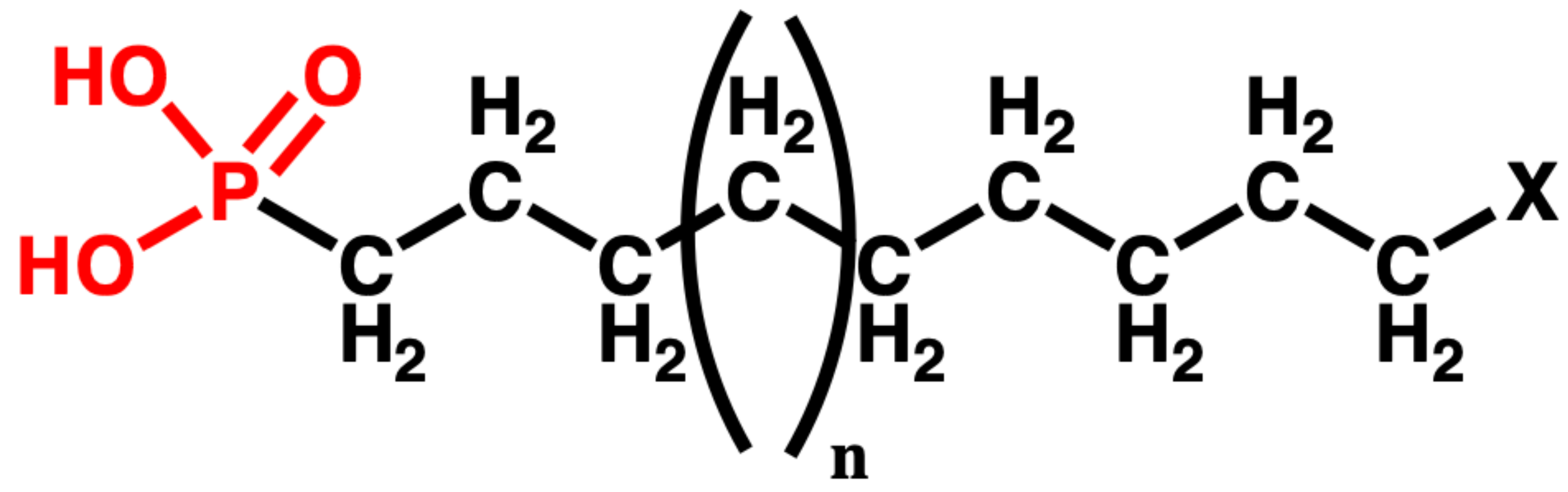
Alkylphosphonates for Surfaces with –OH Groups (Metal Oxides)

Advantages to alkylsilanes:

- High stability in water
- Improved reproducibility
- Form true monolayers

metal oxides

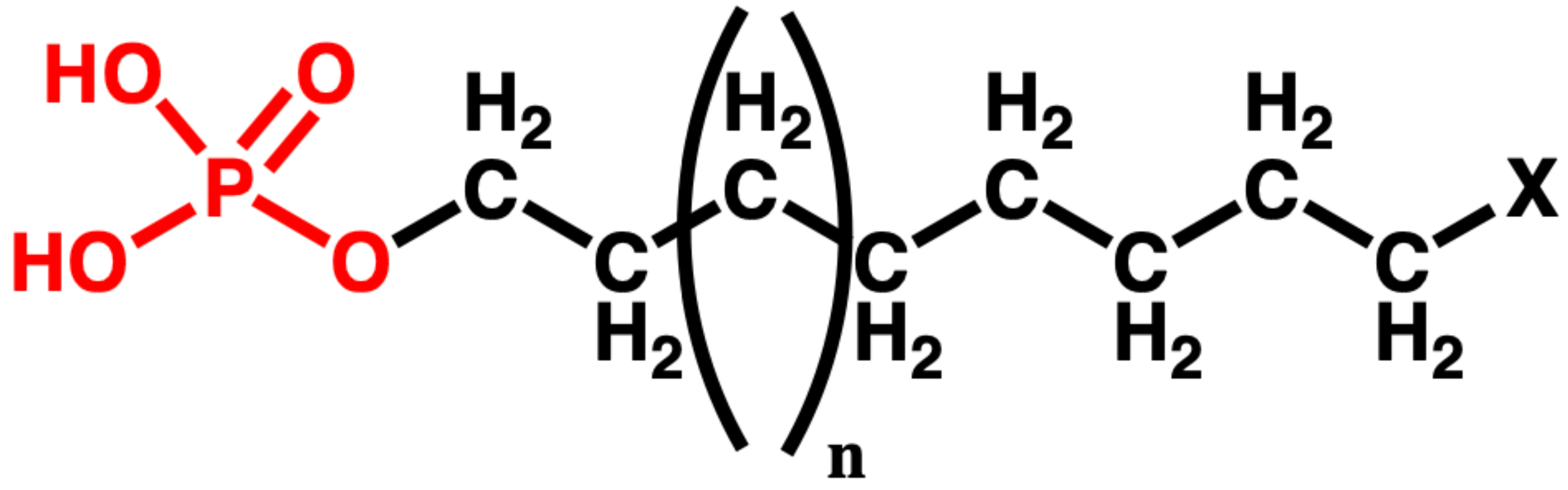


Phosphonate
Phosphonic acid

Disadvantages to alkylsilanes:

- Fewer commercial variants
- No adsorption on SiO_2
- Requires perfect oxide surface
- Solution phase deposition

metal oxides



Phosphate
Phosphoric acid

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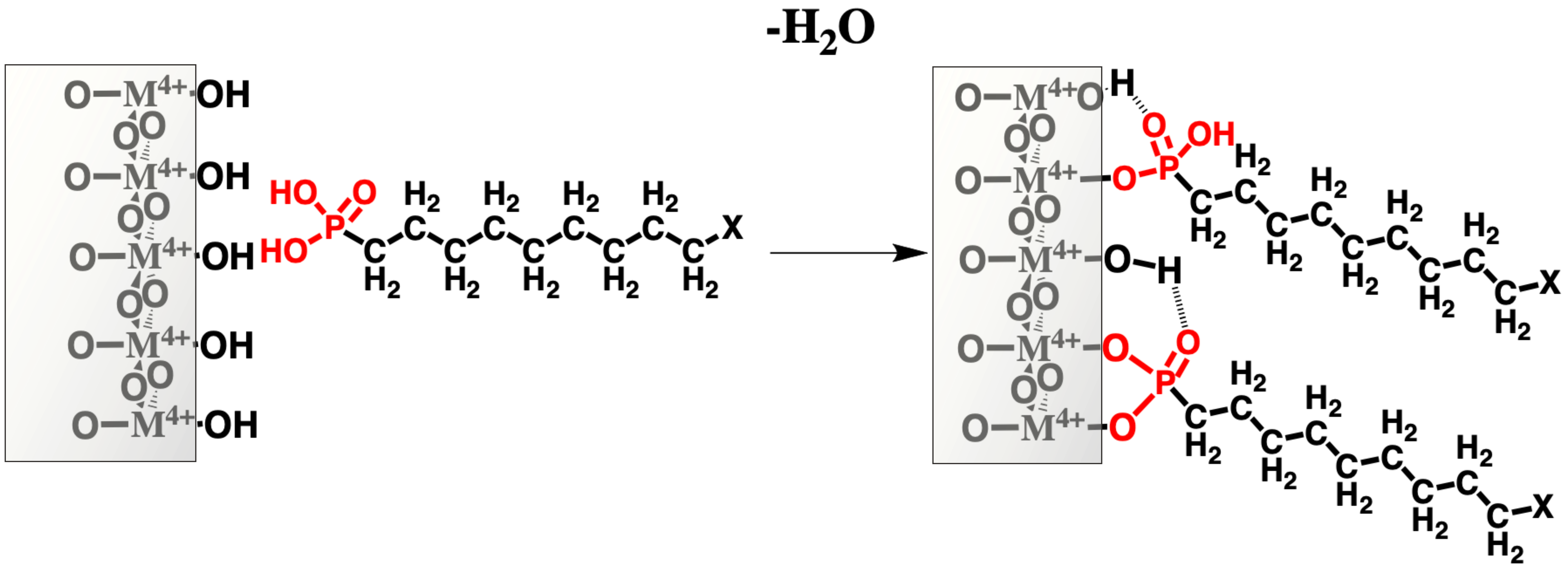
Alkylphosphonates for Surfaces with –OH Groups (Metal Oxides)

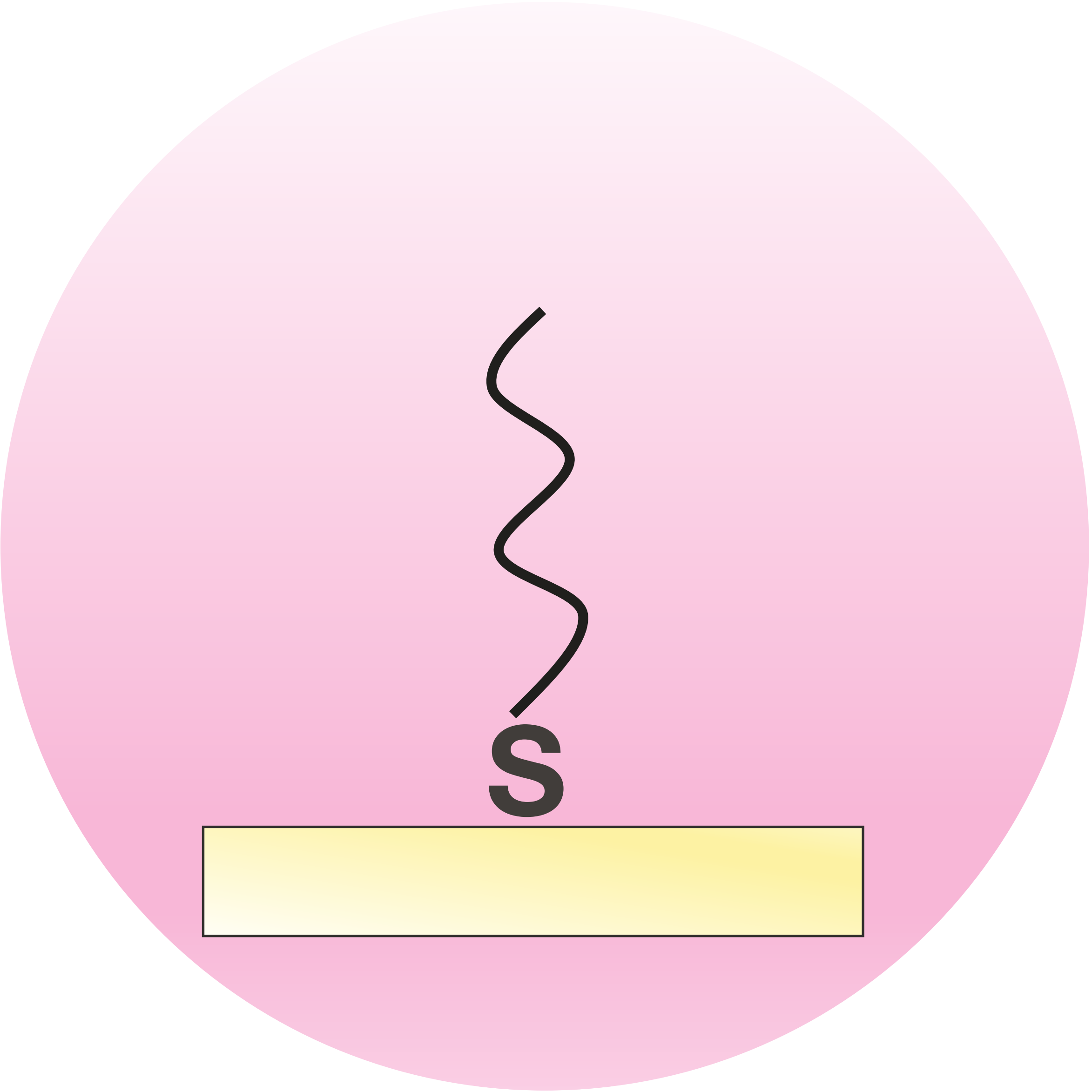
Condensation reaction on metal oxide surface



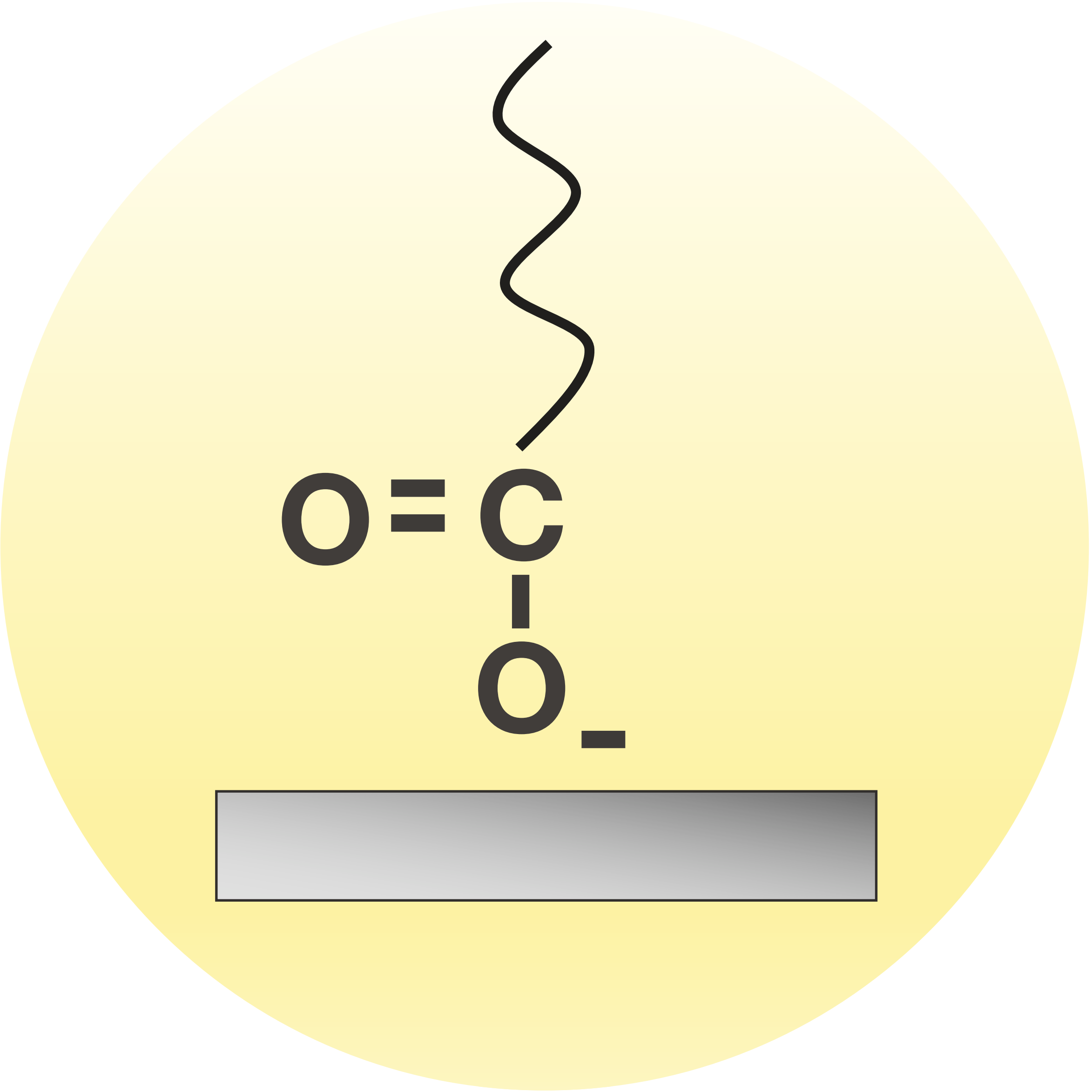
Formation of one or even two bonds with surface –OH groups
➔ stable and well-defined monolayer on surfaces

Moving Beyond Alkylsilanes

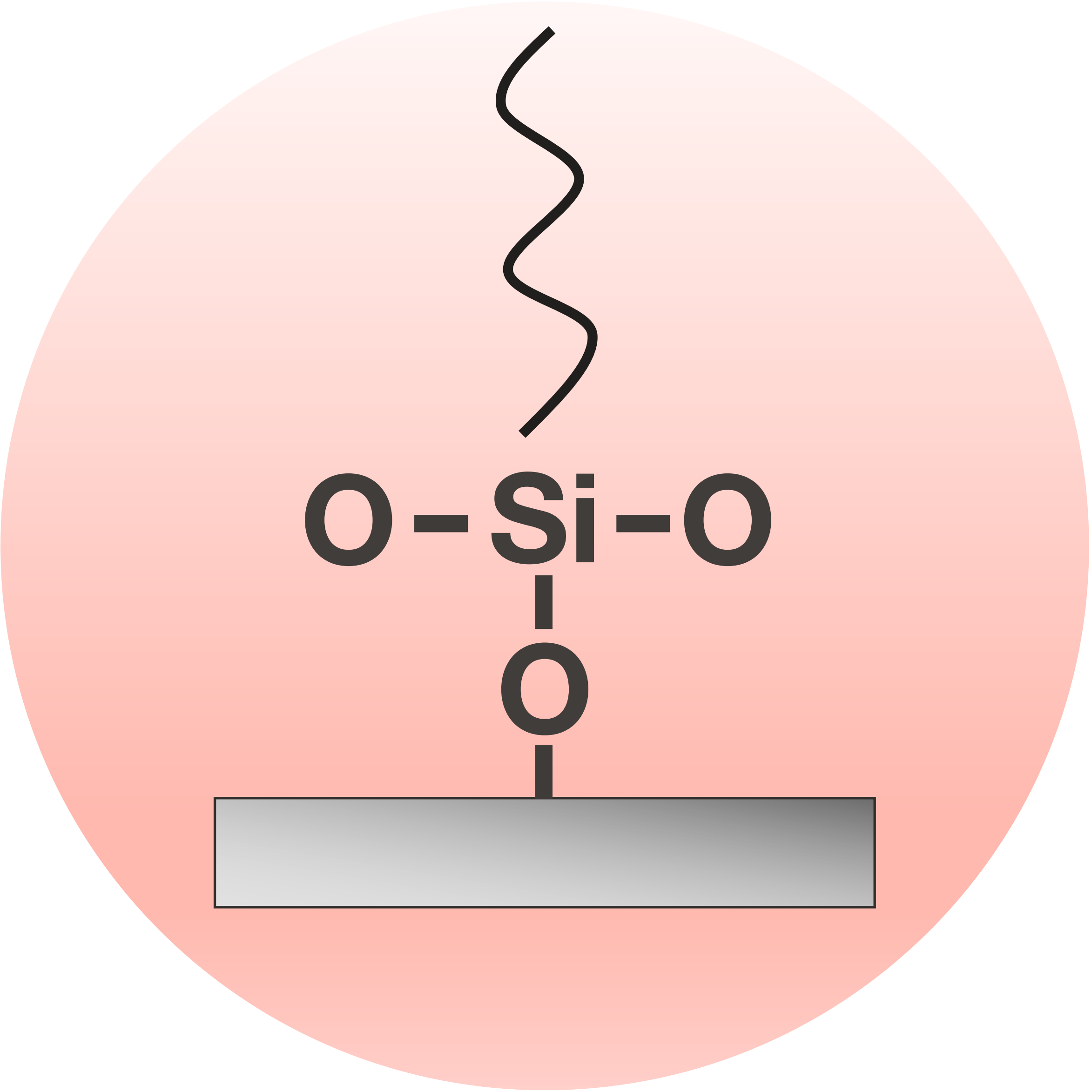
Surface chemistry



Thiol-based chemistry
Metallic surfaces



Carboxylic acids
Metal oxide surfaces



Silane, Phosphonates
Metal oxide surfaces

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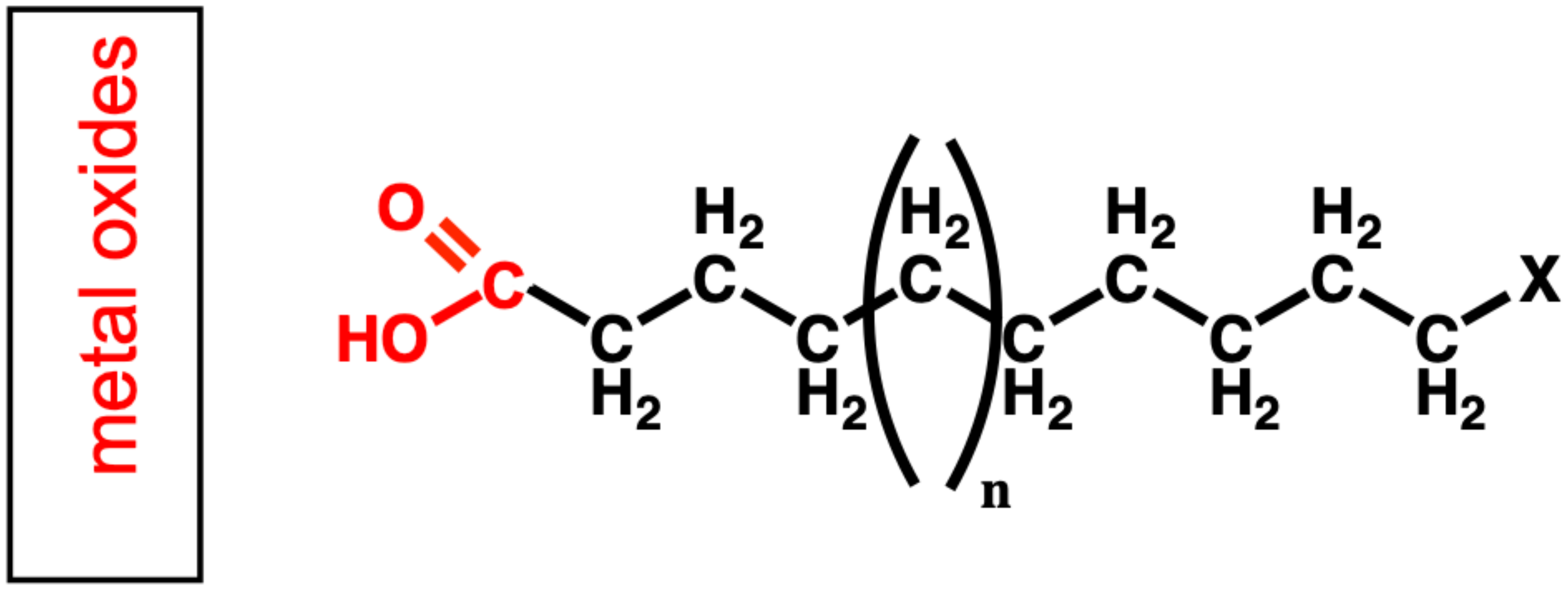
Carboxylic Acids Adsorb Non-Covalently (Electrostatically)

Acid-base reaction on oxide surfaces



Weak attachment (electrostatic)

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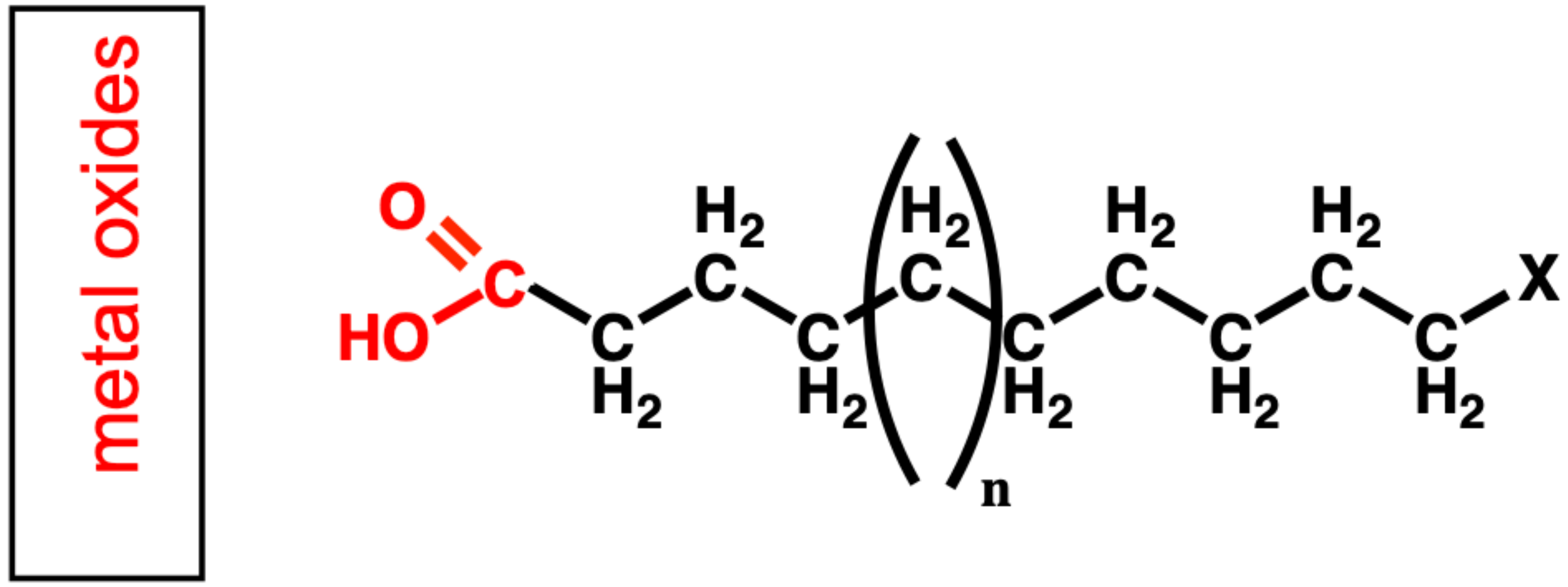
The Pros and Cons of Carboxylic Acids



Adsorbs onto Al_2O_3 – one of the most important engineering materials

Unstable (weak surface interactions)

Less densely packed that all other systems

Limited range of functional groups

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Key Takeaways

- We can functionalize oxide surfaces using covalent chemistries
- Silane chemistry is robust but hard to control reproducibly
- Alkylphosphonates have improved reproducibility but with challenges
- Non-covalent interactions can also assemble molecules but are weaker

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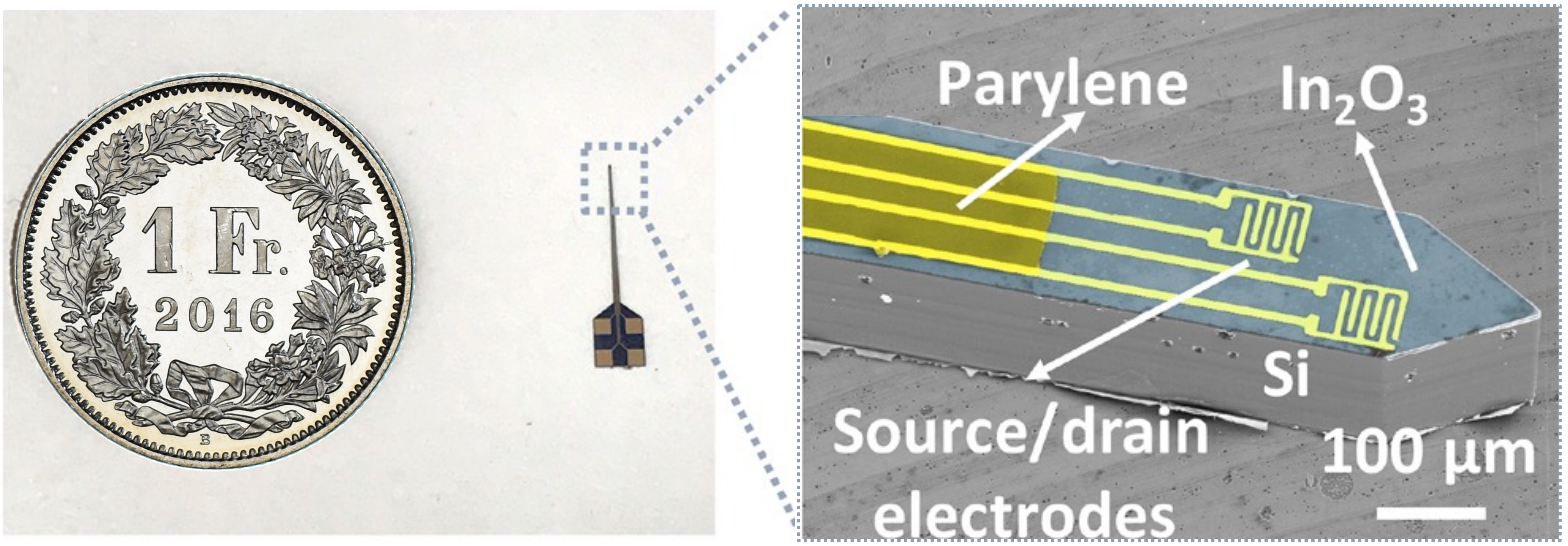
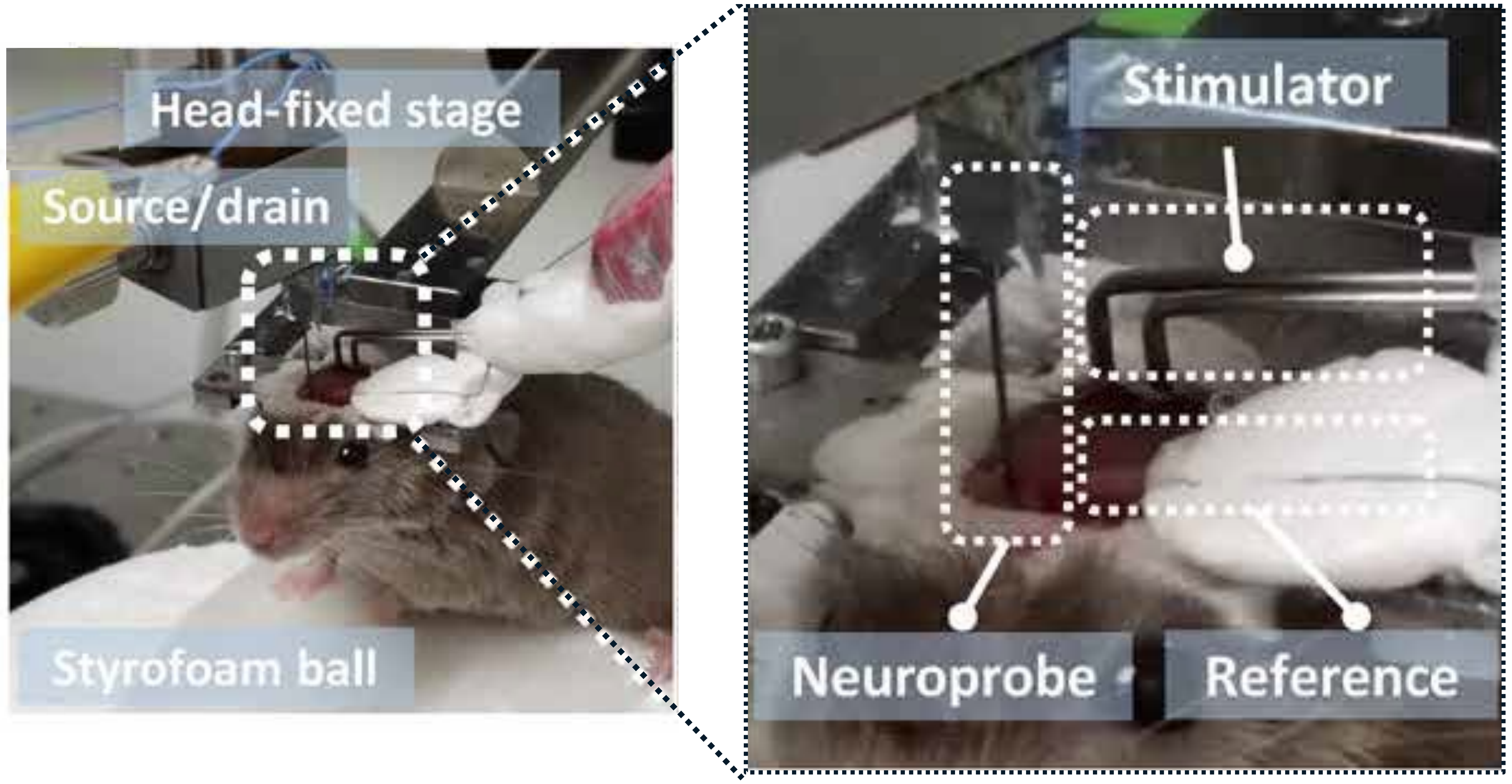
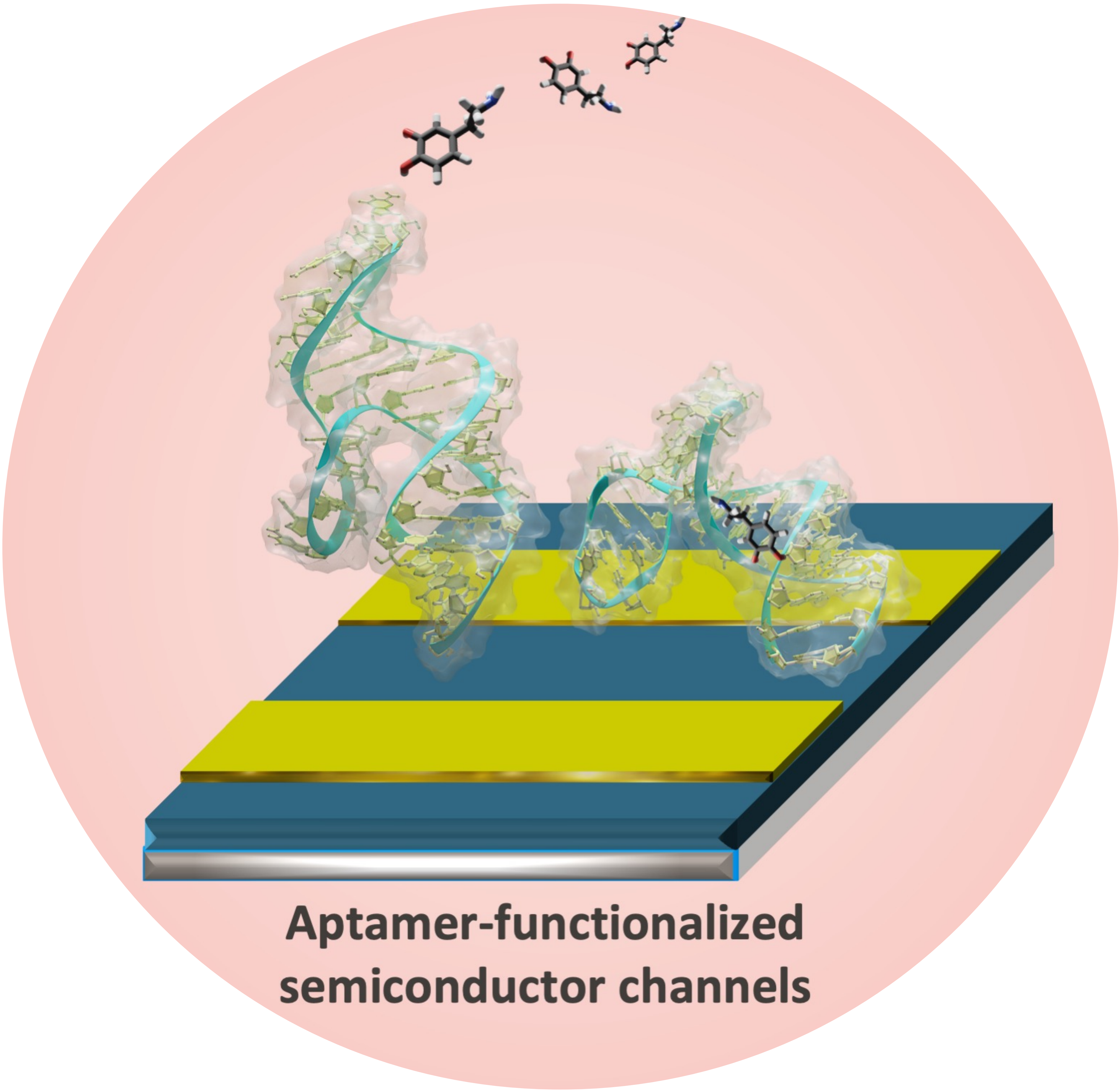
Employing Surface Chemistry in Our Research





Zhao *et al.* | *Science Advances* | 2021



Nakatsuka *et al.* | *Science* | 2018

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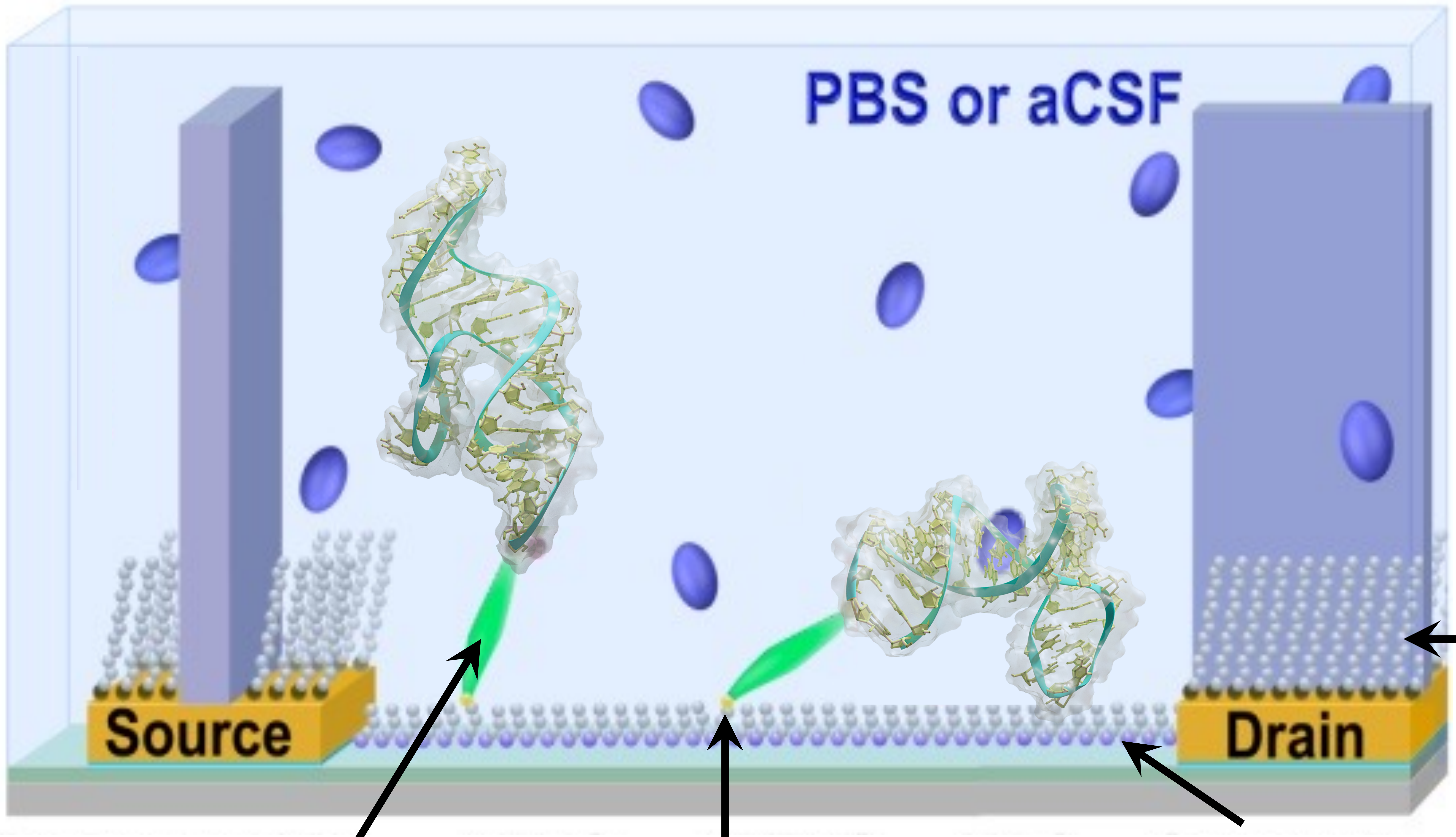
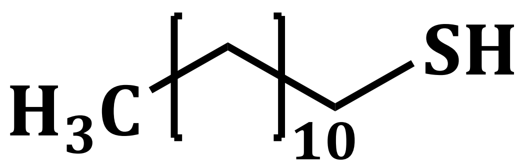
Various Surface Chemistries Enable Biosensor Configuration

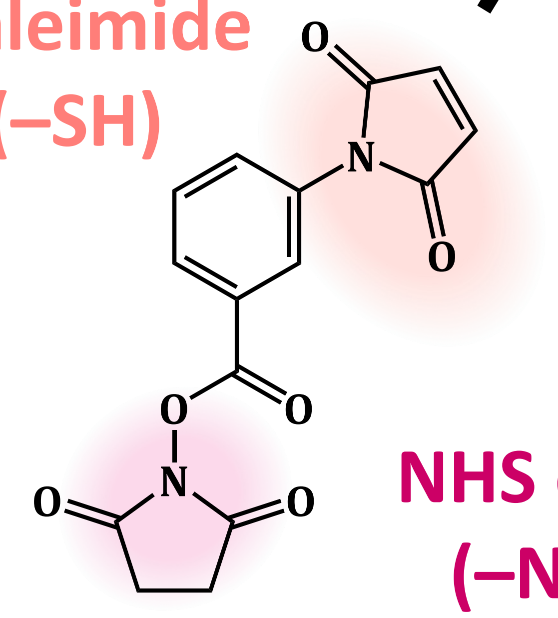


Au (30 nm)	Conductive electrodes
Ti (10 nm)	Adhesive layer
In_2O_3 (4 nm)	Semiconductor
SiO_2 (100 nm)	Dielectric layer
Si Wafer (500 µm)	Substrate

Dodecanethiol

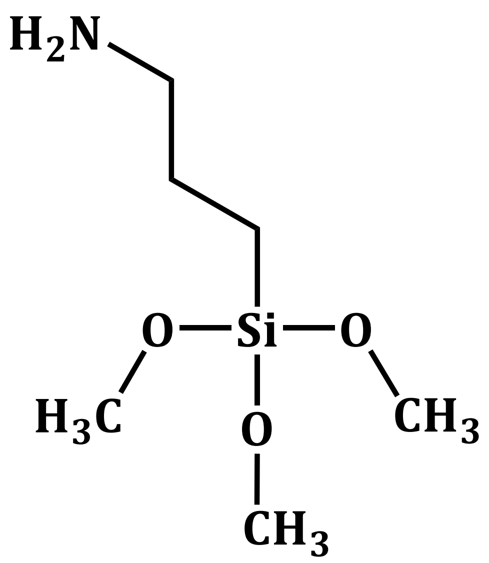


Maleimide (–SH)

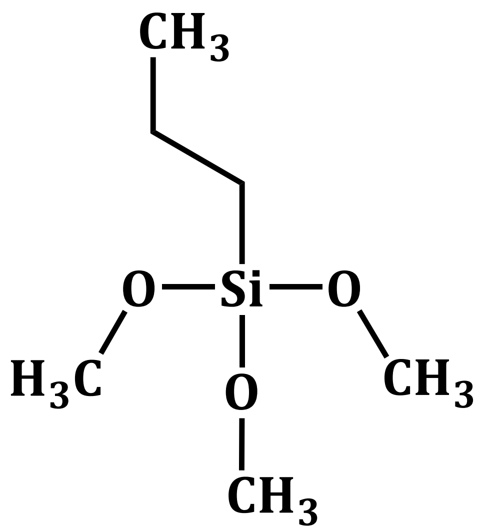


NHS ester (–NH_2)

3-Aminopropyltrimethoxysilane



Propyltrimethoxysilane



Nakatsuka *et al.* | *Science* | 2018

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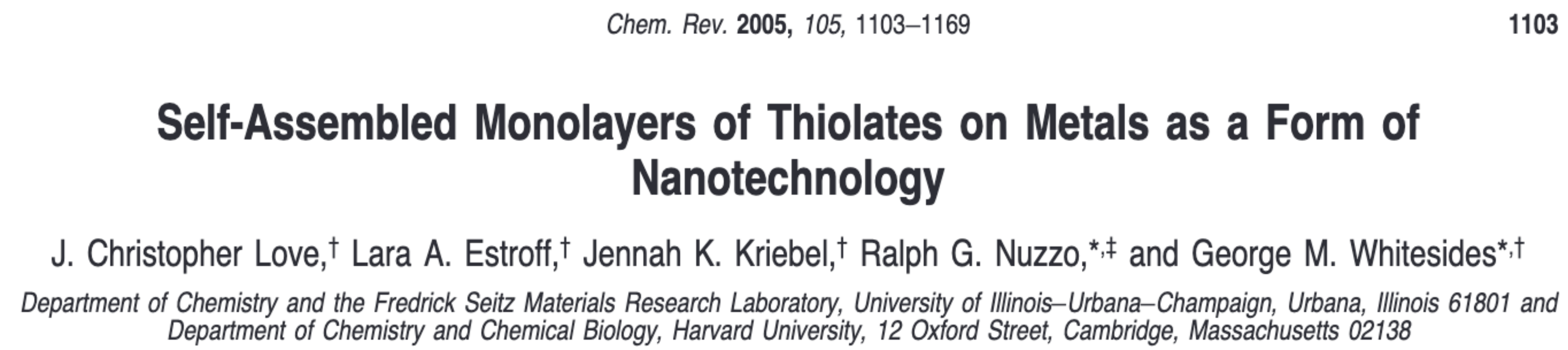
Intrinsic and Extrinsic Defects Found in Polycrystalline SAMS

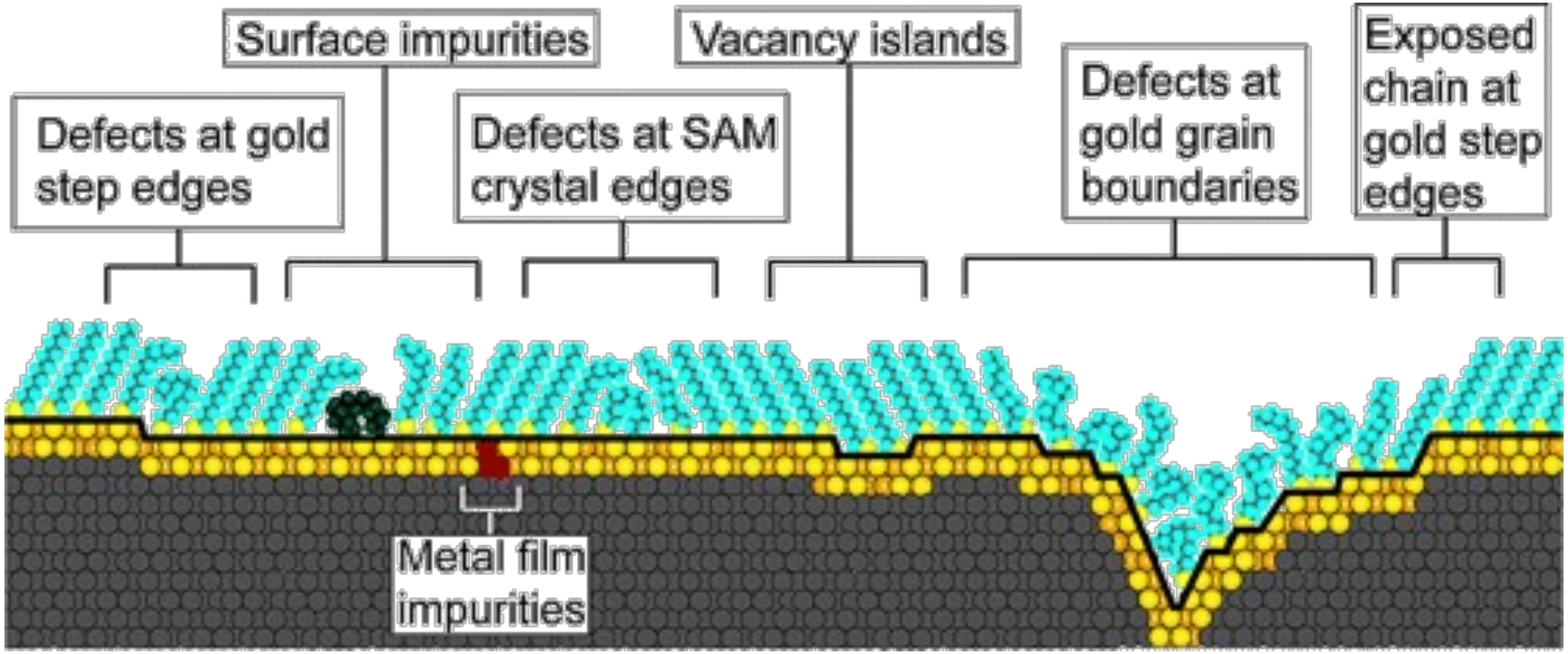

Chem. Rev. 2005, 105, 1103–1169 1103

Self-Assembled Monolayers of Thiolates on Metals as a Form of Nanotechnology

J. Christopher Love,† Lara A. Estroff,† Jennah K. Kriebel,† Ralph G. Nuzzo,*,‡ and George M. Whitesides*,†

Department of Chemistry and the Fredrick Seitz Materials Research Laboratory, University of Illinois–Urbana–Champaign, Urbana, Illinois 61801 and Department of Chemistry and Chemical Biology, Harvard University, 12 Oxford Street, Cambridge, Massachusetts 02138

Cited > 10,000 times!



Intrinsic and Extrinsic Defects Found in Polycrystalline SAMS

Chem. Rev. **2005,** *105,* 1103–1169 **1103**

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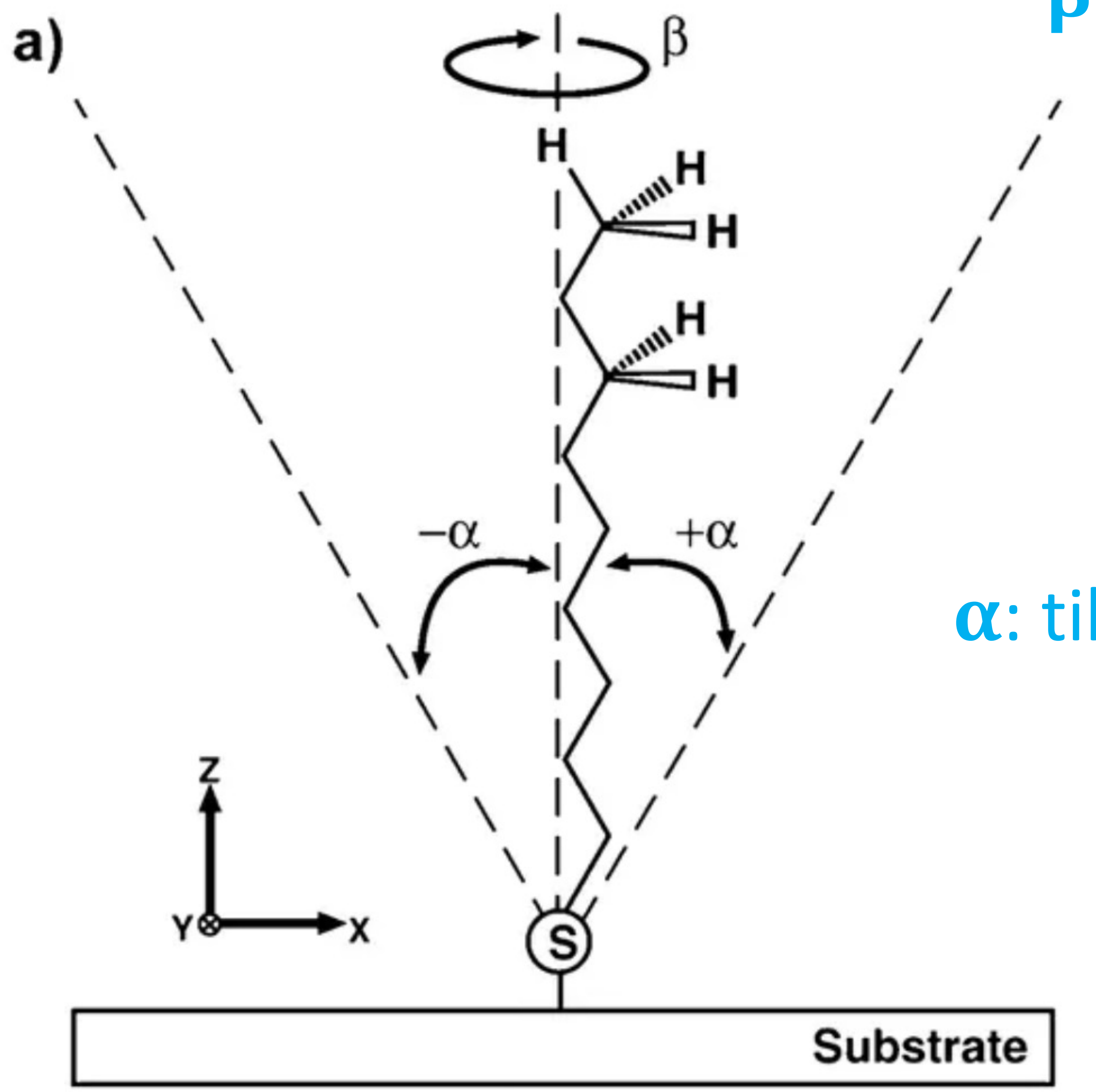

β: rotation of C-C bond

α: tilt angle

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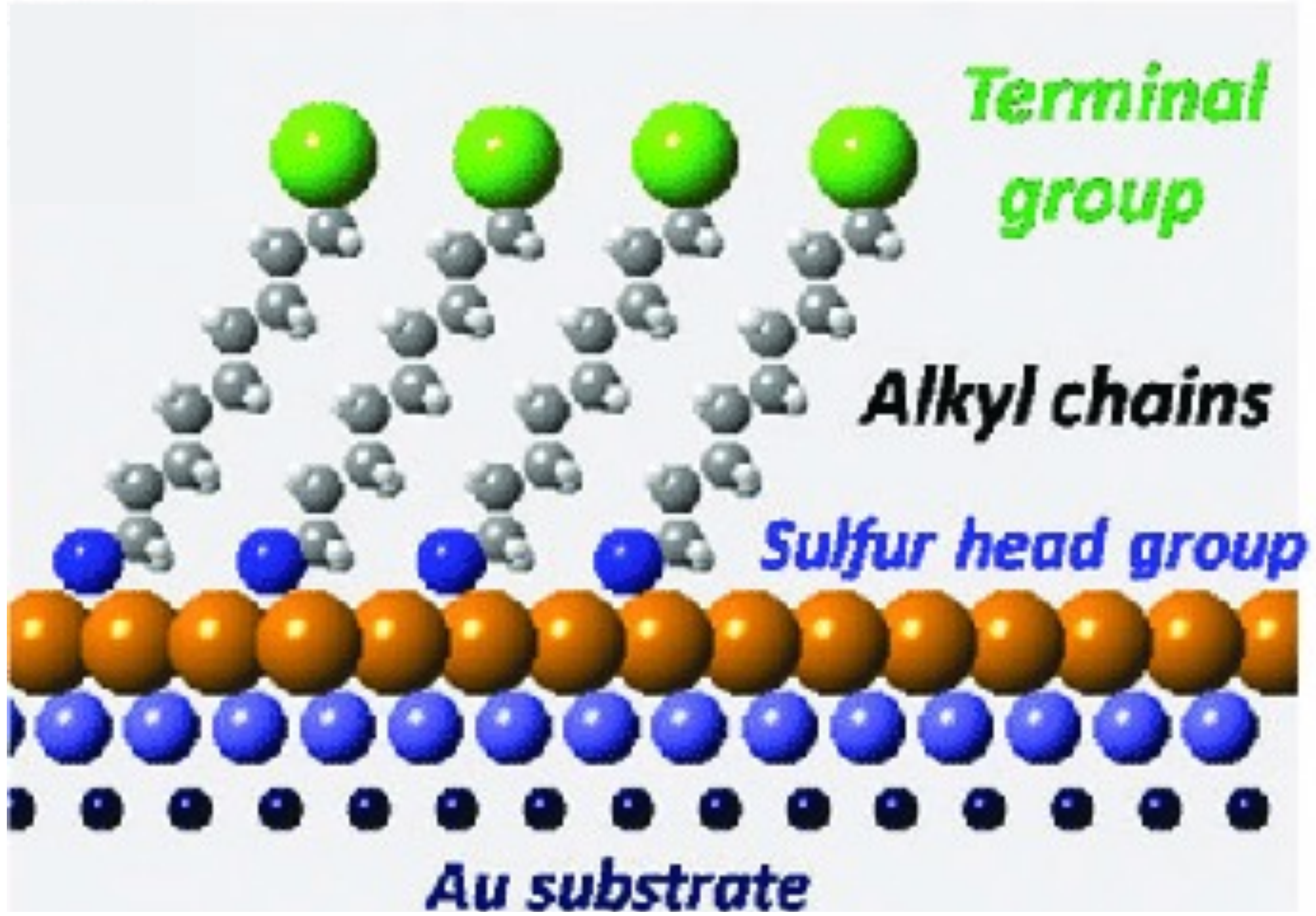
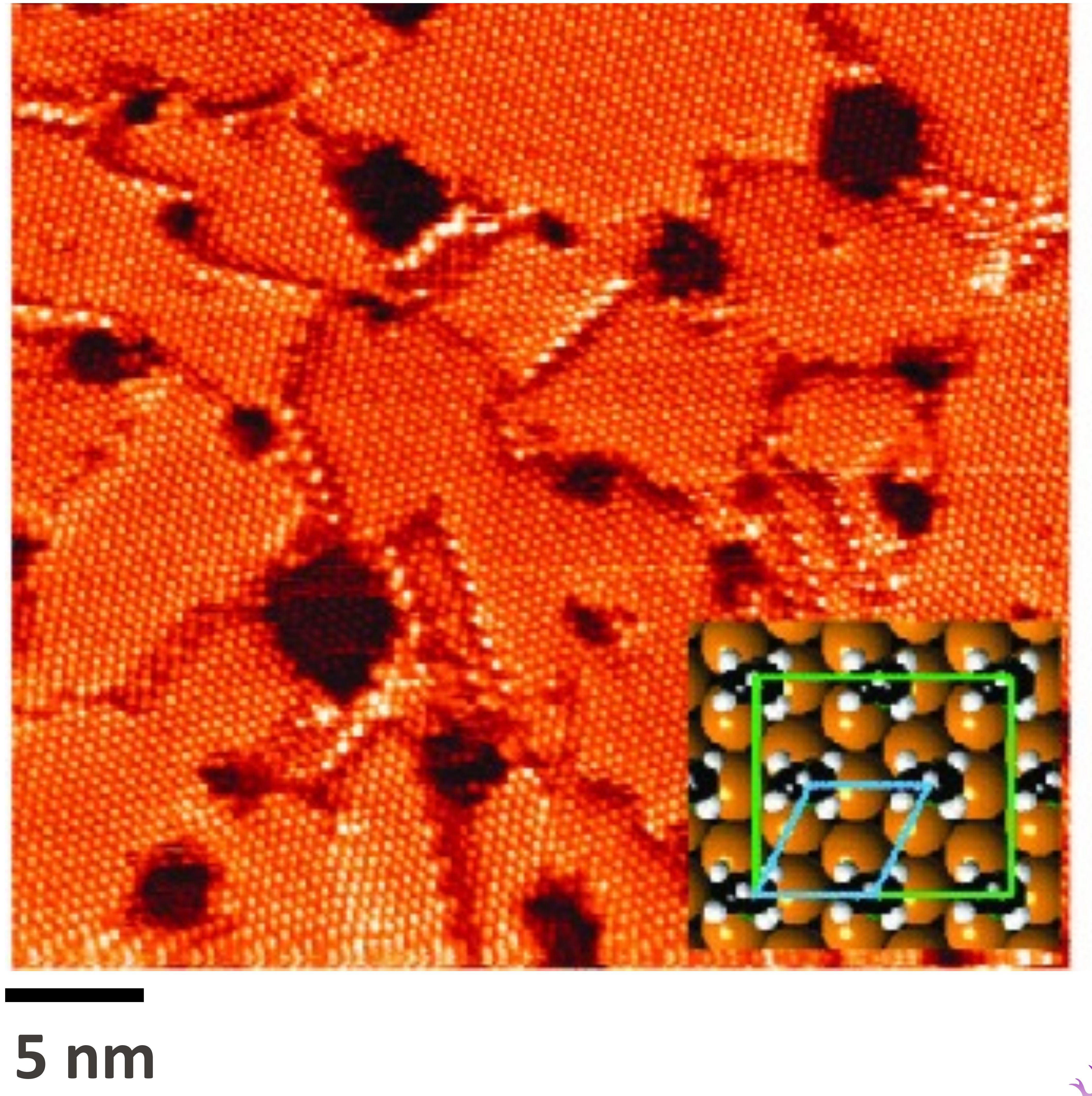
How Do We Characterize SAMs?

Visualize *via* scanning tunneling microscopy (STM)

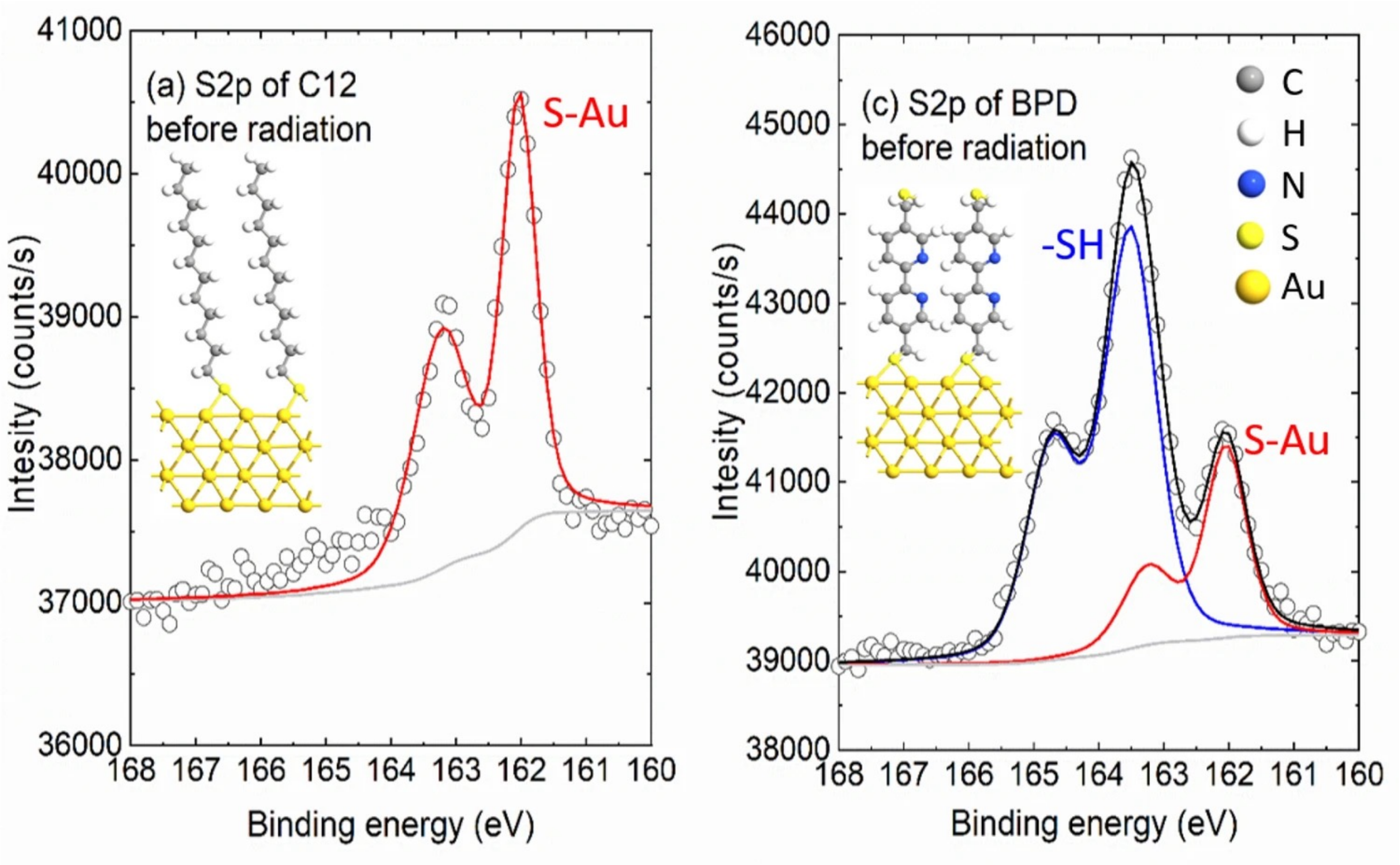


Guo & Li | *Phys. Chem. Chem. Phys.* | 2014



How Do We Characterize SAMs?

Chemical composition, orientation, film thickness *via* X-ray photoelectron spectroscopy (XPS)



Tong *et al.*| *Sci. Reports*| 2021

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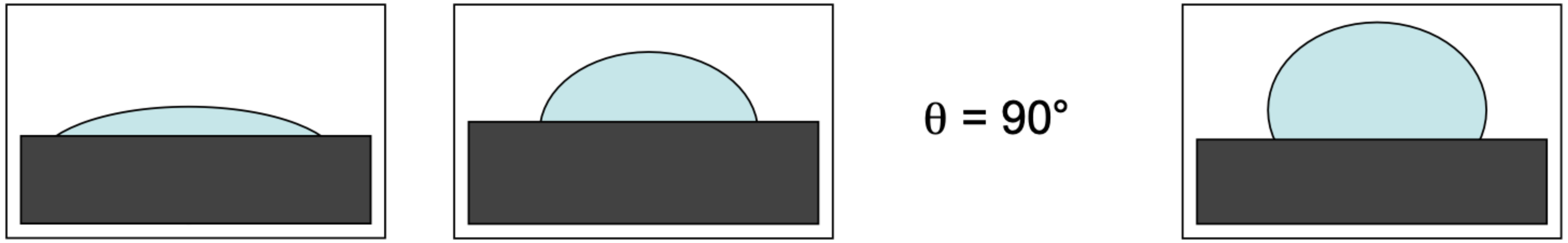
How Do We Characterize SAMs?

Surface energy (hydrophibicity/hydrophilicity) *via* contact angle

- Teflon: 110°
- Glass (untreated): 40°
- Glass (cleaned): wetting (< 5°)

- Full CH_3-terminated SAM: 110°
- Full OH-terminated SAM: 20°
- Mixed OH/CH_3-t. SAMs: $20° < \theta < 110°$



hydrophilic hydrophobic

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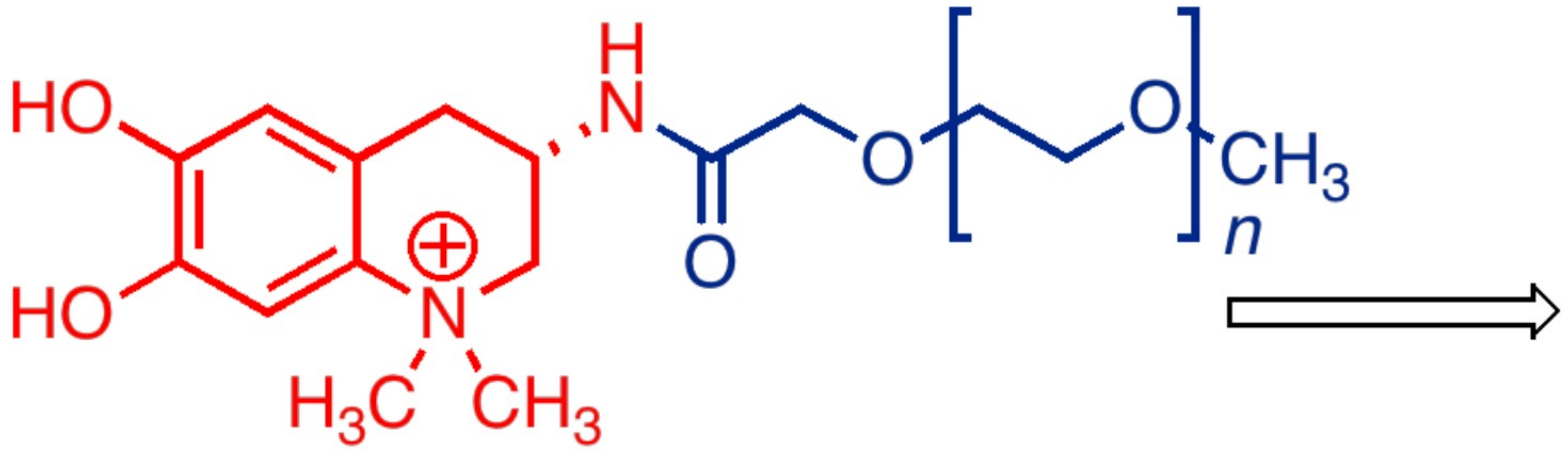
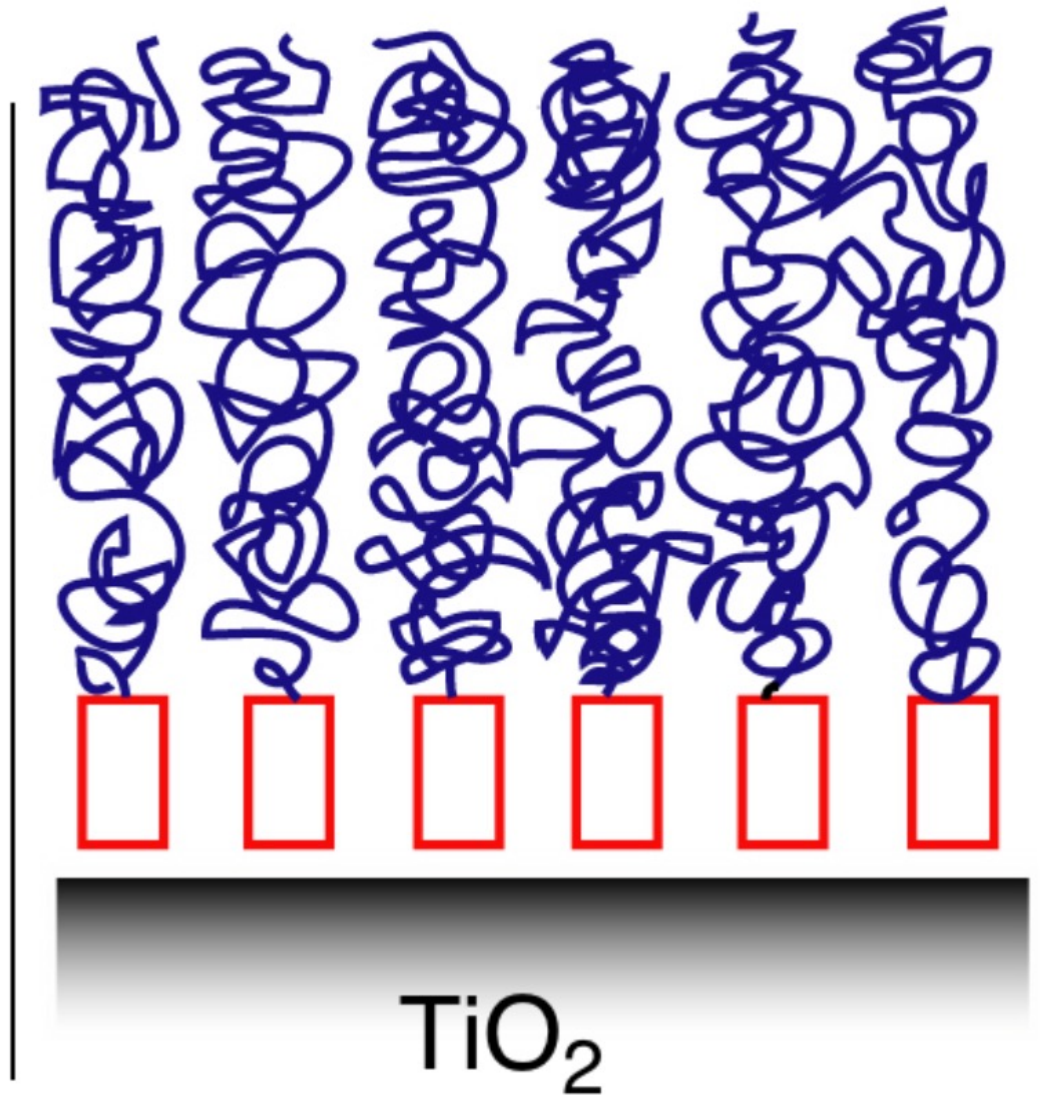
Is this System a SAM?





Flexible polymer chain

Catechol-based system

Catechol groups have high affinity for metal oxide surfaces

Formation of stable, oriented, and packed monolayer

Zürcher et al., | *J.Am.Chem.Soc.* | **2006**

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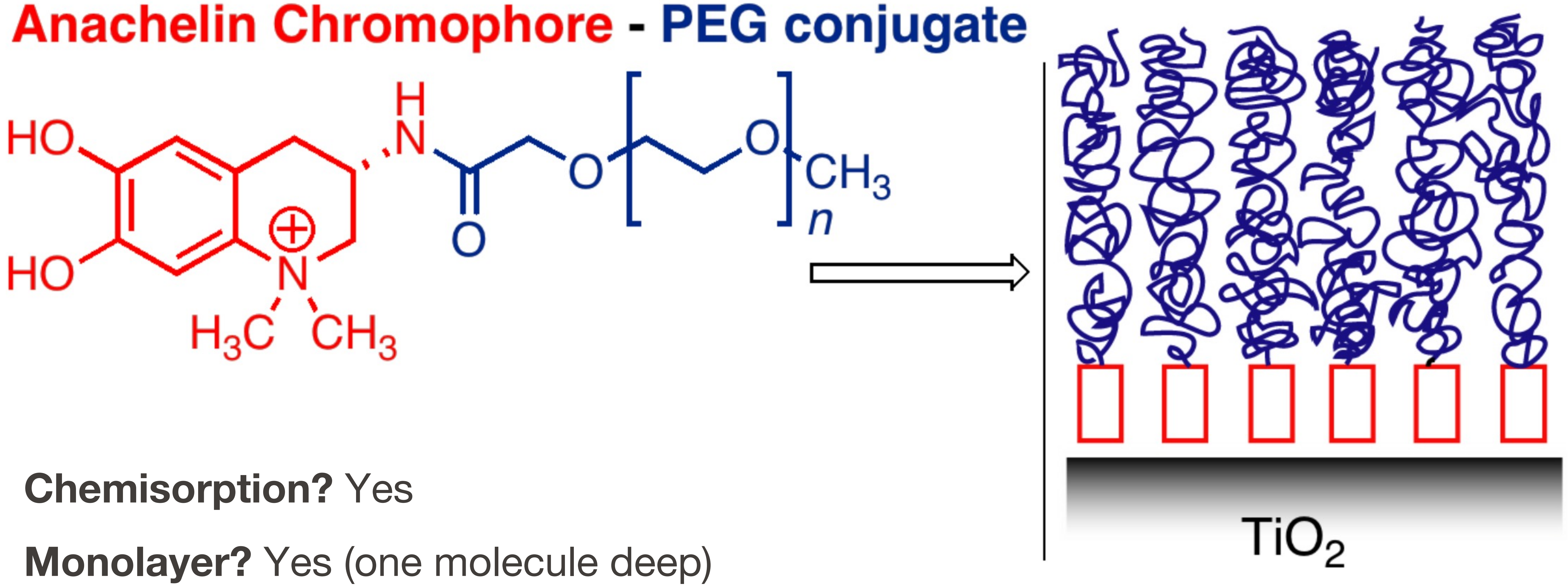
"SAM-Like" Polymer Brush

Anachelin Chromophore - PEG conjugate



Chemisorption? Yes

Monolayer? Yes (one molecule deep)

Ordered like a SAM? No ➔ It forms a polymer brush

✓ Anchored surface coating

✗ Not a classic SAM

Zürcher et al., | *J.Am.Chem.Soc.* | **2006**

Correct terminology: Catechol-anchored PEG polymer brush on TiO_2

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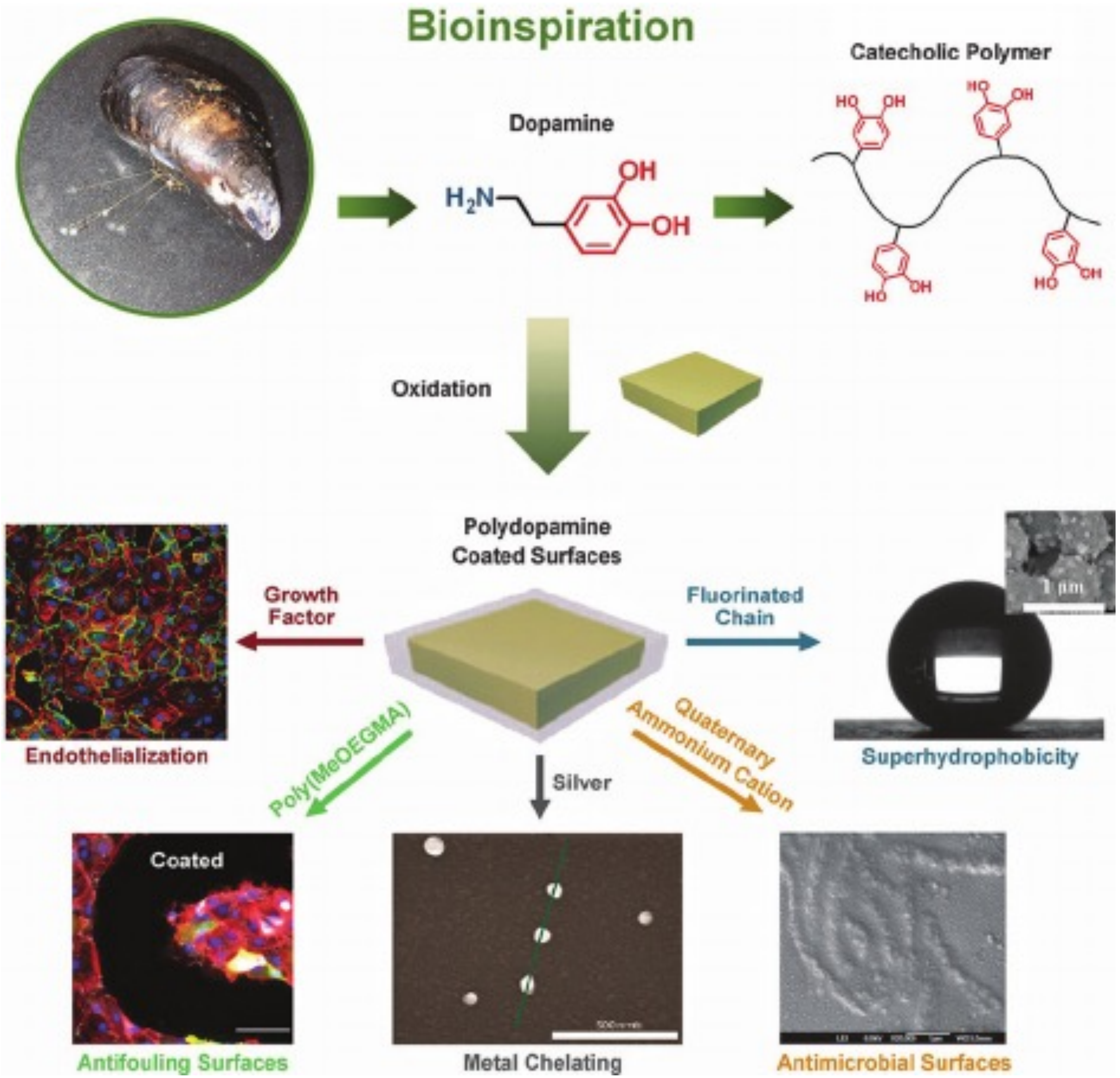
Catechol-Based Assembly - Polydopamine



Wei et al. | *Mater. Horiz.* | 2015

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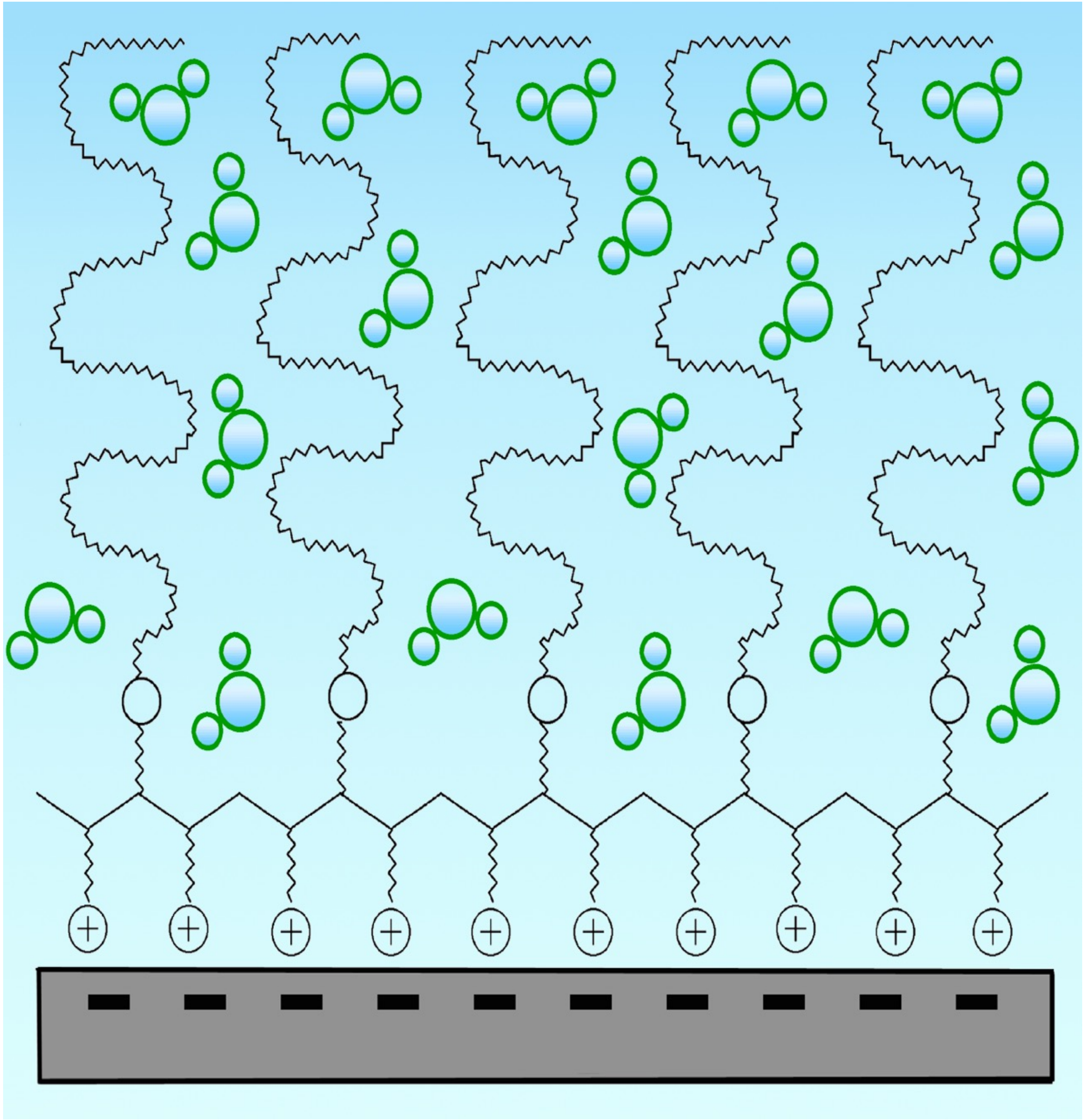
Is this System a SAM?

Grated polymer assembly of PEG molecules

Poly(*L*-lysine)-*graft*-poly(ethylene glycol)
(PLL-*g*-PEG)

Monolayer formed on metal oxide surfaces due to electrostatic interactions



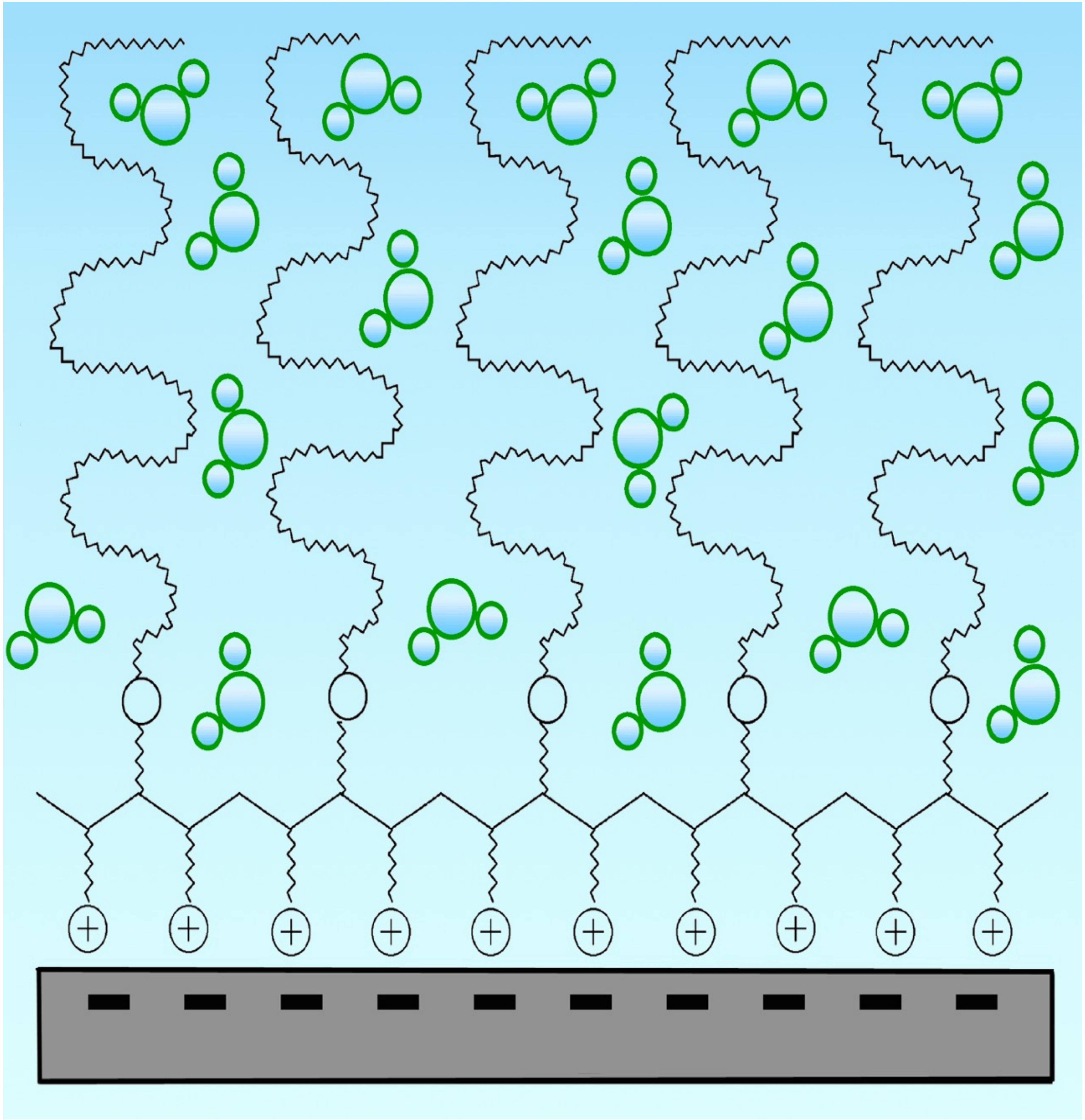
Grafted Polymer Assembly of PEG Molecules Not a SAM

No!

PLL-*g*-PEG is grafted along the positively charged backbone of PLL to attach through electrostatic interactions to negative oxide surfaces

SAMs are dense, highly ordered monolayers.

Alternatively, PLL-*g*-PEG forms a more flexible, brush-like structure leading to more hydration.



PEG side chains

PLL backbone

Metal oxide surface

SuSoS SURFACE TECHNOLOGY

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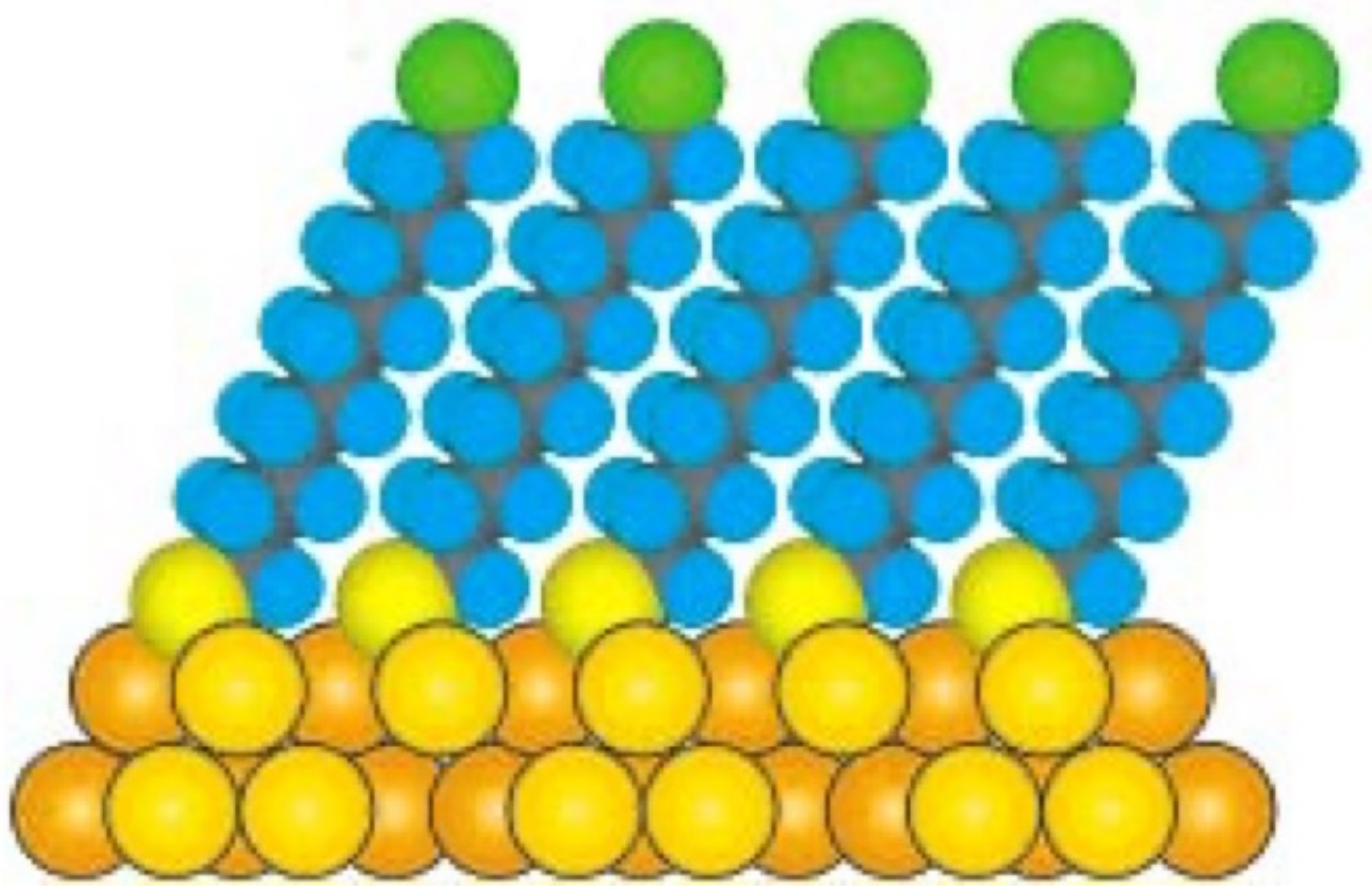
So Then What Constitutes A SAM?

Spontaneously adsorbed, single layer of species with a high degree of lateral organization



- A single layer of molecules
- Chemisorbed to a surface (*e.g.*, Au–thiol, Si–silane)
- Well-defined, ordered packing and specific molecular orientation
- Usually small molecules, not long flexible polymers

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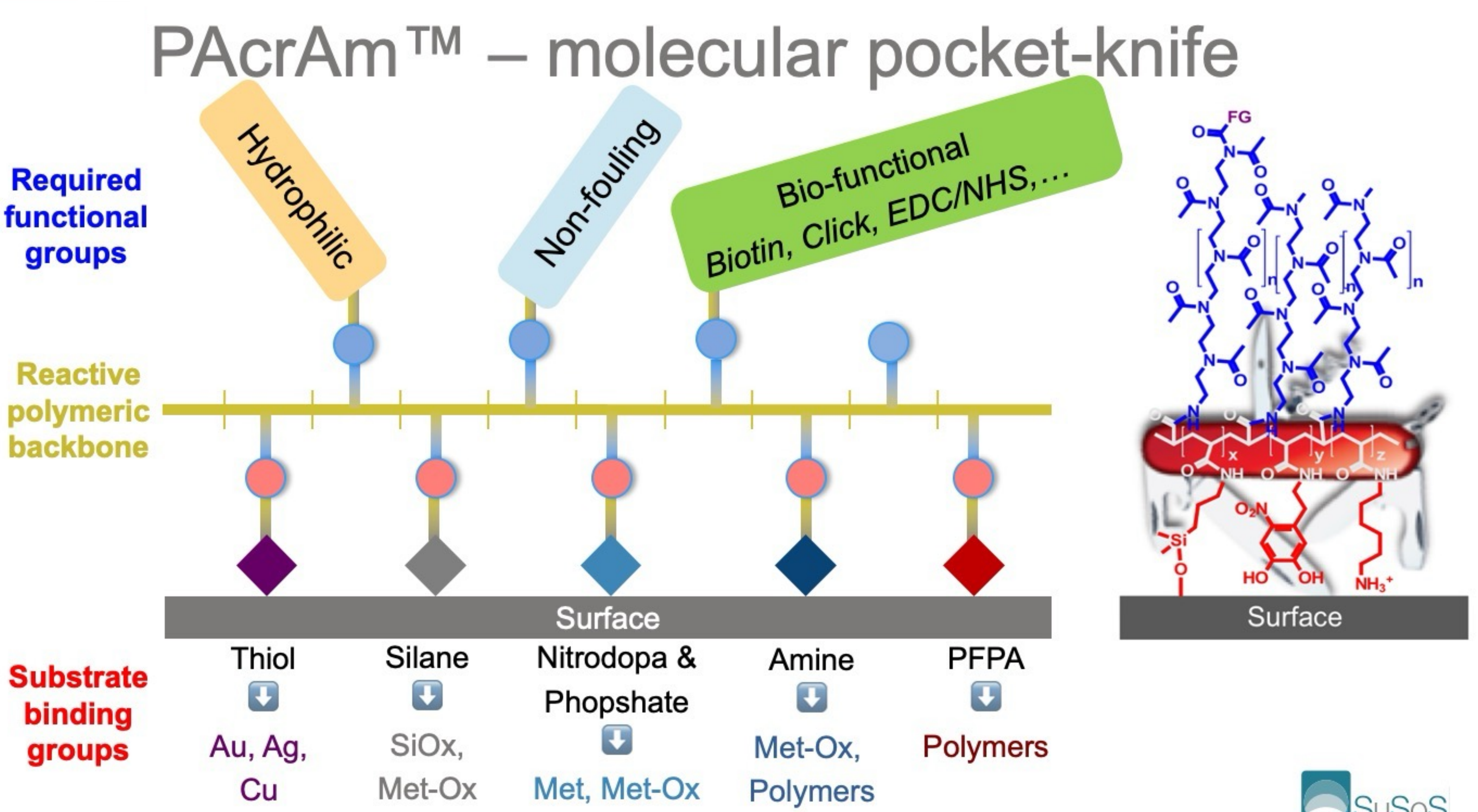
Key Takeaways

- We employ surface chemistry in our research (and many others do too!)
- Defects found on surfaces influence the formation of SAMs
- We can characterize SAMs using STM, XPS, contact angle
- The definition of a SAM may sometimes be confusing depending on system

Next Week: "Swiss Army Knife" of Polymer Surface Chemistry



Summary of Today's Class

- Why and how we functionalize surfaces – tuning properties!
- Alkanethiols, alkylsilanes, carboxylic acids – pros and cons and material selection
- Surface defects/lattices influence the assembly of molecules
- The tools we have learned prior can help us to characterize SAMs
- Surface chemistry is cool and can help us to learn how to work at the interface ☺

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https://www.epfl.ch/labs/chemina/