Self-Cleaning Coatings for Transparent Glass: A Systematic Review

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Dedicatoria

Dedico este trabajo de grado a mi familia, quienes desde el comienzo de mi pregrado en ingeniería química estuvieron apoyándome y me dieron todas las herramientas para culminar este trabajo.

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Resumen

Título: Recubrimientos autolimpiantes para vidrio transparente: revisión sistemática[*]

Autor: Juan Sebastian Carvajal Gelves y Francisco José Torres Martínez[**]

Palabras Clave: Superhidrofóbicidad, autolimpiante, vidrio, recubrimiento, revisión sistemática, transparente

Descripción: En los últimos años, el desarrollo de recubrimientos superhidrofóbicos autolimpiantes sobre vidrios transparentes ha sido un tema de gran interés debido a su amplia gama de aplicaciones que van desde fachadas de edificios hasta paneles solares. La implementación de esta tecnología permite reducir los gastos de mantenimiento asociados a la limpieza de estos dispositivos, además de mejorar su desempeño óptico. Esta revisión sistemática se realizó con el fin de identificar las tendencias y limitaciones actuales de esta tecnología, siguiendo los lineamientos relacionados con estudios sistemáticos del protocolo Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA). La recolección de información fue conducida en 5 bases de datos disponibles dentro de los recursos de la UIS: Web of Science (WOS), Scopus, Taylor & Francis, Springer, and ACS Publications. La filtración de los estudios pertinentes se llevó a cabo bajo 9 criterios de exclusión. En esta revisión, se lograron clasificar las técnicas de fabricación de 175 artículos en 3 categorías principalmente: Bottom-up, Top-down y la combinación de ambas aproximaciones. Posteriormente, se realizó un análisis comparativo de los métodos encontrados en función de 3 parámetros de gran interés identificados en la literatura: el desarrollo de procesos económicamente sostenibles y escalables, la capacidad de producir recubrimientos estables y duraderos para aplicaciones exteriores, y la incorporación de procesos más ambientalmente amigables. Luego, se abordaron los principales pretratamientos y postratamientos complementarios para alcanzar la propiedad autolimpiante deseada. Finalmente se presenta una discusión sobre el panorama actual de este tipo de recubrimientos recapitulando las tendencias científicas y exponiendo desafíos emergentes.

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[*] Trabajo de Grado

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Abstract

Title: Self-cleaning coatings for transparent glass[*]

Author(s): Juan Sebastian Carvajal Gelves y Francisco José Torres Martínez[**]

Key Words: Superhydrophobicity, self-cleaning, glass, coating, systematic review, transparent

Description: In recent years, the development of self-cleaning superhydrophobic coatings on transparent glass has been a topic of great interest due to its wide range of applications ranging from building facades to solar panels. The implementation of this technology allows reducing maintenance costs associated with the cleaning of these devices, also improving their optical performance. This systematic review was carried out to identify current trends and limitations of this technology, following the guidelines related to systematic studies of the Preferred Reporting Items for Systematic Reviews and Meta-Analyzes (PRISMA) protocol. The information collection was conducted in 5 databases available within the UIS resources: Web of Science (WOS), Scopus, Taylor & Francis, Springer, and ACS Publications. The filtering of the pertinent studies was carried out under 9 exclusion criteria. In this review, it was possible to classify the fabrication techniques of 175 articles in 3 main categories: Bottom-up, Top-down, and the combination of both approaches. Subsequently, a comparative analysis of the methods was performed based on 3 parameters of great interest identified in the literature: the development of economically sustainable processes, the ability to produce stable and durable coatings for outdoor applications, and the incorporation of processes more environmentally friendly. Then, the main complementary pre-treatments and post-treatments to achieve the desired self-cleaning property were addressed. Finally, a discussion on the current panorama of this type of coatings is presented, recapitulating scientific trends, and exposing emerging challenges.

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[*] Bachelor Thesis

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Introduction

The settling of airborne solid particles on a given surface, commonly referred to as “dust”, is a complex phenomenon favored by the action of gravitational forces and is influenced by multiple factors related to environmental conditions, the nature and size of the settled particles, the spatial orientation of the surface, as well as the speed and composition of the wind in the vicinity [1-3]. Dust deposition on surfaces is produced when the temperature at the solid interface is lower than the surrounding dry bulb-temperature. This phenomenon causes water condensation that acts as a solvent for particulate contaminants suspended in the air and gives way to the formation of a thin solid layer known as “dirt” [4]. The need of removing this film is mandatory in the transparent surfaces such as glass, and especially in solar cells or lens where it is important to restore its transparency for an optimal performance. However, the cleaning methods currently used, result expensive, complex and, in some cases, may compromise the mechanical, optical, or morphological characteristics of the vitreous surface [5].

In this regard, the conception of the self-cleaning effect from bio-inspired approaches has allowed the design of coatings that can prevent the adhesion of solid particles in an effective and economically sustainable way. Historically, the evolutionary ability of some plants to stay clean even in swampy environments to meet their water needs has been observed [6]. Particularly, the lotus leaf structure has received special attention from the scientific community becoming a reference for the fabrication of what are called "superhydrophobic surfaces" (SH), which allows the condensation or fall of water droplets to drag the dirt without any external energy or mechanical requirement. The latter is attributed to the presence of hierarchical micro-roughness and low surface energy materials that lead to a weak adhesion between the solid sediments and the interface of the surface [7].

In the literature, various methods can be found to easily manufacture such bio-inspired surfaces. However, it is still challenging to synthesize transparent superhydrophobic coatings. In general, there are two different approaches to achieve the morphology and surface energy that lead to superhydrophobicity: *i)* creating a rough structure on a hydrophobic surface; or *ii)* modifying a rough surface employing low surface energy compounds [8]. Nonetheless, in both cases a high final roughness is a key factor in achieving water repellency. Unfortunately, according to Mie's scattering theory and the studies carried out by Fei et al. [9], the roughness required for superhydrophobicity often leads to severe scattering of light, resulting in almost opaque or translucent surfaces due to approximations between the value of the mean square root of roughness (RMS) and the wavelength of light in the visible spectrum (400-700 nm) [10].

In addition to the existing challenge between the transparency and superhydrophobicity discussed above, self-cleaning coatings present several limitations in practical applications. Some of these drawbacks are: *i)* their poor mechanical durability, which leads to the production of mechanically robust surfaces that demand high resistance to wear, abrasion, and scratch [11]; *ii)* their lack of chemical stability, that is necessary for outdoor applications where the coatings have to be stable in a wide range of pH; and *iii)* at the same time, it is increasingly important to mitigate the environmental impact generated by fluorinated and hydrocarbon precursors used to reduce the surface free energy [12,13].

Basis on these premises, this paper aims to provide a better understanding of the current knowledge on the development of self-cleaning coatings on transparent glass, identifying the main fabrication techniques and raw materials, and recognizing the current challenges for future research and practical applications. A systematic review was performed following guidelines of the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA), which is a protocol developed with the intent to increase the transparency and accuracy of literature reviews [14].

1. Objectives

1.1 General Objective

To perform a systematic review of self-cleaning coatings for transparent glass in accordance with the PRISMA protocol.

1.2 Specific Objectives

To carry out a bibliometric analysis of the scientific publications related to self-cleaning coatings for transparent glass since 2010.

To provide an update and comprehensive review of the last scientific advances and technological trends in the field of study.

To identify the limitations and challenges in the field of study that may serve as a basis for the approach of future research proposals.

2. Methods

Figure 1

Methodology steps employed in the present document

2.1 Identification of databases available

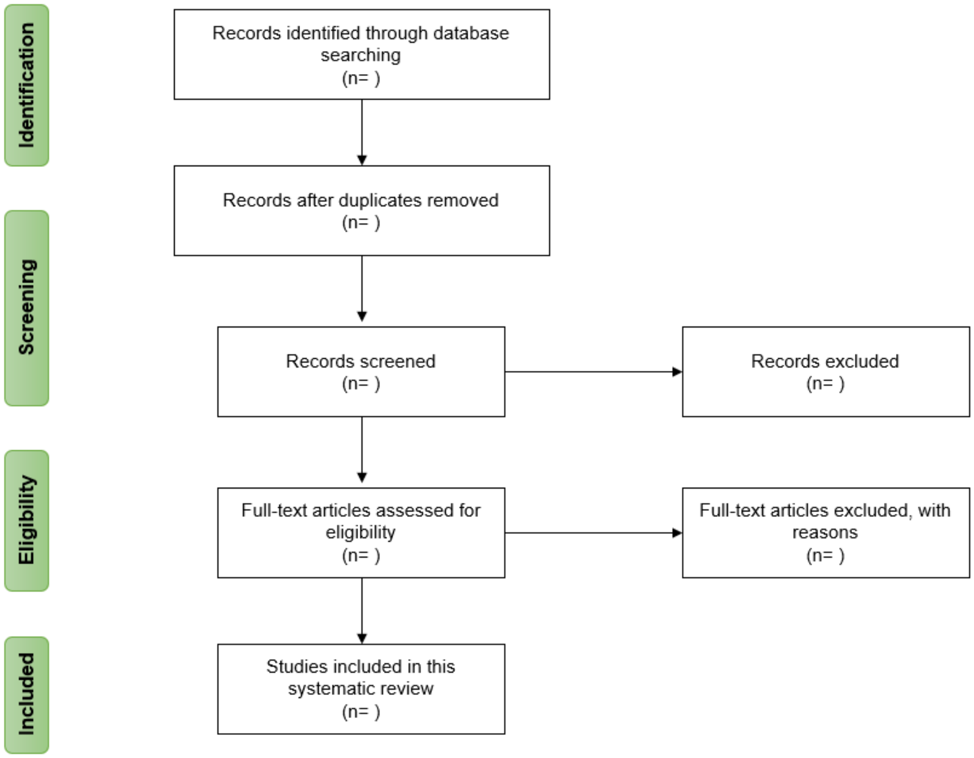
The search was performed using the databases available within the electronic resources of the Industrial University of Santander, i.e., Web of Science (WOS), Scopus, Taylor & Francis, Springer, and ACS Publications.

2.2 Gathering and filtration of information

This systematic review was conducted under the guidelines of the PRISMA protocol [14] and following the recommendations of the articles published by Saeed Pahlevan-Sharif et al. [15], [16], guide documents for conducting systematic reviews. The protocol consists of a checklist of 27 items and a flow diagram (Figure 2). It is important to clarify that the items related to meta-analysis were omitted in this paper.

Figure 2

PRISMA diagram followed in this systematic review



2.2.1 Identification

Initially, it was necessary to create a query and adequate it to each database. Afterwards, the records obtained from the different databases were exported to the free access reference manager “Zotero”, in which the detection and elimination of duplicates were performed.

2.2.2 Screening

The unique records were exported to the free access software “Rayyan” [17]. Using this tool, two reviewers screened the titles and abstracts of all papers independently. The articles had to pass the inclusion and exclusion criteria. During this phase, disagreements between reviewers were discussed until a consensus was reached. If an agreement could not be reached, a third reviewer joined the discussion, and his opinion was taken into consideration.

2.2.3 Eligibility and Included records

Full texts of the potentially eligible studies were examined individually by two authors for compliance with the exclusion criteria. The remaining records were included in this systematic review.

2.3 Bibliometric analysis

A bibliometric analysis was carried out using Power BI line-stacked and donut charts based on the data collected in the previous step, grouping in an Excel spreadsheet the articles by year of publication, research center, countries that have published on the subject, and main author.

2.4 Recognition of advances and technological trends

The main information of the included records was registered in an Excel spreadsheet that subsequently allowed them to be classified based on the synthesis methods, precursors used, the existence of pre-or post-treatment, and other features of interest. This facilitated the identification of advances in the manufacture of this type of coatings.

2.5 Discussion of current challenges

According to the data collected in the steps above, challenges and outlooks for future investigations were presented.

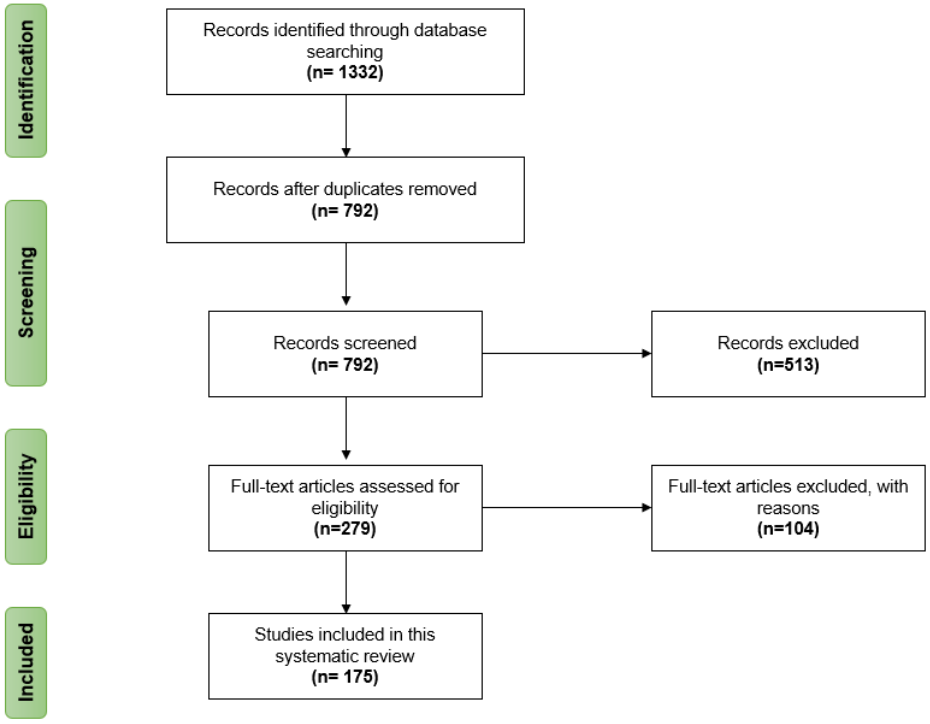
3. Results

3.1 Information gathering and filtering

Results obtained in each stage of this process are presented in the Figure 3.

Figure 3

Results obtained through the gathering and filtration stage



3.1.1 Identification

For this study, articles published in Spanish or English related to the synthesis of self-cleaning coatings on glassy surfaces in the last 11 years (2010- June 2021) were analyzed. Initially the query was designed in WOS and is shown in Equation 1.

(((((TS=(self-clean NEAR (*glass* OR window*))) OR TS=(anti-soil* NEAR (*glass* OR window*))) AND TS=(coating* OR film* OR surface*)) AND PY=(2010-2021)) AND LA=(English OR Spanish))* *Eq (1)*

The papers found by this query had to contain these terms in their titles, abstracts, and keywords. A preliminary query was carried out using a similar one without the "NEAR" operator. Then, a sample of non-common records between the two queries was reviewed to assess the effect of this operator on the records found. Title, authors and abstract of the articles were exported to an Excel spreadsheet (207 articles were screened). From this preliminary test, it was decided to remove the "NEAR" operator because this operator requires that the words linked to it are no more than 15 words apart. Thus, many results related to the aim of the research ended up being excluded. Furthermore, it does not exist in other databases such as ACS Publications or Springer, compromising its replicability. On the other hand, this preliminary query allowed to identify additional relevant terms for the query such as "screens, lens and solar cells", which are other types of glassy surfaces of interest. Another term of great importance was identified as "hydrophobic" since this systematic review was focused on obtaining the self-cleaning property employing this kind of surface, these terms were included in Equation 2.

(((((((TS= (self-clean OR anti-soil* OR anti-wet*)) AND TS= (*glass* OR window* OR screen* OR lens* OR "solar cell*")) AND TS= (coating* OR film** *Eq (2)*

OR surface*)) AND TS=(*hydrophobic*)) AND PY= (2010-2021)) AND LA= (English OR Spanish))

It is important to mention that in the Taylor & Francis and ACS Publications databases the terms of the query were contained only in the abstract, and for Springer database just articles containing these words in their title were reported due to the limitations of each tool. The summary of the queries employed is presented in Table 1. The last search was run on June 10, 2021. A total of 1332 articles were exported to Zotero, where it was possible to detect and eliminate 540 duplicate documents.

Table 1

Queries employed in this systematic review

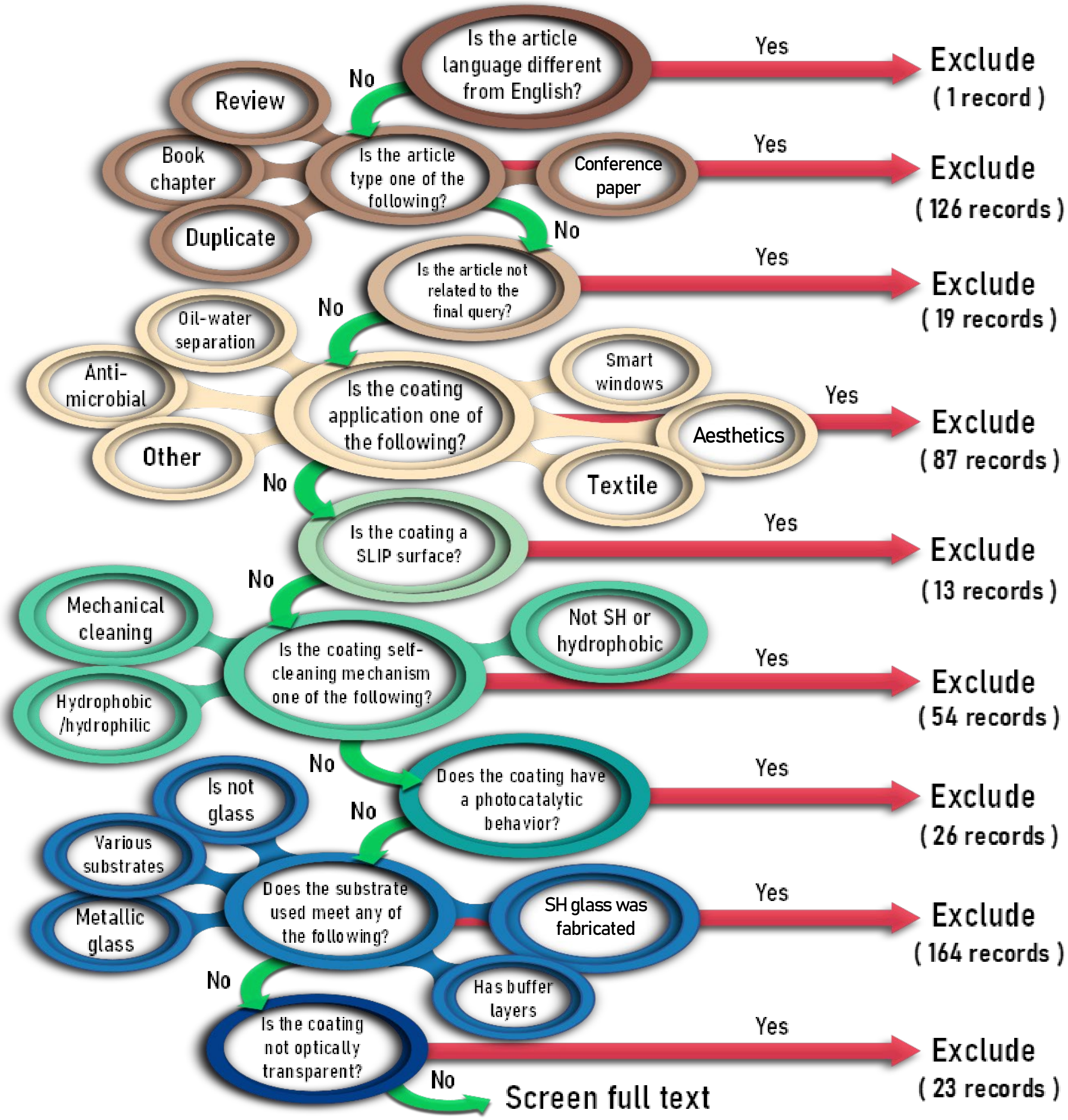
Query	Database	Records
(((((((TS= (self-clean* OR anti-soil* OR anti-wet*)) AND TS= (*glass* OR window* OR screen* OR lens* OR "solar cell*")) AND TS= (coating* OR film* OR surface*)) AND TS=(*hydrophobic*)) AND PY= (2010-2021)) AND LA= (English OR Spanish))	WOS	593
TITLE-ABS-KEY(self-clean* OR anti-soil* OR anti-wet*) AND TITLE-ABS-KEY(*glass* OR window* OR screen* OR lens* OR "solar cell*") AND TITLE-ABS-KEY(coating* OR film* OR surface*) AND TITLE-ABS-KEY(*hydrophobic*) AND LANGUAGE(English OR Spanish) AND PUBYEAR > 2009	SCOPUS	612
[[Abstract: self-clean] OR [Abstract: anti-soil] OR [Abstract: anti-wet]] AND [[Abstract: glass] OR [Abstract: window] OR [Abstract: lens] OR [Abstract: screen] OR [Abstract: "solar cell"]] AND [[Abstract: coating] OR [Abstract: surface] OR [Abstract: film]] AND [Abstract: hydrophobic] AND [Publication Date: (01/01/2010 TO 12/31/2021)]	Taylor & Francis	12
(self-clean OR anti-soil OR anti-wet) AND (glass OR window OR lens OR screen OR "solar cell") AND (coating OR surface OR film) AND (hydrophobic)	ACS Publications	52
(self-clean* OR anti-soil* OR anti-wet*) AND (*glass* OR window* OR lens* OR screen* OR "solar cell*") AND (coating* OR surface* OR film*) AND (*hydrophobic*)	Springer	63
TOTAL		**1332**

3.1.2 Screening

At first, two independent reviewers screened the titles, keywords, and abstracts of all records (792) separately and a hierarchical decision tree scheme was established (shown in Figure 4) to organize the exclusion reasons before performing a full text screening. Records that met one of the following reasons were excluded: *i*) if the record language was different from English; *ii)* if the record type was a conference paper (it loses reliability when published without peer review, commonly performed by scientific journals); also*,* reviews, book chapters and duplicates that Zotero could not identify; *iii)* if the record topic was vaguely or not related to the final query of this review; *iv)* if the record coating application was not focused on self-cleaning functional glass for anti-soiling (e.g., oil-water separation, textiles, anti-microbial surfaces, etc.) or corresponded to smart windows; *v)* if the record coating was a Liquid Infused Porous Surface (SLIPS); *vi)* if the record self-cleaning mechanism did not come from a hydrophobic, superhydrophobic, amphiphobic or omniphobic behavior but exclusively a hydrophilic, superhydrophilic, oleophobic or superoleophobic one; in addition, switchable hydrophobic/hydrophilic coatings and publications with a mechanical cleaning mechanism (e.g., vibrations, robots, etc.) were not taken into account neither; *vii)* if the record coating had a photocatalytic behavior; *viii)* if the substrate mentioned in the record was not transparent glass (metallic glass, polymers or metals), had buffer layers, was a manufactured superhydrophobic glass or the glass substrate was used among other substrates; and, *ix)* if the coating mentioned in the record was semi-transparent, translucent, or opaque. As a result of this screening process a total of 513 records were excluded leaving 279 records.

Figure 4

Hierarchical decision tree for screening titles, abstracts, and keywords



3.1.3 *Eligibility and included records*

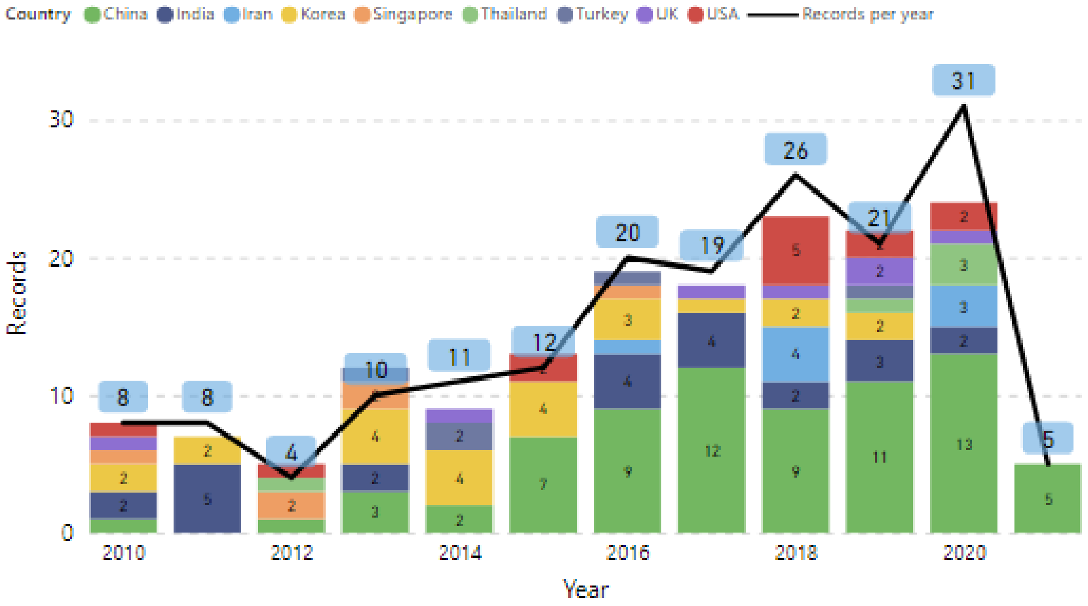
Regarding the number of records remaining there was a total of 104 records excluded in this step. Some articles were inaccessible for full text reading (48 out of 279 records), while the remaining 56 did not meet the inclusion requirements described in Figure 4 (53 records) or did not provide a detailed description of the coating fabrication (3 records). As a result, 175 records were finally included in this systematic review.

3.2 Bibliometric analysis

The research on self-cleaning transparent SH coatings for functional glass has grown significantly over the past 10 years, increasing from 8 articles published in 2010 to 31 published in 2020 (Figure 5). The latter may be due to the great potential of these coatings to reduce maintenance costs or increase the durability of a wide range of daily life devices made of glass. It is important to mention that the records from 2021 are not representative because this systematic review only considered the articles published up to June 10 of this year.

Figure 5

Publications per year and participation of main countries



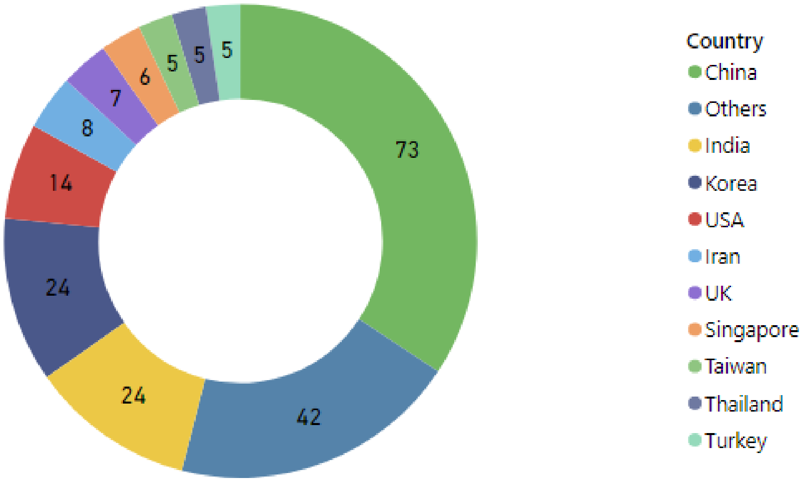
Particularly, China, among the rest of the countries, have been showing great interest that is reflected in the growing trend of its number of publications along the last decade. Furthermore, some countries such as South Korea, India or USA followed a certain periodicity in the publication of records.

Regarding the global data of the entire time interval considered in this study, the region with the most overall records is the Asian continent. China is present in 73 of the 175 (41.7%) publications analyzed, followed by Korea and India with 24 (13.7%) each, as shown in Figure 6. These countries participate in 69.1% of the papers, with their main research centers located in universities such as the Chinese Academy of Sciences (6 records), Shivaji University (5 records), South China University of Technology (5 records), Jilin University (3 records), Kyung Hee University (3 records) and Seoul National University (3 records), among others. Some of these institutions equal or even exceed the number of articles published by various western nations individually. It should be noted that Figure 6 shows a total sum of 213 records rather than the 175 records included in this review because some articles are international collaborations between different colleges from two or more countries.

Countries that have less than 5 publications related to transparent SH self-cleaning coatings on glass, including Malaysia, Saudi Arabia, Italy, Germany, Spain, Canada, Indonesia, Brazil, among others, giving a total of 24 different countries are listed as "others" and represent about 20% of the total of 213 participations.

Figure 6

Number of participations per country in the included records



3.3 Recognition of advances and technological trends

3.3.1 Fundamentals of wetting states

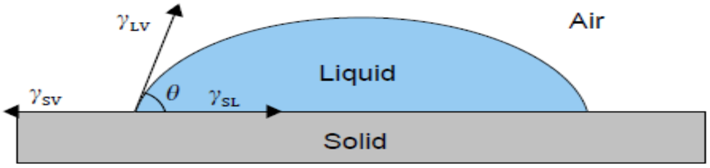
There are several parameters to measure wettability, such as contact angle (CA), contact angle hysteresis, sliding angle, shedding angle, among others. The CA is the main factor to determine the wettability state, because it is an indicator of the interfacial forces of the 3 phases (solid-liquid-vapor) that are in contact during the thermodynamic equilibrium. Depending on the measurement of this parameter, surfaces are categorized as superhydrophilic (0 ° < CA <10 °), hydrophilic (10 ° < CA <90 °), hydrophobic (90 ° < CA <150 °) and superhydrophobic (150 ° < CA <180 °) [18].

The main properties that affect the wetting state of the surface are surface chemistry and surface roughness. The chemical structure of solid materials influences what is called “surface free energy”, which is the resulting energy on the surface derived from the unbalanced interactions of the molecules in the boundaries of the bulk that generates a net force towards the interior. In general, low surface energy decreases the adhesion force of the surface with water molecules, promoting the non-wettability state [9]. The latter is also favored by the design of highly rough surfaces, which can trap more air, decreasing the contact area between the solid-liquid phases and increasing the CA. In this regard, four surfaces are commonly considered: flat, microstructure, nanostructure, and hierarchical (a combination of micro-nano structure) [8]. Gao et al. [19] found that the hierarchical structure presents the greatest superhydrophobic properties due to its kinetic and thermodynamic performance.

The wettability of surfaces is a highly complex phenomenon, therefore several theoretical models of the behavior of drops on surfaces have been developed. Young's model was the first model proposed and served as the basis for later models [8,18]. This model does not consider the roughness of the surfaces; hence, it describes the behavior of a drop on an ideally flat surface. Figure 7 shows the thermodynamic equilibrium between the three existing interfaces: solid-liquid (SL), liquid-air (LV) and solid-air (SV). Also, the balance between forces in the x-axis is represented in Equation 3, being γ the surface tensions of the corresponding interfaces and θ represents the CA [20].

Figure 7

Force balance of interfacial tensions for Young's Model



Note. Taken from: Yilbas et al. [20].

$$\sum F_x = \gamma_{SL} + \gamma_{LV}\cos\theta - \gamma_{SV} = 0 \qquad \text{Eq (3)}$$

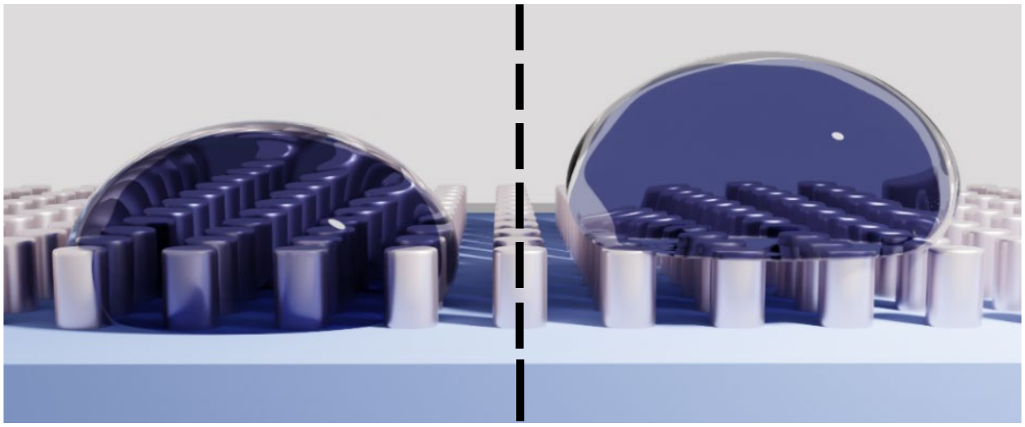
This equation can be rearranged as:

$$\cos\theta = \frac{\gamma_{SV} - \gamma_{SL}}{\gamma_{LV}} \qquad \text{Eq (4)}$$

From Equation 4, a surface can improve its hydrophobicity (a CA increase) by decreasing the solid-air interfacial tension. However, Young's model does not take into account factors such as roughness and other surface conditions that are present on most non-ideal surfaces. For this reason, the Wenzel model was developed, in which is assumed that the liquid is in complete contact with the solid surface, even going through the holes generated by roughness (Figure 8a) [6]. The relation between the CA of Wenzel's model to Young's model is presented in Equation 5, where θ^{w} represents the CA on a rough surface and r is the surface roughness factor, which is defined as the ratio between the actual surface area and the flat projected area. For smooth surfaces, r = 1 but real surfaces are not smooth at the molecular level, thus r will always be greater than 1 [21]. This equation allows to evidence two relationships (Equation 6 and 7).

Figure 8

3D representation of a water droplet on a rough surface following a) Wenzel's model; b) Cassie-Baxter's model



$$\cos\theta^{w} = r\cos\theta \qquad \text{Eq (5)}$$

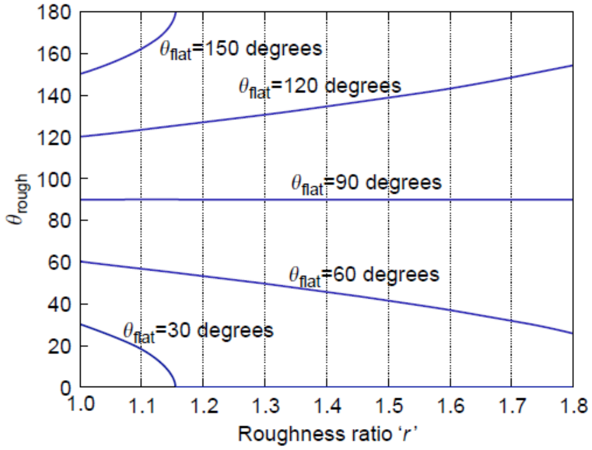
$$If\ \theta > 90, \qquad then\ \theta^{w} > \theta \qquad \text{Eq (6)}$$

$$If\ \theta < 90, \qquad then\ \theta^{w} < \theta \qquad \text{Eq (7)}$$

Physically, it means that a hydrophobic surface will become more hydrophobic when roughness is considered. By contrast, a hydrophilic surface will be more hydrophilic when roughness is introduced. This can be seen more clearly in Figure 9.

Figure 9

Influence of roughness ratio on Wenzel's CA



Note. Taken from: Yilbas et al. [20].

The assumption considered by Wenzel that the liquid totally wets the surface (homogeneous solid-liquid interface) represents a disadvantage for hydrophobic surfaces, since the complete submersion of the structure into the liquid, relatively increases the energy of the system, being less energetically favorable for this type of surface [20]. Herein the Cassie-Baxter model appeared, incorporating the effect of air pockets trapped in highly rough surfaces, thus the liquid will be in contact with a heterogeneous solid-liquid-air interface (Figure 8b) that fits quite well to the behavior of the drops on the lotus leaves [9]. The CA for the Cassie-Baxter model (θ^{CB}) is influenced by the fraction of the surface that is in contact with the liquid (f_1) and the fraction of the liquid-air interfacial area into the grooves (f_2). The mathematical model is presented in Equation 8, where θ_1 and θ_2 are the CA between the liquid-solid and the liquid-air pockets, respectively.

$$\cos\theta^{CB} = rf_1\cos\theta_1 + f_2\cos\theta_2 \qquad Eq\ (8)$$

State of non-wettability ($\theta_2 = 180°$) can be considered between the air and water, leading $\cos\theta_2 = -1$ and with the relation of fractions: $f_1 + f_2 = 1$. Then, the Equation 9 is obtained:

$$\cos\theta^{CB} = rf_1\cos\theta_1 - (1 - f_1) \qquad Eq\ (9)$$

Where is possible to notice that a superhydrophobic surface can be obtained by decreasing the interfacial area between the liquid-solid and increasing the surface roughness.

3.3.2 Fabrication processes and materials

Fabrication of self-cleaning transparent SH can be reached from two different approaches: Bottom-Up and Top-Down. The first approach is based on the growth or deposition of "building blocks" on a substrate and may have different shapes such as nanorods, nanotubes, raspberry-shaped particles, among many others [22,23,24]. These are regularly synthesized from a mixture of compounds, which individually contribute to achieve the necessary combination of roughness and low surface energy. An example is a solution of SiO_2 nanoparticles giving the roughness, and a fluorinated compound that decreases surface energy. Generally, coatings produced by Bottom-up techniques are thought to have poor durability, since micro/nano structures are not part of the substrate or a bulk solid. In contrast, in the Top-Down approach, the roughness is originated directly on the coating material by carving, machining, or molding; thus, allowing to obtain greater durability [9]. Furthermore, a combination between both techniques can be performed to obtain hierarchical nanostructures by generating a topographic pattern onto the coating (Top-down) and sequentially growing or depositing different types of nanoparticles (Bottom-up) on the top of the rough surface.

In this systematic review, a total of 23 different techniques for fabricating transparent SH coatings on glass were identified. Bottom-up approaches (139 records distributed in 20 techniques), Top-down approaches (32 records distributed in 3 techniques) and combinations of both (4 records with independent combinations) are listed on Table 2. The fabrication methods are organized vertically by the number of records from highest to lowest in the approach which they belong to. The numeric results by technique are shown in Figure 10.

Table 2

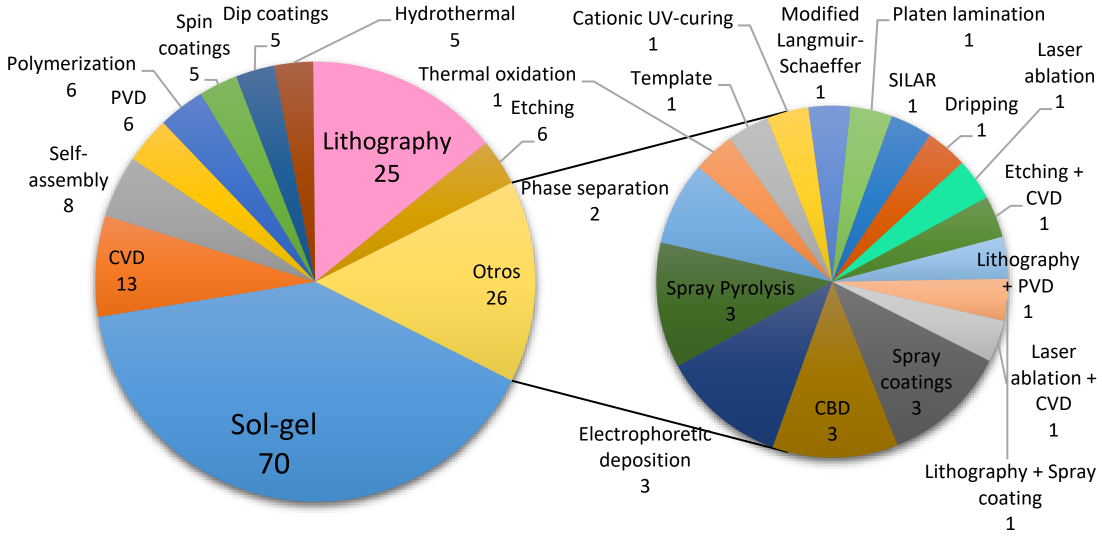
Summary of fabrication methods and synthesis comparative features by technique

General classification			Synthesis route features		
Approach	**Fabrication or deposition technique**	**Records by technique**	**Cost-effective synthesis**	**Eco-friendly synthesis**	**Robust coating synthesis**
Bottom-up	Sol-gel	[25-94]	[27,30-32,35,36,38-43,45,46,48,51-53,56,59,61,63-65,68,71,73-75,77,80,82,84,85,87-90,92,93]	[34,38,41,42,44,45,61,63,68,71,74,76,77,84,88,90,93]	[25-28,30-32,34-38,41,44,47-50,54,56-65,69,70,72,73,75,79,80,83-85,87-89,93,94]
	CVD	[95-107]	[95,100,101,105]	[96,99,100]	[96-104,106]
	Self-assembly	[108-115]	[108,109,112,114]	[109,112,114,115]	[108,109,112-115]
	PVD	[116-121]	[117]	…	[117]
	Polymerization	[122-127]	[127]	[122]	[125-127]
	Spin coating	[128-132]	[131]	[131]	[128]
	Dip coating	[133-137]	…	…	[133-137]
	Hydrothermal method	[138-142]	[138,141,142]	…	[139,141]
	Spray coating	[143-145]	[143-145]	[145]	[143-145]
	CBD	[146-148]	[146-148]	[147]	…
	Electrophoretic deposition	[149-151]	[151]	…	[149-151]
	Spray Pyrolysis	[152-154]	[152-153]	…	…

General classification			Synthesis route features		
Approach	**Fabrication or deposition technique**	**Records by technique**	**Cost-effective synthesis**	**Eco-friendly synthesis**	**Robust coating synthesis**
	Phase separation	[155-156]	…	…	[156]
	Thermal deposition	[157]	…	…	[157]
	Template method	[158]	[158]	[158]	…
	Cationic UV-curing	[159]	[159]	…	…
	Modified L-S deposition	[160]	[160]	…	…
	Platen lamination	[161]	…	…	[161]
	SILAR	[162]	[162]	[162]	…
	Dripping	[163]	[163]	[163]	[163]
Top-down	Lithography	[164-188]	[164,168,169,171-177,180,181,187,188]	[169,173,179,186,188]	[164,166,167, 171,172, 181,182]
	Etching	[189-194]	[191], [194]	…	[193,194]
	Laser ablation	[195]	…	…	[195]
Bottom-up + Top-down	Etching + CVD	[196]	[196]	…	…
	Lithography + PVD	[197]	…	…	[197]
	Lithography + Spray coating	[198]	…	…	[198]
	Laser ablation + CVD	[199]	[199]	…	…

Figure 10

Main techniques found in this review



3.3.2.1 Bottom-Up

3.3.2.1.1 Sol-gel. As its name indicates, the method begins with the formation of a colloidal suspension (sol), followed by its gelation to form a network in a continuous liquid phase (gel), which is subsequently dried to obtain a denser product. A typical sol-gel process requires a metallic or metalloid source, a solvent, a catalyst, and water [200]. The most widely used precursors are metal alkoxides (M-OR) and especially alkoxysilanes due to their good solubility and stability in solution with organic solvents, easy purification, high compatibility with a wide variety of glasses, and the formation of a glass-like material, which prevents recycling problems on the treated glass [201]. The most common precursors are listed on Table 3. These compounds will carry out simultaneously hydrolysis and condensation reactions catalyzed by a basic or acid agent. In hydrolysis reaction, the metal alkoxide (M-OR) reacts with water, generating metal hydroxides (M-OH); then, in condensation reaction, these hydroxides (M-OH) react with each other or with metal alkoxides (M-OR) to finally obtain an inorganic network (M-O-M) and producing water and alcohols as by-products. In acid conditions the hydrolysis reaction will be catalyzed, promoting the formation of films; by contrast, in basic conditions the condensation reaction will be catalyzed, favoring the synthesis of nanoparticles [202].

Table 3

Main sol-gel precursors found in this review

Type of precursor	Precursor subcategory	Chemical compound	Reference
Solvent	Organic solvents	Methanol	[25,26,28,29,32,35,44,47,49,58]
		Isopropanol	[29,34,38,54,60]
		Ethanol	[25-29,31-33,35-49,51,52,54-60,62-67,69,70,72,75,77-84,86-89,91-94]
Catalyst	Basic catalyst	Aqueous ammonia	[29,32,33,35,36,38-40,43,45,47-49,51,54,56,57,59.60,62,63,66,69,70,74,75,79,83,84,86-88,90,92]

Type of precursor	Precursor subcategory	Chemical compound	Reference
	Acid catalyst	Hydrochloric acid (HCl)	[31,32,34,39,41,44,47,57,69,75,82-84,88,90,93,94]
Metal source	Metal alkoxides	Tetraethylorthosilicate (TEOS)	[27-29,31,32,36-40,42-49,51,54-57,59,60,63,67,69,70,75,77-79,81-83,86-88]
		Methyltrimethoxysilane (MTMS)	[25,28,32,35,58,62]

The sol-gel method is a versatile technique since it is possible to obtain organic-inorganic hybrid materials where the inorganic part fulfills the function of the backbone of the coating and the organic part can provide various functional properties. This is achieved by including co-precursors in the solution, such as organosilanes; among them chloroalkyllsilanes or fluoroalkylsilanes are of special interest due they play the role of hydrophobic agents.

3.3.2.1.2 Chemical Vapor Deposition (CVD). Films deposition by CVD method is carried out by means of the adsorption and heterogeneous reaction of the gas reagents with the solid interface of the substrate surface, followed by the release of the volatile by-products generated [203]. A reaction chamber that supports high temperatures is necessary since the reaction begins when the temperature is high enough (up to 1600 °C) or additional energy is introduced to the system, such as plasma. Generally, the process is operated at low pressures, to guarantee the purity in the deposition of the films [204]. It is possible to obtain uniform distribution on substrates with large surfaces and precise control of the coating thickness [205]. However, it is considered a costly technique for its operational conditions, thus variants of the technique have been developed, e.g., Plasma Enhanced CVD (PECVD) where the process temperature is lowered because the energy necessary to initiate the reaction is provided by an ionized gas [95], [99], [103-104]; Atmospheric Pressure CVD (APCVD) in which the reactor design is simpler without the vacuum requirement [98]; Aerosol Assisted CVD (AACVD) in which an aerosol liquid/gas is used as carrier of the precursors instead of a gas mixture, among others [101], [102], [105].

3.3.2.1.3 Self-assembly. Self-assembly is a reversible and spontaneous process by which the formation of nanostructures takes place from disordered components that are arranged forming defined and stable patterns [206]. In the present review, two main subcategories were identified for this technique: Layer by Layer (LbL) assembly and nanoparticle self-assembly.

Among the different types of self-assembly, LbL is the most popular [108-109], [111], [113]. This method typically consists of dipping the substrate alternately in positively and negatively charged solutions for several cycles to deposit several thin layers until the desired coating thickness has been achieved. Nevertheless, usually this is not enough to achieve the required roughness in a superhydrophobic coating, so it is common the deposition of nanoparticles during the process to be adsorbed by electrostatic forces in the previously deposited layers. This method can have certain variations as shown by Chen et al. [113] who performed a series of spray-coatings using first a trialkoxysilyl-substituted polymethyl (hydro)/polydimethylsilazane solution and later silicon nanoparticles functionalized with 1H, 1H, 2H, 2H-Perfluorodecyl-trichlorosilane (PFDTCS), repeating this bilayer coating for several cycles.

Another procedure used is the self-assembly nanoparticle. This is a process of physical organization between nanoparticles that interact with each other through attractive and repulsive forces to form agglomerations of different geometries on the surface of the substrate. As an example, Cho et al. [110] were able to fabricate raspberry-like structures using an emulsion of immiscible liquids (water and hexadecane) to confine islands of silicon nanoparticles in water droplets and thus induce self-assembly by evaporation. Additionally, Sarkar et al. [112] synthesized copper nanostrips in a single-stage process by depositing a solution of copper nanopowder, ethanolic tethradecanoic acid, and ethanol on the substrate and then allowing it to dry overnight.

3.3.2.1.4 Physical Vapor Deposition (PVD). PVD method is based on the ejection of atoms or molecules from a target material. These particles are released in the gas phase and are subsequently condensed on the substrate. Generally, a high vacuum is necessary to prevent undesired reactions with environmental agents such as oxygen or humidity [207]. There are several subcategories of this technique depending on how the atoms or molecules are removed from the target material. Among them: E-Beam Evaporation and Sputtering stand out [204], [208]. In the first one, electrons play the role of energy source, and they will bombard the target, releasing the atoms, ions or plasma that will be deposited [117-118], [120-121]. In the latter one, a potential difference is generated between the target material (cathode) and the substrate (anode) having an inert and heavy gas between them (usually Ar). The supplied voltage ionizes the gaseous cloud and these energized Ar particles bombard and tear off the target's atoms or molecules. This alternative is attractive due to the good adhesion obtained [116].

3.3.2.2 Top-Down

3.3.2.2.1 Lithography. Lithography is the most representative technique of the Top-Down approaches to generate nanostructures. There are several methods that belong to lithography classification, but the most widely used are: i) Photolithography and ii) Soft-Lithography. The first one specializes in the microelectronics industry, being generally more expensive due to the equipment required for the process. It also has the limitation of only being able to make patterns on photomask or photoresist films with poor control over surface chemistry [209].

On the other hand, soft lithography is a group of striking techniques due to its low implementation cost and wide versatility of generating patterns with a variety of micro/nano structures. These techniques are based on printing, molding, and embossing a pattern using a mold that can be rigid or flexible on soft surfaces such as polymers and gels [210], [211]. The most common soft lithography methods are: *i)* Nano Imprint Lithography (NIL), which uses a rigid mold to press a polymer in liquid phase that later will be solidified. If the solidification is carried out by thermal treatment it is called “Hot-embossed NIL” [165], [176], [182], [185], [187], but if UV-radiation is used (with a UV-curable polymer) it is called “UV-NIL” [175], [177-178]; *ii)* Replica Molding (REM), is a simple process where an elastomer is used (commonly is crosslinkable PDMS) to fill a mold, then it is heated for crosslinking (between 100-150° C) and finally peeled-off from the mold obtaining a flexible negative replica of the mold [169-174], [179], [181], [183-184]; and *iii)* Micro Transfer Molding (μTM), which has an analogous mechanism to REM, but with the implementation of a solvent vapor that can penetrate the polymeric mold, facilitating the detachment of the replica. This technique allows the creation of 3D microstructures [164], [168], [180].

***3.3.2.2.2 Other Top-Down methods*.** The remaining top-down techniques are etching and laser ablation. The first one is based on the selective removal of material from the surface of the substrate using an etching agent. It is called “wet-etching” if a liquid phase agent is used, and the surface will be only chemically attacked [189], [191], [193-194]. On the other hand, it takes the name of “dry-etching” when a gas or plasma phase agent is used in a vacuum chamber. In this case, the surface will be physically and chemically attacked [190], [192]. The second one is based on the direct writing of a micro-pit array on the glass substrate to provide the necessary roughness, using an ultrafast laser [195].

3.3.3 Comparative analysis of fabrication techniques

It was possible to identify three striking parameters of the synthesis methods listed in Table 2: cost-effectiveness, environmental impact of the fabrication strategy, and robustness of the coating.

In the first place, the economic aspect of the coatings synthesis was considered. It was obtained a total of 86 articles that claimed to be economically feasible by using words such as "cost-effective", "low-cost" or "inexpensive" in the description of its manufacturing process regarding other studies. As expected, a large number of articles related to Bottom-up approach were obtained for this category (68 records). Among them, Li et al. [39], [108] created repeatedly a highly transmissive antireflective (AR) coating using a LbL self-assembly technique, which allowed the formation of multilayered hierarchical nanostructures through electrostatic interactions between polyelectrolyte layers and mesoporous silica nonspherical nanoparticles. Thus, they avoided multi-step processing [197], [198] or high vacuum treatments typically used in physical vapor deposition (PVD) [117-121] or dry-etching [190], [192] techniques only one and two of which were reported as cost-effective respectively.

In the same way, sol-gel processing is a very appealing technique for a scalable cost-effective synthesis since it represents more than half of the Bottom-up cost-effective records (40 out of 68) as demonstrated extensively by different authors in the literature. In this regard, Du et al. [39] were able to obtain a double-layered transparent coating combining both small silica nanoparticles and dendritic silica nanoparticles with hierarchical pores synthetized with readily available materials (TEOS and aqueous ammonia) and great control over the thickness and microstructure distribution by tunning dip-coating parameters and the concentration of the sol-gel solution. Similarly, Yuan et al. [36], Li et al. [41], Li et al. [43], Mittal et.al [45], Quan et al. [46], Song et al. [48], Nanda et al. [50], among many other authors ([51-53], [59], [61], [64], [75], [77], [85], [87], [90], [93]), have reported inexpensive one-step sol-gel processes either by manufacturing SiO_2 nanoparticles using a hydrophobic chemical agent as co-precursor or by dipping pre-acquired microparticles into a surface modifier compound mixture to render low surface energy and achieve superhydrophobicity.

Furthermore, contrary to the common belief ([38], [43], [48], [63]), various top-down methods have been reported to be cost-effective and potentially applicable over large area for industrial implementation beyond the wafer scale (16 records out of 32). Although there are numerous challenges regarding existing lithographic methods such as costly mold patterning [166], [179], difficult control of the geometrical feature of the generated pattern or high process temperatures [165], [167], [182], [198-199], many soft lithography techniques have been demonstrated to be of great interest because of its versatility and scalability without the use of high energy-consuming fabrication steps unlike some sol-gel process reported [30], [32]. [35], [36], [39], [63], [65], [70], [79]. Among them, the replica molding technique stands out with a total of 10 cost-effective records [169-174], [179], [181], [183], [184] regarding the 16 of the whole top-down approach.

In addition to finding cost-effective routes to produce transparent SH coatings, their durability is also a critical parameter to be considered. Publications listed in the robustness category of Table 2 were carefully examined to determine whether after the coating was manufactured, any tests simulating harsh environmental conditions were performed. A total of 94 records were identified as robust and the procedures carried out were the following: thermal annealing at high temperatures; prolonged outdoor exposure involving conditions of high humidity or UV radiation [25,30,34,41,44,61-63,69,70,73,80,83,84,87,88,94,98,102,104,106,108,109,114,115,117,126-128,133,135,137,156,157,161,164,166,167,171,172,181,197]; prolonged indoor lab ambient conditions [64,75,99,136]; tape peeling tests to asses adhesion of the coating to the substrate

[27,31,32,44,47-50,57,73,75,85,125,141,151,163,198]; sand abrasion or scratch hardness tests (mechanical stability) [27,37,44,48-50,54,56,57,59,62,63,69,75,80,84,85,89,93,94,96,97,100-104,106,113-115,128,136,137,139,143-145,151,157,163,193,194,198] ; immersion into a wide range of pH solutions (chemical stability) [26,36,38,48,50,56,59,61,70,72,75,84,93,94,100-102,104,106,113,114,125-126,134,135,143,145,149,150,156,157,163,197] ; water impact using droplet fall or a water jet (resistance to rainfall) [32,37,50,56,62,65,72,85,101,104,114,115,135-137,150,151,157,166,182,195]; washing cycles [48,60,195]; icing cycles (for multifunctional anti-icing SH coatings) [58,79]; high hydraulic pressure tests [151]; and static Laplace pressure tests through evaporation of water droplets on the surface of the coating [35]. There are several authors who performed more than one of these experiments to ensure the durability of the manufactured coatings. In this regard, Bake et al. [62] fabricated via sol-gel a coating based on the deposition of superhydrophobic SiO_2 nanoparticles that kept its properties intact after 12 hours of exposure to UV radiation, being thermally stable up to 300 °C and presenting good resistance to the abrasion test, taking 5 test cycles to affect its non-wetting behavior. Furthermore, Lin et al. [195] presented a laser ablation process that allowed the coated glass to retain its superhydrophobicity after air exposure for up to one month and water soaking for one week. The coating also showed thermal stability and both good impinging and sand abrasion resistance.

Regarding the distribution of durable coatings by approach, great effort has been made to overcome the existing robustness limitations of the Bottom-up technology. It is known in the literature [9] that one of the main limitations of this approach is the weak adhesion of the nanostructures generated or deposited on the surface of the substrate, which leads to lose their self-cleaning properties when exposed to certain conditions of abrasion, scratching or water impact present in the environment. Surprisingly, among the 94 robust records, it was found a total of 82 publications belonging to this approach. Rather than the formation of coatings by dense close-packed monolayers, a significant number of these records (56 articles) report a great variety of deposited nanostructures such as: silica (SiO_2) nanoparticles [25,28,36,38,41,47-49,54,57-59,62,64,69,70,72,75,83,85,93,94,96,102,108,109,113-115,126,128,133,135-137,143-145,163], SiO_2 microparticles [50,61,87,114], SiO_2 raspberry-like nanostructures [37,60], SiO_2 nanotubes [79], hollow nanorod-like magnesium fluoride nanoparticles (MgF_2) [47,49], titanium oxide (TiO_2) nanoparticles [65], TiO_2 nano-rice structures [30], calcium carbonate ($CaCO_3$) nanoparticles [80], polytetrafluoroethylene (PTFE) nanoparticles [101], zeolite nanocrystals [44], zinc oxide (ZnO) nanorods [141], ZnO scale-like nanostructures [139], pseudobohemite nanoflakelets [89], copper (Cu) nanostrips [112] and titanate nanobelts [149]. In this regard, the wide spreading use of silica-based nanostructures has been demonstrated specially feasible to fabricate durable superhydrophobic coatings not only because its low cost but also due to its excellent scratch, abrasion, and water impact resistance [37,47-50,54,57,59,62,69,72,75,85,93,94,96,102,113-115,128,135-137,143-145,163]. Moreover, some authors have incorporated binding chemical agents such as polydimethoxysilane (PDMS) [54,93,163], bisphenol-A epoxy resin [59,114], N-(β-aminoethyl)-γ-aminopropylmethylbimethoxysilane (KH-602) [48], interface adhesive Qsil 216 [57], (3-Glycidyloxypropyl) trimethoxysilane (GLYMO) [62], polyurethane (PU) [128], aminopropyltriethoxysilane (APTES) [137], hydroxyl-terminated polystyrene [144], among many others [69,75,113,145], to further improve the adhesion of these building blocks.

The last characteristic analyzed was the environmental impact generated by the fabrication methods here presented regarding the use of both fluorinated compounds, which are toxic and represent a high risk of bioaccumulation in ecosystems, and some conventional organic solvents. Despite of being the parameter with the lowest number of records (36 out of 175), there is a trend towards more environmentally friendly processes since roughly 83% (29 records) of these studies are concentrated in the last five years [41,42,44,45,61,63,68,71,74,76-78,84,88,90,93,99,100,112,114,115,122,131,145,147,158,163,179,186,188]. It was identified two ways of achieving an eco-friendly process: either by substituting environmentally harmful chemical fluorinated compounds, as done by Rezayi et al. [147] who deposited zinc oxide (ZnO) nanoparticles that were functionalized using a stearic acid modifier as an eco-friendly material by means of the Chemical Bath Deposition (CBD) method; or by using non-toxic residues from other processes as raw materials to circumvent the use of potentially hazardous precursors as is the case of Saharudin et al. [61], who used palm oil fuel ash (POFA) waste as a source of silica and PDMS as a functionalizing fluorine-free agent to produce a chemically stable coating that achieved a CA of $156 \pm 1\,°$.

3.3.4 Pre-treatments and post-treatments

Interestingly, most of the included records not only apply a great variety of techniques to produce transparent superhydrophobic coatings but there are also numerous pre-treatments, traditionally used as a complement of the studied methods, that can greatly influence both the coating and the synthesis route final attributes in terms of cost-effectiveness, robustness, and eco-friendliness. In this context, unwanted contamination of the glass substrate prior to coating deposition may lead to poor adhesion and is a serious aspect that have been addressed by all the reviewed records through ultrasonic rinsing in a wide range of chemical agents such as: ethanol, acetone, hydrochloric acid (HCl), distilled water (DW), sulfuric acid (H_2SO_4) or even highly concentrated piranha solutions ($H_2SO_4 + H_2O_2$).

Additionally, several procedures have been made to improve the coating adhesion by hydroxylation (-OH) of the surface, which increases the density of available sites in the glass for further reactions. For this purpose, a solution of H_2O_2: H_2O: NH_4OH was implemented by Panyo et al. [90] for 45 minutes at 70°C, and Saharudin et al. [61] for 5 minutes at 80°C; likewise, it is possible to carry out the hydroxylation by oxygen plasma treatment [73,127]. Another pretreatment used is the deposition of a "seed layer" for the subsequent growth of zinc oxide (ZnO) complex nanostructures [138-142,146]. This is commonly performed by radiofrequency magnetron sputtering [141,142] or successive ionic layer adsorption and reaction (SILAR) method [146,148]. For example, Luo et al. [141] deposited a ZnO film by radio frequency magnetron sputtering to later obtain vertically aligned ZnO nanorods via the hydrothermal method; Kim et al. [142] followed a similar process but with the inclusion of galio as a dopant.

As a complement, modification or derivatization of rough surfaces is commonly used as post-treatment with the aim of enhance the hydrophobicity of the coatings. It takes advantage of low surface energy materials that can replace the highly active hydrophilic groups (Si-OH) by hydrophobic groups (-Si-R) commonly derived from alkylsilanes, chloroalkylsilanes, or fluoroalkylsilanes. These compounds are used as co-precursors in the synthesis of nanoparticles or as passivation layers typically deposited as self-assembled monolayers (SAM) by vapor deposition. The most popular compounds found in this review are presented in Table 4.

Table 4

Most popular surface modification compounds used in different fabrication techniques

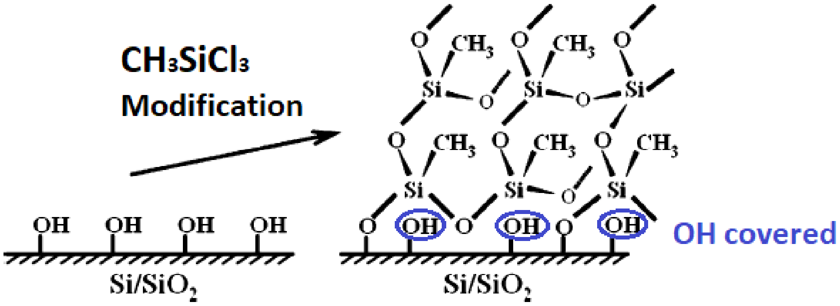
Surface modification compounds	Abbreviation	Technique	Reference
Hexamethyldisilazane	HMDS	CVD	[104]
		Sol-gel	[26,27,31,36,46,55-57,67,69,70,77,78,83,91]
1H, 1H, 2H, 2H- perfluorooctyltrichlorosilane	PFOCTS	Lithography	[176]
		CVD	[99,106]
		Spray coating	[144]
		Spin coating	[128,130]
		Dip coating	[133]
		Modified Langmuir-Schaefer	[160]
		Sol-gel	[30,54,62,73,75,79,85,89]
1H, 1H, 2H, 2H-perfluorooctyltriethoxysilane	POTS	CVD	[97,102]
		Spray coating	[143]
		Hydrothermal method	[139]
		Self-assembly	[108,109]
		Electrophoretic deposition	[149-151]
		Sol-gel	[39,47,83,94]
Polydimethylsiloxane	PDMS	Template method	[158]
		Sol-gel	[29,53,57,61,71,80]
Octadecyltrichlorosilane	ODTS	Modified Langmuir-Schaefer	[160]
		Sol-gel	[50,74,76,84]
Chlorotrimethylsilane	CTMS	CVD	[96]
		Sol-gel	[44,57,58,73]
1H,1H,2H,2H-perfluorodecyltriethoxysilane	PFDTES	Spray coating	[145]
		Hydrothermal method	[142]
		Self-assembly	[111]
		Laser ablation	[195]
		Sol-gel	[60]
Polymethyl-methacrylate	PMMA	Sol-gel	[25,70,72,85]
Hexadecyltrimethoxysilane	HDTMS	Polymerization	[123]
		Sol-gel	[49,72,87]
Stearic acid	SA	CVD	[196]
		Hydrothermal method	[140]
		CBD	[147]
		Sol-gel	[64]
Methacryloxypropyltrimethoxysilane	TMSPMA	CVD	[102]
		Polymerization	[125]
		Sol-gel	[33,43]
Heptadecafluoro-1,1,2,2-tetrahydrodecyl trichlorosilane	HDFS	Lithography	[167,170,197]
		Etching	[191]
1H,1H,2H,2H-perfluorodecyltrichlorosilane	PFDTCS	Lithography	[166]
		CVD	[95]
		PVD	[116]
		Self-assembly	[113]

The reason why organofunctional silanes are the most widely used compounds (11 of the 13 compounds presented) lies in the presence of organic substituents and the ease of forming covalent bonds (via an oxygen linkage) with glass. Organic substituents contain low surface free energy terminal groups that reduce interactions between the surface and water molecules [212]. These groups can be organized from lowest to highest surface free energy in the following order: $-CF_3 < -CF_2H < -CF_2 < -CH_3 < -CH_2$, which are present in several of the compounds listed above. For example, $-CF_3$ and $-CF_2$ terminal groups are found in PFOCTS, POTS, PFDTES, HDFS, and PFDTCS [213]. The formation of the O-Si-O bond occurs in a simple way thanks to the hydrolysable groups of the silanes. In general, its reactivity with hydroxylated surfaces decreases as follows: Si-NR2> Si-Cl> Si-NH-Si > $Si\text{-}O_2CCH_3$ > $Si\text{-}OCH_3$ > $Si\text{-}OCH_2CH_3$. Thus, PFOCTS (Si-Cl) and HMDS (Si-NH-Si) were the most used compounds (both present in 16 records). Although methoxy and ethoxysilanes have the lowest reactivity on the list, they are among the most popular (such as POTS) due to their easy handling and the non-corrosive and volatile nature of the alcohols that are generated as by-products (methanol and ethanol, respectively) [214].

Moreover, a successful hydrophobic modifier agent must act as a wall and cover residual polar sites on the surface that did not react to prevent them from reacting later with water, the hydroxyl groups are the most prone to hydrogen bonding [214]. This shielding effect can be seen more clearly in Figure 11.

Figure 11

Shield effect of surface modifier agent



Note. Adapted from: Gao et al. [215].

3.4 Discussion of current challenges and outlook

The fabrication of transparent self-cleaning superhydrophobic coatings for glass is a fast-growing investigation field due to its wide variety of potential outdoor applications. Conventional applications of transparent glass are windows, car windshields, lenses, screens, and glass facades, furthermore, a large part of the articles included in this review also encompass the use of coated glass as an external cover for photovoltaic solar cells and incorporate antireflective properties in order to increase the final output power generated by these modules. In this context, it is not a coincidence that in the last 10 years many of the reviewed publications come from China, South Korea and India, which are part of the most advanced countries in the solar energy field [216]. On the other hand, the participation of american countries is limited: only the United States and Brazil have published studies on the subject, therefore if subsequent investigations are conducted in Colombia, it would be a cutting-edge topic not only in the country, but throughout Latin America.

The increasing interest in this technology has been accompanied by the development of various fabrication techniques, within which the sol-gel method stands out. In the search for cost-effective, durable, and eco-friendly alternatives, numerous advances have been taking place over the last years, mainly in Bottom-up approaches and especially in the sol-gel synthesis, in which most of the reviewed investigations are concentrated. This method is simple and does not require rigorous sample preparation, complex multistep processing, or high vacuum environments. Moreover, it generally uses inexpensive and easily accessible precursors such as TEOS, ethanol or ammonia, typically obtaining nano or microparticles as roughness building blocks to achieve the self-cleaning effect. Many of these particles are made of silica due to its good affinity with glass and low toxicity, allowing better adhesion and avoiding subsequent recycling problems. Recent research has allowed the inclusion of new sources of raw material that take advantage of the residues of other processes such as palm oil fuel ash (POFA) or sugarcane bagasse ash. However, it is not the only way to reduce the environmental impact. Several authors avoid the use of fluorinated surface modifiers, which despite their ideal characteristics for hydrophobicity, turn out to be toxic and bioaccumulate in the ecosystems. Consequently, some other chemical compounds such as chloroalkoxysilanes, stearic acid, N. nimmoniana leaf extracts, among others, have been studied as eco-friendly alternatives. Despite the fact that the advances of this technology are evident, it was not found a study that raised the scaling of this process, so the development of a sustainable process for the manufacture of this type of coatings would be an interesting topic for future research including concepts of circular economy, such as the possibility of recovering used solvents or the utilization of recycled plastic as surface modifier agent.

Top-down methods have also been inclined towards these new trends; however, they are being overshadowed by the rapid improvement of bottom-up techniques. The coatings synthesized by the top-down approach have found inexpensive and scalable ways to stay relevant in recent years by utilizing cheaper and simpler techniques such as replica molding nanoimprint lithography.

Nonetheless, it was found that coatings made by these methods undergo almost no durability or stability tests and only few mention the environmental impact of the fabrication process.

Finally, it is a current challenge for superhydrophobic coatings the arising of new promising and competitive self-cleaning technologies that have emerged in response to the long-term stability issues addressed in this review. Although there are multiple ways to obtain self-cleaning coatings in addition to the superhydrophobic behavior (superhydrophilicity, oleophobicity, photo-induced self-cleaning, etc), each one has their own durability drawbacks. Slippery liquid-infused porous surfaces (SLIPS) are presented as an interesting bio-inspired alternative that can overcome the robustness obstacles of superhydrophobic surfaces by incorporating self-healing properties, thus having the ability to self-repair after exposure to harsh environmental conditions [217,218].

4. Conclusions

Based on the systematic review of the fabrication of self-cleaning coatings for transparent glasses carried out under guidelines of the PRISMA methodology, a growing trend was found in publications over the last 10 years. Asian countries are the main drivers of this technology, being present in 69.1% of the studies published.

The main techniques that make it possible to achieve the roughness and low surface energy requirements to reach the state of non-wettability were identified and categorized, being able to classify all the articles reviewed in three main categories: Top-down, Bottom-up, and the combination of both approaches. Subsequently, a comparative analysis of the techniques was carried out considering the main trends identified in the literature i.e., the development of more cost-effective coatings, their ability to maintain their properties under harsh environmental conditions, and their concern for processes more environmentally friendly. In addition, the main pre-treatments and post-treatments used were addressed highlighting the most implemented hydrophobic compounds.

Among the 23 techniques identified, the sol-gel method turned out to be the predominant technique in the reviewed articles due to its simple procedure and the readily availability of its main precursors. The most common nanostructures deposited by this method are silica nanoparticles, which present a great affinity with glass resulting in strong adhesion and good long-term durable coatings for outdoor applications. In addition, most of the sol-gel studies claim to be cost-effective and are concerned about the robustness of the coating obtained. Regarding the eco-friendly trend, it was observed that some efforts have been done incorporating new raw materials and avoiding the use of toxic compounds. Hence, this aspect has been gaining attention over the last 5 years.

On the other hand, lithographic methods are quite attractive for the development of self-cleaning coatings due to their ability of generating bio-inspired patterns with high precision and the tendency to opt for inexpensive methods (replica molding soft-lithography) than traditionally costly ones (photolithography).

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