Colorimetric Detection and Adsorption of Mercury Using Silver Nanoparticles: A Bibliographic and Patent Review

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Abstract: Mercury (Hg) contamination is a problem that currently affects not only the environment but also human health. Various types of commercial adsorbents have been proposed for its removal. Silver is a noble element that can chemically adsorb mercury, forming amalgams. However, its use as an adsorbent presents the following disadvantages: rapid surface saturation and high cost. These limitations can easily be overcome using silver nanoparticles (AgNPs). With a size of less than 100 nm, their reactivity, their high surface area, and a minimal amount of metallic precursor, they are ideal candidates for mercury removal. This study presents a compendium of the use of conventional mercury adsorbents and the use of AgNPs for their colorimetric detection and removal in different matrices, in both the aqueous and gas phases of Hg^0 and Hg^{2+}. In addition, the number of patents available in each case is analyzed. AgNPs as colorimetric sensors allow for quick detection of mercury *in-situ*. Additionally, the adsorption systems formed with AgNPs, allow for the obtaining of stable and chemically inert complexes, facilitating their recycling. It is concluded that the use of AgNPs is particularly efficient for the detection and removal of mercury, presenting a removal percentage of over 90%. As a result of the patents analyzed, its use is perfectly applicable at an industrial level.

Keywords: Mercury, silver, adsorption, nanoparticles and sensor

1. INTRODUCTION

1.1. Mercury

Mercury (Hg) is a ubiquitous toxic metal which is considered a global pollutant, and which affects the ecosystem and human health, depending on its amounts and chemical forms [1, 2]. Hg is emitted into the atmosphere as a result of anthropogenic or human activities, namely, industrial, agricultural, mining, and medical waste discharges, as well as from natural sources and processes such as rock weathering, volcanoes, and the degasification of the earth´s crust [3, 4].

According to the Global Mercury Assessment 2018 [5], in the 21st century, Hg emissions have been estimated at around 2000-2500 tons per year, suggesting that Artisanal and Small-scale Gold Mining (ASGM) may be the largest source of Hg, with 838 (675-1000) tons released in 2015 [5-7].

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As a result of international concern over the use of mercury and its effects on human and environmental health, the Minamata Convention on Mercury, which is supported by the United Nations Environment Program (UNEP) [8-10], was adopted in 2013. Its main purpose is to protect human health and the environment from the adverse effects of Hg and its compounds [11].

Mercury enters the environment in its divalent inorganic form (Hg^{2+}) through wet (precipitation) and dry (dust) atmospheric deposition [12-14]. In terrestrial ecosystems, Hg^{2+} is oxidized into inorganic species such as $HgCl_2$, HgO, or HgS, or organo-Hg compounds, depending on appropriate soil conditions that can lead the reaction (adsorption, complexation with soil humic acids or with inorganic ligands) [15, 16]. Hg can be temporarily deposited into the soil. Nevertheless, its mobility is complex. It is influenced by many factors, such as soil type, watershed hydrology, redox conditions, mineralization of organic matter present, and pH, among others, until it is remobilized into rivers and seas, in various forms [17].

In the aquatic environment, the riverine and submarine groundwater discharge are sources of Hg contamination [13]. This pollutant is readily bound by particulate matter and deposited into the bottom sediments, presenting a complex cycle as it is easily transported between water and sediment [18, 19]. Aquatic sediments are considered major sinks of Hg, or hot spots of Hg methylation [20] because Hg^{2+} can be methylated by abiotic and biotic processes into methylmercury (MeHg), which becomes available throughout the aquatic food web [21, 22]. Inorganic mercury can be methylated and accumulated in food webs depending on multiple biotic and abiotic factors such as pH, water temperature, and the presence of microorganisms [8].

Methylmercury (MeHg) is the most toxic form of Hg, which can bioconcentrate and biomagnify up in the food chain, where MeHg reaches high trophic levels as predators eat contaminated prey and concentrate it in their tissues [23-25]. Humans are exposed to MeHg mainly through fish consumption, which presents a serious health risk, especially to vulnerable groups such as pregnant women, breastfeeding women, and children [26-28].

This neurotoxin can affect the liver, heart, kidneys, reproductive organs, and central and peripheral nervous systems, as it is easily absorbed across biological membranes, causing potential damage. [29-32]. During pregnancy, MeHg exposure has been associated with a decrease in intelligence, behavioral changes, and developmental delays [9].

Table 1.   Commercial materials for mercury remediation

Nº	Publication number	Title	Inventor	Year	Contaminant	Phase	Sorption capacity (%)	Reference
1	US7101115B2	In situ stabilization of persistent hydrophobic organic contaminants in sediments using coal- and wood-derived carbon sorbents	Richard G. Luthy, Stanford, (CA, US), Upal Ghosh, Ellicott City, (MD, US)	2006	Hg MeHg	Sediment	94-98 [*][a] 31-73 [**][a] 73-92 [*][a] 57-86 [**][a]	[39]
2	US20160244379A1	Biochar	David Shearer John Gaunt George Vernon Cordner Peacocke	2014	Hg MeHg	Aqueous	60-90 [a] 5-80 [a]	[40]
3	US20100222481A1	Product and method for creating an active capping layer across surfaces of contaminated sediments	Joseph Jersak Tore Lundh	2008	MeHg	Aqueous *In situ* Soil	99 [a] >90 [a]	[41, 42]
4	US20190023994A1	Removal of mercury by chemical addition and mechanical separation	Evan Shigeto Hatakeyama (Richmond, CA) Kyle Kozo Higashidani (Walnut Creek, CA) Francisco Lopez-Linares (Richmond, CA), Michelle Marie Hart (Richmond, CA)	2019	MeHg	Aqueous	95-97 [a]	[41]

* Low native Kd values
** High native Kd value
[a] Uncertainty not available

Though the implementation of the Minamata Convention has started, it will take many years before Hg reaches non-hazardous levels [15, 33]. Considering the risk and effects of Hg and its compounds to the environment and human health, it is imperative to develop remediation technologies aimed at removing mercury from natural resources such as soils, water bodies, and contaminated sites.

Mercury remediation technologies involve adsorption, desorption, oxidation, and reduction mechanisms [34], where different materials have been developed to immobilize mercury, thus decreasing its mobility/solubility, and preventing its migration or transport to plants and water [35].

Table **1** presents the most common materials used for mercury remediation, commercially known as Biochars, SediMite, ThiolSAMMS, and Organoclay (PM199), which are applied as sorbents in contaminated ecosystems. These materials have different sorption capacities, depending on the concentration of mercury and its compounds.

At the same time, several technologies used to remove mercury from water have been reported, such as membrane separation or filtration, precipitation, ion exchange, adsorption, bioremediation using microorganisms, and reverse osmosis [36-38]. Nevertheless, their cost, efficiency, advantages, and time consumption are always discussed by authors. Table **2** summarizes the different remediation options developed to remove mercury from water, such as adsorption, biosorption, bioremediation, brass shaving, chemical reduction, precipitation, sorption, and the use of various materials or organisms which can present diverse adsorption capacities.

Currently, the detection and removal methods for Hg have gained prominence in the field of research due to the use of versatile materials that re-enhance the optical properties and increase the removal capacity of Hg. One of the materials that is being used is silver (Ag), as it has catalytic, optical, antibacterial, and amalgamation properties [53]. However, its industrial use has been limited by the low ratio of surface area and volume. One of the alternatives that have been proposed to avoid surface limitation

Table 2.   Technologies used for mercury and its compounds remediation in water.

Nº	Remediation technology / treatment	Material	Hg removal	References
1	Adsorption	Thiol functionalization of short channel SBA-15	Adsorption capacity of 195.6 mg g^{-1} at pH of 8 [a]	[36]
2	Adsorption	Biochars	8-80% [a]	[40]
3	Adsorption	Thiol-functionalized graphene oxide/Fe-Mn composite	Adsorption capacity 233.17 mg g^{-1} [a]	[43]
4	Biosorption	Immobilized *Cladophora sp.* algae	80% [a]	[44]
5	Biosorption	Immobilized microalgae *Chlamydomonas reinhardtii*	95% [a]	[45]
6	Bioremediation	Algae *Cyclotella cryptica* *Phaeodactylum tricornutum* *Scenedesmus subspicatus* *Porphyridium purpureum*	Reaction ratio LnR 2.087-1.016 [a]	[46]
7	Brass shaving / amalgamation	Cu and Zn	60-80% [a]	[47]
8	Brass shaving	Cu	96-98% [a]	[48]
9	Chemical reduction, air stripping	Stannous chloride dihydrate	94% [a]	[49]
10	Chemical reduction, air stripping	Stannous chloride dihydrate	90% [a]	[50]
11	Precipitation, co-precipitation, chelating agents	Polyaluminum chloride Ferric sulphate	62-63% [a] 50-76% [a]	[51]
12	Sorption	Biochar	71-100% [a]	[52]

[a] Uncertainty not available

is the use of silver nanoparticles (AgNPs), which re-enhance the optical properties due to their high surface plasmon resonance efficiency, which allows them to adjust to various visible wavelengths, while in the field of removal, its high ratio of surface area to volume increases the removal capacity of mercury ions [54]. It is important to mention that the AgNPs, having a reactive surface, can be coupled to different matrices that re-enhance their properties. The Hg adsorption technique using AgNPs anchored to different matrices is based on taking advantage of the amalgamation properties of Ag, which makes the process faster and more profitable [55, 56].

1.2. Colorimetric detection

Classical methods for the detection of mercury include emission spectrometry/atomic absorption (AAS/AES), inductively coupled plasma mass spectrometry (ICPMS), atomic fluorescence spectrometry (AFS), high-performance liquid chromatography (HPLC), ion-selective electrode (ISE), and flame photometry. Although they are powerful techniques for the determination of Hg^{2+}, their excellent performance requires expensive instrumentation and sample preparation that involves a lot of time and laborious procedures [57-61]. The use of colorimetric sensors reduces analysis costs, time, and the level of operation, as the color change can be directly identified by a person without the need to resort to a laboratory for the use of high-level equipment [62]. The colorimetric method consists of the detection of an analyte using the optical sensor absorption variation (color change is generated by the reactions that the analyte presents upon contact with the sensor). This method can be applied to samples, e.g., molecular, gas, biological, and ionic [63].

In contrast, colorimetric biosensors have quickly gained popularity and are now considered among the main application methods. However, published information shows that many biosensors have limitations and little stability in practical use. These problems have been solved through the use of metallic nanoparticles. Although most studies focused on gold nanoparticles (AuNPs), at present studies with AgNPs have demonstrated increased sensitivity when absorption spectroscopy is used, thus improving visibility due to variations in optical brightness [64, 65].

The evolution of the use of binders with metallic nanoparticles offers new opportunities for the development of colorimetric sensors that can detect various chemical species with high selectivity and sensitivity [66, 67].

Due to the optical properties of AgNPs, they have been considered promising nanomaterial for the identification of various analytes. The detection properties of AgNPs can vary depending on the chemical properties of the ligand [68]. Commonly used ligands are carboxymethyl cellulose, isonicotinic hydrazine acid, tartrate, 6-thioguanine, citrate, etc. These modify the surface of the nanomaterial, enhancing the degree of selectivity and sensitivity toward Hg^{2+} ions. Additionally, adjusting the analytical application allows specific interactions [68, 69]. However, it is worth mentioning that some metallic nanoparticles can detect mercury without the need for a binder [69].

Table **3** below presents a summary of studies carried out with AgNPs as colorimetric sensors.

The AgNPs and AuNPs nanosystems are considered the main sensors for the detection of metal ions due to their significant color change for different transition metal ions. The traditional chemical synthesis of nanosystems presents several drawbacks in addition to being very toxic to the environment. In response to this problem, nanoparticles developed through green synthesis have provided highly efficient sensors that can detect Hg^{2+} in aqueous solutions.

Choudhary *et al*. [79] studied the AgNPs as Hg^{2+} sensors, synthesized with aqueous extract of *Vigna mungo* seeds (black beans). This seed contains many flavonoids, proteins, and phenolic acids that allow the nanoparticles to be produced and stabilized. The methodology consists of a redox reaction between silver and Hg^{2+} in the aqueous medium obtaining a LOD = 130 nM, which constitutes a novel practice for the identification of mercury in wastewater. Fig. (**1**) shows an illustration of the Hg^{2+} detection method with AgNPs generated by green synthesis.
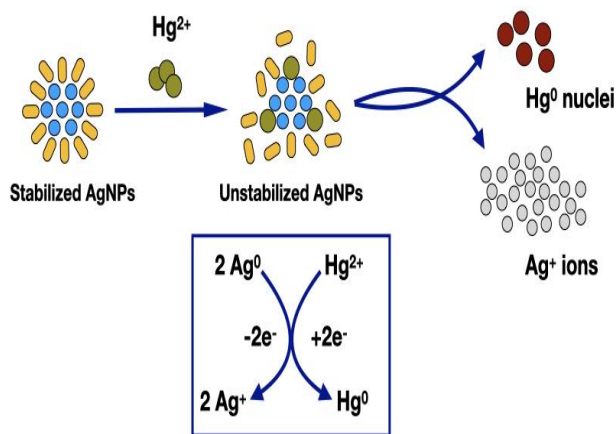

Fig. (1). Mechanism of Hg^{2+} detection using AgNPs. Adapted from [79].

Table 3. Colorimetric sensor studies for the detection of mercury Hg^{2+}

Nº	Sensor	Range	LOD	Analytical technique	Reference
1	AgNPs-aptamer	-	5 nM	Surface enhanced raman spectroscopy SERS	[56]
2	AgNPs-chitosan-carbon	1×10^{-8} to 1×10^{-6} M	7 nM	UV-Vis	[57]
3	AgNPs-gelatin	-	25 nM	UV-Vis	[70]
4	AgNPs-sodium alginate	0.025-60 μM	5.290 nM	UV-Vis	[71]
5	AgNPs-rhodamine B	-	2 nM	UV-Vis	[72]
6	AgNPs-cellulose	5-75 mg L^{-1}	0.12 mg L^{-1}	UV-Vis	[73]
7	AgNPs-cellulose	-	0.2 ng L^{-1}	-	[74]
8	AgNPs-carrageenan	≥ 350 mg L^{-1}	2.92×10^{-4} M	Digital photometry	[75]
9	AgNPs-Cl-carrageenan	350-450 nm	1 μM	UV-Vis	[76]
10	AgNPs with NaBH$_4$	25-500 nM	17 nM	UV-Vis	[64]
11	AgNPs with NaBH$_4$ based on anti-aggregation	0-333 nM	4 nM	UV-Vis	[77]
12	AgNPs-glutamine and histidine	1.0-500 μM	0.90 μM	UV-Vis	[78]
13	AgNPs-*Vigna mungo* extract	-	130 nM	UV-Vis	[79]
14	AgNPs-*Murraya koenigii* extract	500 μM	-	UV-Vis	[80]
15	AgNPs-*Manna hedysarum* extract	-	2.2×10^{-6} M	UV-Vis	[81]

Various synthesis strategies have been studied, including chemical and photochemical methods in the presence of various reducing agents that have some limitations and that contaminate the environment [82, 83]. Developing ecological approaches to prepare AgNPs motivated the synthesis with the use of microwave irradiation; this technique was fast, efficient, and viable. The use of microwaves improves reaction kinetics, forms uniform nucleation, and produces monodisperse nanoparticles [84-86].

It has been demonstrated that AgNPs generated by microwave irradiation with a coating agent such as L-cysteine produce spherical nanoparticles that rapidly detect Hg^{2+} in aqueous solution, as L-cysteine has a strong interaction with noble metals forming covalent bonds [85, 87].

Nanoenzymes have been very important in the field of catalysis; these can be found as oxide nanoenzymes, noble metal nanoenzymes, and carbon-based nanoenzymes [88]. Zhang *et al.* [57] developed a new oxidative enzyme consisting of chitosan-modified hierarchical porous carbon silver nanoparticles, which he named DHPC @ CSAgNP. The new nanomaterial allowed Hg^{2+} to be reduced on the surface formed by silver nanoparticles without the need to generate hydroxyl radicals using an external reagent. Additionally, the presence of carbon increased the catalytic capacity, benefiting the oxidation process, which resulted in a coloration change from yellow to blue. For these reasons, the use of this new nanomaterial was determined to improve selectivity and sensitivity with mercury [89]. Fig. (**2**) presents the sensor preparation (DHPC @ CSAgNP).

The colorimetric approach can generate both qualitative and quantitative results, as the concentration of the analyte has a direct and proportional relationship with the color change [90, 91]. Lobregas *et al.* [75] identified Hg^{2+} at concentrations of $\geq$350 mg L^{-1} with detection limits of 2.92×10^{-4} M using digital photometry. For this, AgNPs synthesized with a carrageenan biopolymer (Carr-AgNP) were used as a reducing agent and stabilizer for nanoparticles. The objective of this study was to develop a kit that allows for the detecting and quantifying of Hg^{2+} in contaminated media. The colorimetric kit coupled with digital photometry makes the technique fast, easy, cost-effective, qualitative, and in real-time. In Fig. (**3**), we can observe the variation in UV-vis absorbance of AgNPs at 430 nm and digital images of Carr-AgNPs solutions.
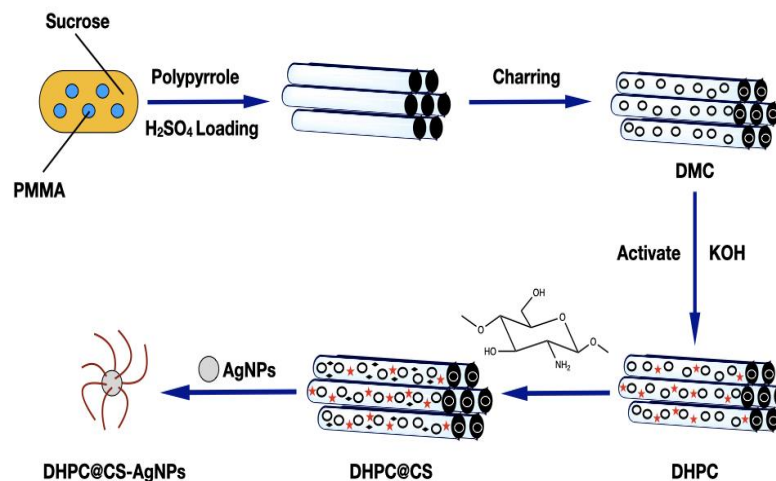
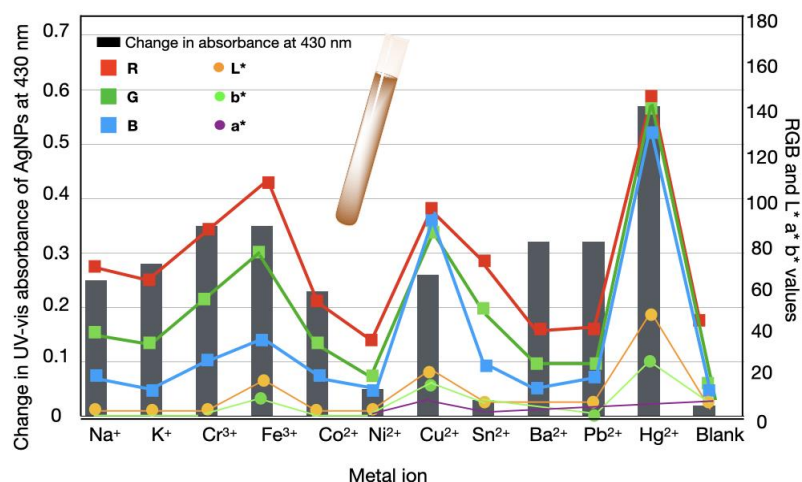**Fig. (2).** Preparation of the sensor using enzymes and AgNPs. Modified from [57].

Fig. (3). Variation of absorbance of AgNPs solutions in the presence of different metal ions Adapted from [75].

Sodium alginate is a biopolymer that is used as a reducing and stabilizing agent to produce colloidal AgNPs solution. This new film presents a high mechanical resistance, insolubility, and environmental stability. The film impregnated with AgNPs makes it possible to detect Hg^{2+} ions in aqueous solutions [70, 92].

Fraghiri and Ghorbani [71] studied the expansion of the nanosensor formed, AgNPs impregnated with sodium alginate (SA-AgNPs)-biopolymer biodegradable in the detection of Hg^{2+} in aqueous solutions as a substrate for AgNPs. In this study, it was concluded that the linear relationship of the absorbance of the surface plasmon resonance and the concentration of the Hg^{2+} ions is within a range of 0.025-500 µM. Fig. (**4**) contains a summary of the study carried out with SA-AgNPs for the detection of Hg^{2+}. It can be seen that AgNPs functionalized with organic molecules improve sensitivity and allow for the development of sensors that detect mercury ions in environmental and biological samples [93, 94]. The glutamine and histidine functionalization can act as a different, selective, and simple colorimetric sensor for the detection of Hg^{2+}, where a cooperative interaction between the binder and the metal is generated.

Buduru *et al*. [78] studied the influence of shape and size using a controlled synthesis with glutamine and histidine (Gln-His-Ag). The reaction between the analyte and the sensor produces a displacement in the adsorption band to red. Also, *in-situ*, a color change from yellow to orange could be observed. The method based on Gln-His-Ag reported a Hg^{2+} concentration in a range of 1 - 500 µM with a detection limit of 0.90 µM. This novel method is inexpensive and simple, and allows for the determination of various metal ions, such as Pb^{2+} and Mn^{2+}. Fig. (**5**) shows a scheme that explains the color change that occurs when Gln-His-Ag interacts with Hg^{2+}.

The versatility of AgNPs as colorimetric sensors for the detection of mercury ions has motivated the implementation of several research articles. Table **4** presents patents based on their research on metal nanoparticles as colorimetric sensors useful in detecting Hg^{0} and Hg^{2+}. The most successful inventions use sensors composed of silver nanoparticles that can be used for the total interaction of its surface with the sample or as receptors with unique surface characteristics to host molecules with functional

groups capable of interacting with the sample [95]. Most of the inventions focus on methods to detect Hg^{2+} in aqueous solutions, where the solution is brought into contact with the product formed between the reaction of a plant extract and biosynthesized silver nanoparticles [96]. The size and shape of the products obtained play an important role in the detection of mercury, highlighting that at sizes less than 100 nm, the nanomaterial is manageable and efficient, providing detectability ranges up to 200 nM. Regarding the shape of the product, the most efficient are selected from a group that can have a spherical, triangular, bar, or cubic shape. These methods have been tested with a high selective yield in aqueous systems in the presence of interfering compounds such as Ca, Mn, Ni, Na, Zn, Ba, and K.
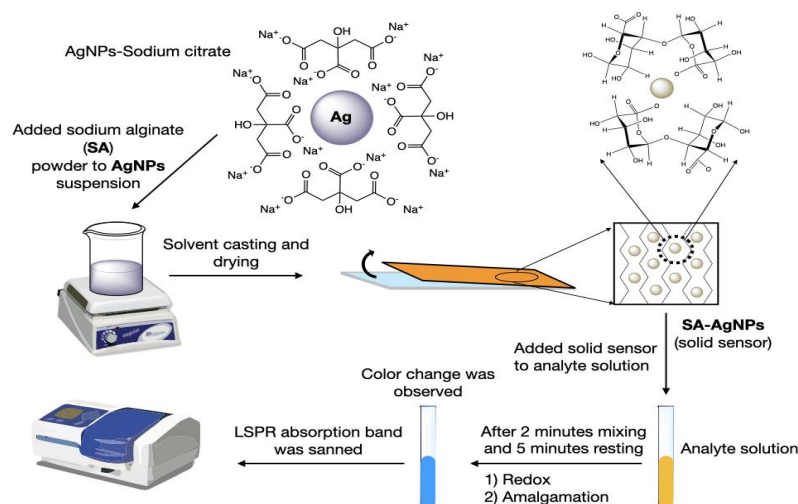

Fig. (4). AgNPs sensor for the detection of Hg^{2+}. Adapted from [71].
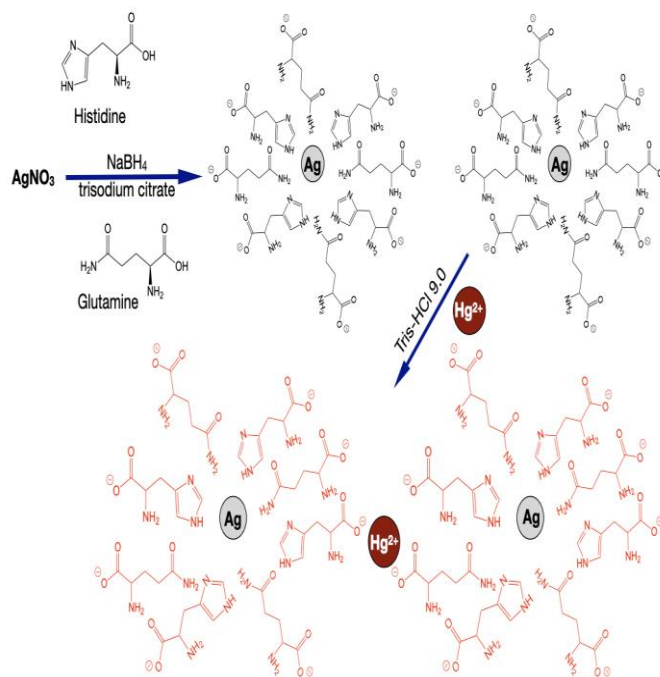

Fig. (5). Amino acid-Ag sensor in the presence of Hg^{2+}. Modified from [78].

In the most efficient inventions, the plant extract comprises at least one conglomerate of species consisting of alkaloids, flavonoids, saponins, carbohydrates, polysaccharides, terpenoids, steroids, sterols, phenols, tannins, anthraquinones, anthocyanins, amino acids, proteins, and vitamins. The detection limit for Hg^{+2} of 40 nM has been achieved. In contrast, in inventions where carbon-supported AgNPs have been used [97], detection limits of 0.15 nM in Hg^{+2} have been reached; results have been associated with the increase of the surface porosity due to the presence of carbon, which benefits the process.

Table 4. Colorimetric sensor patents for mercury detection

Nº	Publication Number	Title	Inventor	Year	Product	Detectability	Reference
1	JP4787938B2	Detecting sensor using silver nanoparticles	Armstrong, G. *et al*	2004	AgNPs functionalized with catalytic receptors	-	[95]
2	US9719975B1	Mercury detection in water-based compositions	Chanbasah B. and Salalo O.	2016	AgNPs-*Ocimum basilicum extract*	40 a 200 nM	[96]
3	CN106290186B	A kind of doping porous carbon Nano silver grain visualizing rapid mercury detection ion	Wei S. *et al.*	2017	AgNPs-carbon supported	0.15 nM	[97]

1.3. Adsorption systems

The importance of reducing mercury emissions, mainly from industrial wastewater, is unavoidable, as they harm human health [98]. Several expensive technologies with high operational requirements have been commonly used for this type of contamination. Among the best-known technologies are precipitation, ion exchange, membrane filtration, bioremediation, and amalgamation [99-101].

Adsorption is a profitable and effective alternative for the removal of heavy metals in water [102,103]. For this reason, various adsorbents have been studied, such as clays, zeolites, silica, chitosan, various polymers, and activated carbon. All of them present a weak bond between the metal adsorbent and a low adsorption capacity [104-107]. The importance of developing an adsorbent that can efficiently remove mercury and that is easy to recycle has motivated the use of noble metals such as Au and Ag to improve adsorption capacity [99, 108].

The Au and Ag surfaces are used for the analysis of traces of mercury in combustion gases or in the environment. These surfaces, called "collectors," remove mercury from the area, or from the contaminated medium [109, 110]. The collectors can appear with different morphologies, such as spheres, tubes, thin films, wires, networks, etc. [110-112]. The technique consists of concentrating the mercury on the surface of the collector and then it is quantified in the laboratory to determine the adsorption efficiency. The results obtained must consider the recollection mechanism; the phenomenon of adsorption, and other factors that reduce the efficiency of the technique [113, 114]. A very important analysis factor is surface saturation because it limits the adsorption efficiency. Many studies have reported results on this topic, but they could not be standardized because, at different operating conditions, the results vary drastically from each other [109, 110, 114]. At the macromolecular level, information is known about the adsorption mechanism between mercury and Au and Ag surfaces. However, small exposures of mercury, at high temperatures, saturate the surface of the metals. This phenomenon has not yet been explained at the atomic level [115].

Levlin *et al.* [115] studied the mechanism of mercury adsorption on Au and Ag surfaces, where it was determined that adsorption has a direct dependence on mercury concentration and the temperature at which the tests are carried out, highlighting that these results will be comparable only to others in the same operating conditions.

Fig. (**6**), shows the results of Levlin *et al.'s* research into mercury adsorption on the surface of silver at different exposure times, temperatures, and concentrations.
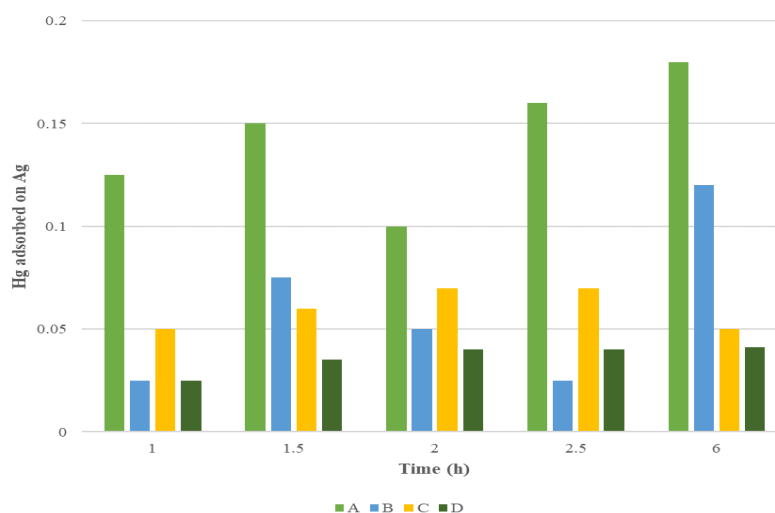

Fig. (6). Amount of Hg adsorbed on Ag samples. Data obtained from [115].

Previously, it was mentioned that Ag is used as an adsorbent and a colorimetric sensor for mercury due to its optical properties and its ability to be amalgamated. The amalgam that can form is easily decomposed by action of the temperature [116, 117]. However, silver alone as an adsorbent has a low surface area in relation to volume, which limits the adsorption efficiency. One solution to this problem in surface limitation is to use AgNPs.

Various nanomaterials are currently used, especially those prepared from metal oxides, transition metals, carbon, and silicon. These new materials show great differences in the properties at macroscale. For example, a nanomaterial has a

greater relationship between the surface and the volume, which allows for greater contact with the environment [118].

AgNPs can take on different shapes and sizes depending on the synthesis method used whether physical, chemical, biological, or hybrid. The latter nanomaterial has gained notoriety due to its chemical stability, catalytic activity, high conductivity, and its antibacterial activity [119]. Due to its large surface area and small size, it has been used as a blood sugar controller for pesticide removal, or to remove heavy metal ions in water (Hg^+, Hg^{2+}, Pb^{2+}, and Cd^{2+}) [104, 120].

Metallic particles have high surface energy, which, by reducing them in size (nanoscale), increases generating aggregates that limit the surface available for adsorption. Many alternatives have been proposed to solve this problem. For example, the use of silicon hydride (SiH) as a reducer to form zero-valent silver, according to the following reaction [121].
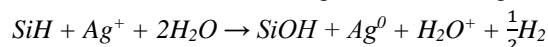
$$SiH + Ag^+ + 2H_2O \rightarrow SiOH + Ag^0 + H_2O^+ + \frac{1}{2}H_2$$

Table 5. Adsorption studies to remove mercury in the aqueous and gas phase.

Nº	Adsorbent	Symbology	Adsorbate	Removal capacity	Quote
1	Ag carbon fiber supported	Ag-ACF	Hg^0	192.3 ± 4 mg Hg g^{-1} adsorbent	[116]
2	Ag supported on granular activated carbon	Ag-GAC	Hg^0	80 ± 1.5 µg Hg g^{-1} adsorbent	[124]
3	Ag supported on titanium dioxide	Ag-TiO$_2$	Hg^0	100 ± 2 µg Hg g^{-1} adsorbent	[124]
4	Activated carbon impregnated with AgNO$_3$	Ag-AC	Hg^0	-	[125]
5	Ag-vanadium silicate	Ag-EVS	Hg^0	63.4 ± 7.7 µg Hg g^{-1} adsorbent	[126]
6	Ag supported in graphene	Ag-graphene	Hg^{2+}	280.8 ± 0.1 mg Hg g^{-1} adsorbent	[99]
7	Ag-mercaptosuccinic acid	Ag-MSA	Hg^{2+}	800 mg Hg g^{-1} adsorbent [a]	[127]
8	Ag supported on nanomesoporous silica	SBA-Ag	Hg^{2+}	42.26 ± 0.56 mg Hg g^{-1} adsorbent	[128]
9	AgNPs cellulose supported	cellulose -AgNPs	Hg^{2+}	95.261 ± 0.779 mg Hg g^{-1} adsorbent	[104]
10	AgNPs-*Aloe vera* extract	AgNPs	Hg^{2+}	95 ± 0.5 % [a]	[119]
11	Activated carbon impregnated with AgNPs	AgNPs-AC	Hg^{2+}	500 ± 25 mg Hg g^{-1} adsorbent	[129]
12	Activated carbon impregnated with AgNPs (regenerated)	AgNPs-AC	Hg^{2+}	≥ 90 % [a]	[129]
13	AgNPs anchored to a silica matrix	AgNPs-SiO$_2$	Hg^{2+}	0.4 ± 0.75 mmol Hg g^{-1} adsorbent	[130]
14	AgNPs anchored to quartz	AgNPs-Q	Hg^{2+}	First model 18.3 ± 0.31 mg Hg g^{-1} adsorbent Second model 19.2 ± 0.23 mg Hg g^{-1} adsorbent	[131]
15	AgNPs anchored to synthetic zeolites	Ag-ZFA	Hg^{2+}	≥ 99 % [a]	[132]

[a] Uncertainty not available

This reaction allows the size of the AgNPs to be controlled, as the hydroxyl groups have a weak reducing capacity enough to generate "pure" silver and limit the increase in surface energy, which implies a greater adsorption surface [121, 122].

It is important to mention that the use of AgNPs as direct adsorbents for removal of metal ions can present difficulties at the time when the nanomaterial is extracted from the contaminated area, due precisely to its diminutive size [123]. In recent years, Ag and AgNPs have been implemented as adsorbents supported in different matrices that take advantage of their properties, increase adsorption efficiency, and facilitate extraction [99, 120]. Table **5** presents several studies with different adsorbents; it is reported that the use of silver or silver nanoparticles (AgNPs) together with another compound benefits the adsorption process.

Silver, as mentioned above, has a limited surface extension that can be improved using various supports [100, 133].

Ag adsorbents supported on activated carbon fiber have certain advantages, such as a large surface area, high adsorption capacity, the presence of oxygen groups on the surface that can retain mercury, rapid desorption, and easy regeneration. The silver charge on the activated carbon surface increases the adsorption efficiency due to the amalgams that can form upon contact with mercury. Kuang *et al.* [116] report that the presence of silver can increase up to six times the adsorption capacity than a natural activated carbon.

Mercury gas adsorption is commonly a dry process, which has a lower efficiency compared to wet adsorption [125]. The need to develop effective technologies for the control of mercury has motivated the study of kinetics, the adsorption capacity of each material, and the factors that decrease the efficiency of the process [134]. The way in which mercury is found (Hg^0 or Hg^{2+}) is a factor that limits adsorption because studies indicate that Hg^0 presents greater complexity to be trapped by the surface of an adsorbent [135, 136]. Currently, activated carbon is being impregnated with silver nitrate to improve the adsorption conditions and study the adsorption/desorption mechanism estimating thermodynamic and kinetic parameters [125].

Ag adsorbents supported on TiO_2 are considered to be economically profitable, stable, and reusable. These structures have shown a greater power for mercury vapor removal than traditional adsorbents (Ag-activated carbon) without light dependence, as many metallic oxides anchored to other materials require the irradiation of light (photocatalytic process) so that the adsorption capacity increases [124, 137, 138].

Khamdahsag *et al.* [138] studied the performance of the Ag-TiO_2 adsorbent for the removal of Hg^0. Ag at the TiO_2 interface works by presenting active sites that trap Hg^0, which significantly improves adsorption efficiency. The system can be described as a chemical trap made up of silver ions and metallic silver, which attracts and amalgamates mercury [139, 140]. Fig. (**7**) shows a schematic of the possible mechanism involved.
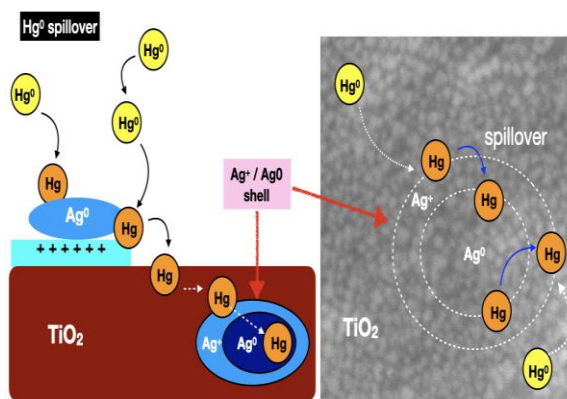

Fig. (7). Ag supported on TiO_2. Adapted from [138]

Graphene, due to its hexagonal structure, generates a thin resistant material with a large surface area like AgNPs [141]. The synergistic effect AgNPs-graphene was shown in the performance in the removal of Hg^{2+} from aqueous solutions [133, 142, 143].

Qu *et al.* [99] synthesized the adsorbent Ag-graphene, which was characterized by (FT-IR), (XDR), (TEM), and (SEM). The synthesis method used to obtain the adsorbent was a hydrothermal reaction. Ag-graphene was used as a Hg^{2+} remover in aqueous solutions. Fig. (**8**) shows a brief outline of the research methodology reported by Qu *et al*.

The variables that influence mercury capture are related to the physical properties, pore size, surface area, functional groups, and chemical characteristics of the adsorbents [138].

Recently, catalytic adsorbents consisting of oxides and noble metals have generated a lot of attention. Transition metal oxides such as MnO_x, V_2O_5, FeOx, and CuOx have multiple states of valence and chemical stability, which translates into a high rate of mercury adsorption [126, 144-146]. Vanadium oxide (V_2O_5) is a selective reduction catalyst that removes NO_x compounds. For this reason, V_2O_5 was applied in the removal of mercury [147]. However, this compound does not present a simple dispersion in the macropores of internal support surfaces such as T_iO_2 and S_iO_2 [148]. This limitation has been reduced using vanadium silicate (EVS), which is a derivative of titasilicate, which itself has channels and pores on a molecular scale [146, 148]. EVS is an adsorbent used to remove certain contaminated gases, making it a promising candidate for mercury removal [145, 149, 150].
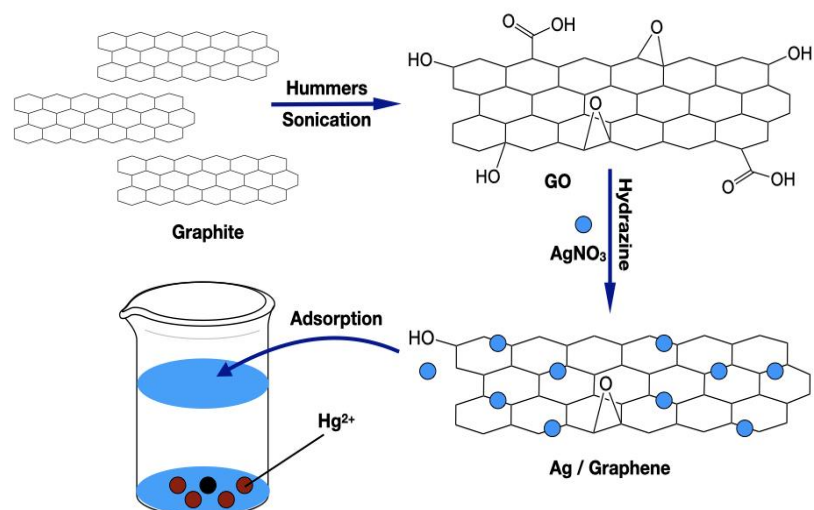
Fig. (8). Ag-graphene as an adsorbent in the removal of Hg $^{2+}$. Modified from [99].

A study by Zhou *et al*. [126] determined that the presence of Ag on the surface of metal oxides improves the adsorption capacities of mercury due to the interactions that take place, as they weaken the forces that bind the metal ions. Fig. (**9**) indicates the primary process for elemental mercury capture.
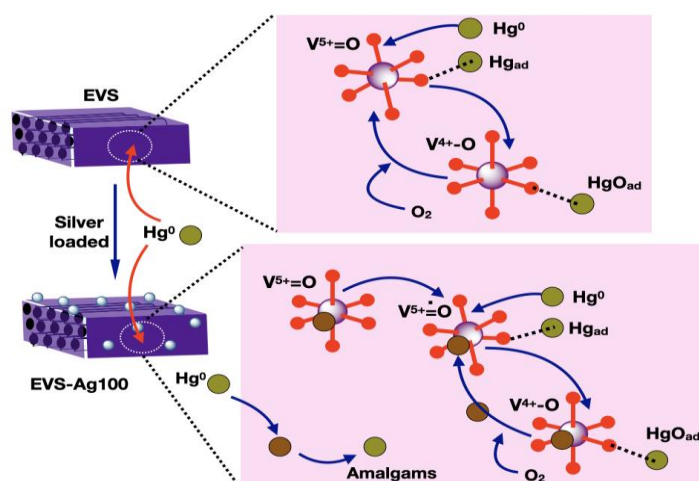

Fig. (9). Primary reaction process for the capture of Hg^0 on vanadium silicate (EVS). Adapted from [126].

For 2,500 years, some noble metals have been extracted by amalgamation. Currently, this technique benefits the mining and jewellery industries [151]. Nanoformulation of this technique uses noble metal nanoparticles to remove mercury from contaminated sources [152].

Nanoscale interactions lie in the size of the nanoparticle; thus, the smaller the particle size, the more the interaction of surface forces will increase. On a normal scale, Hg^{2+} interacts with silver according to the following reaction.

$$Hg^{2+}_{(aq)} + 2Ag_{(s)} \rightarrow Hg_{(s)} + 2Ag^{+}_{(aq)}$$
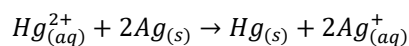
In this reaction, the product generates zero-valent solid mercury [153, 154]. At the nanoscale, AgNPs, with a diameter of less than 32 nm, prevent the oxidation of silver in solution and increase the formation of Ag-Hg amalgams, which can be immobilized depending on the support. These results have contributed to the knowledge of chemistry on a nanoscale and to the study of wastewater purification [154].

Noble metal nanomaterials are highly efficient at removing heavy metals due to their active sites [155]. Nanomaterials anchored to suitable supports, such as graphene, silica, organic polymers, organic metal structures, and metal oxides, can enhance active sites [156-159]. Organic metal structures provide the adsorbent with a high surface and multiple channels at the molecular level, but at acidic conditions, because those present in mercury cannot guarantee stable support [160], while organic covalent structures are porous crystalline materials that have a large surface area and well-defined pore structure and are perfect supports for metallic nanoparticles because, under acidic and basic conditions, it can be stabilized, benefiting the removal of mercury in contaminated waters [129]. Fig. (**10**) shows a schematic of the AgNPs anchored to a covalent organic structure proposed by Wang *et al*. [160].
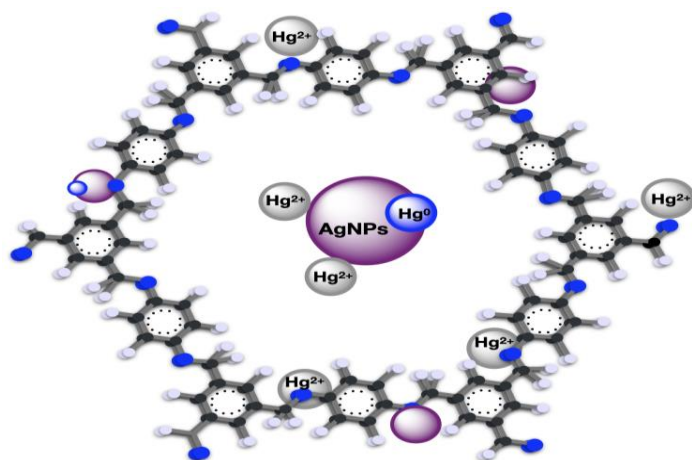
Fig. (10). Hg and AgNPs anchored to covalent organic structures. Adapted from [160].

Supports such as cellulose are used to contain nanoparticles in wastewater treatment. Cotton cellulose is a profitable, abundant, non-polluting biopolymer that has a large surface area and affinity for metal adsorption [161].

Ali *et al.* [162] studied the adsorption and desorption of zinc and silver nanoparticles supported on cellulose as a metal ion remover (Hg^{2+}, Cr^{+3}, Co^{2+}, Pb^{2+}, Ni^{2+}) in water. The reported results for AgNPs-cellulose show a greater tendency to eliminate mercury ions than others ($Hg^{2+} > Ni^{2+} > Cr^{3+} > Co^{2+} > Pb^{2+}$) at low temperatures (30 min). Additionally, cellulose as support minimizes the release of AgNPs in the contaminated medium, making the adsorbent easy to recover (Fig. **11**).
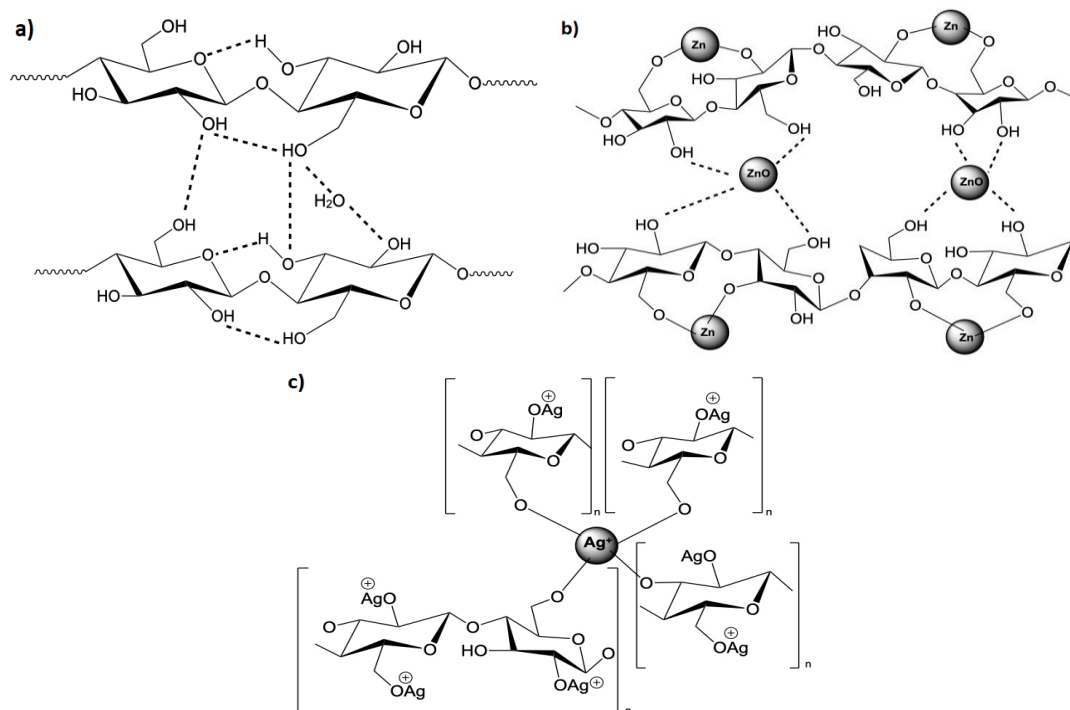

Fig. (11). a) Cotton cellulose, b) cellulose adsorbent-ZnNPs, and c) Cellulose adsorbent-AgNPs. Modified from [104].

The mercury properties to amalgamate silver are also useful at the nanoscale, allowing mercury to be extracted from aqueous solutions to form stable (chemically inert) complexes [130, 163]. Silica has thermal stability and a specific surface area range from 300 to 350 m^2 g^{-1}, making it an attractive material as a support for metallic nanoparticles [164].

Korobeinyk *et al.* [130] chemically synthesized the AgNPs that were supported by means of a redox reaction to a non-porous silica surface that had silicon hydride (SiH) groups. The AgNPs were bound to the SiO_2 matrix by interaction with the SiH groups. The only parameter that was modified in this study was the concentration of the metal salt.

The chemically synthesized AgNPs have a low adsorption capacity due to their low stability. For this reason, the use of various oxides, such as SiO_2, TiO_2, ZnO, and SiO_2-TiO_2, has been encouraged and allowed to be stabilizers of nanocomposites, which increase the adsorption capacity [165, 166]. Quartz sand (Q), which is largely composed of natural sandstone, is used at an

industrial level due to its physical and chemical properties, as well as because of its low cost [167]. Sandstone as an adsorbent has limitations in terms of removing heavy metals in water.

The study by El-Tawil *et al.* [131] focused on using the quartz-supported AgNPs as adsorbents that remove Hg^{2+} from aqueous solutions. The preparation of the AgNPs was carried out using table sugar, which acted as a chelating agent (sol-gel technique assisted). Then this nanomaterial was modified with the presence of quartz, which generated the Ag/QNPs compound [127, 167]. The parameters that were studied were the metal concentration, pH, time, and adsorbent-solution ratio. Additionally, the kinetic models were adjusted to adsorption isotherms that represented the adsorption mechanism. Fig. (**12**) presents the variation of mercury removal as a function of time for both modified and unmodified adsorbents.
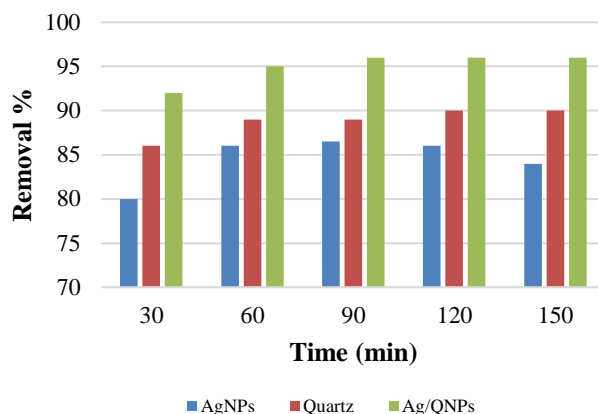

Fig. (12). Effect of time on Hg^{2+} adsorption using different adsorbents. Data obtained from [131].

The ashes generated by the carbon are an industrial by-product produced by its combustion. Annually, there are huge quantities that need a recycling alternative that reduces the environmental impact [112, 113]. Currently, carbon ashes are being transformed into synthetic zeolites by means of hydrothermal synthesis, which can be used in the medical, industrial, agricultural, and environmental fields [114]. In the environmental field, zeolites are being used to eliminate Hg^{2+} in contaminated waters, but their adsorption capacity has limited their industrialization. To enhance the adsorption efficiency, the use of AgNPs containing zeolite compound has been proposed to take advantage of the amalgamation property. These new adsorbents are low-cost; the raw material is an industrial by-product, and they remove mercury in the aqueous and gas phases [114-116].

A case study is the presence of mercury in crude oil during the refining process a big problem caused by the degradation of the industrial plant and aging of the catalyst, which affects not only the efficiency of the process, but also the health of the plant operators [129]. The refined products called hydrocarbons, when burned, produce mercury compounds that pollute the environment. These later condense and contaminate water sources [155]. Saudi Arabia contributes 17% of annual crude oil production and, recently, a high applicability of nanomaterials has been found in the removal of heavy metals, as they have a large surface area in relation to their volume [129, 135, 155].

AgNPs maintain the characteristic of having low solubility, and they can interact with mercury in solution. Fig. (**13**), presents a study of AgNPs supported by activated carbon to remove condensed mercury from Arabia gas [116].
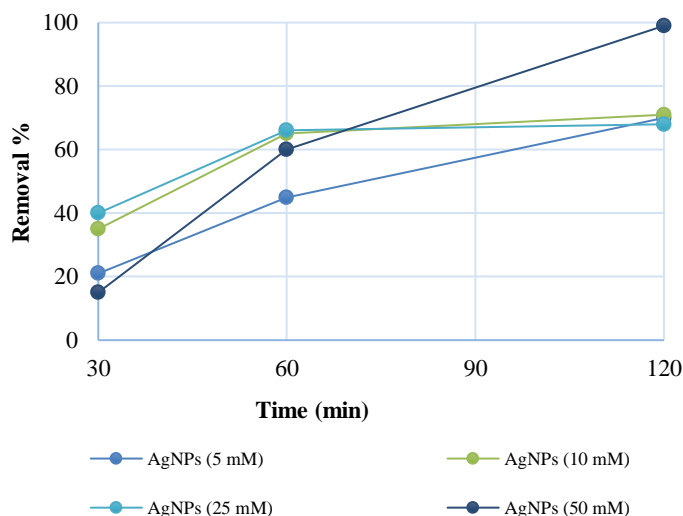

Fig. (13). Effect of the concentration of AgNPs supported on activated carbon at different times. Data obtained from [129].

In Table **6**, patents are presented that are mostly adsorption systems that eliminate Hg^0 and Hg^{2+}. These systems are made of metals and even nanomaterials that enhance the ability to remove mercury. Many of the patents focus their analysis on gaseous emissions due to the large industrial production of hydrocarbons that contain mercury.

Table 6.   Patents for adsorption systems.

Nº	Publication number	Title	Inventor	Year	Product	Adsorbate	Removal capacity	Phase	Reference
1	US5053209A	Removal of mercury from natural gas and liquid hydrocarbons utilizing silver on alumina adsorbent	Tsoung Y. Yan	1991	Hg removal system with Ag supported on alumina	Hg^0	15-450 $\mu g\ m^{-3\ a}$	gas	[168]
2	US5354357A	Removal of mercury from process streams	Markovs J. *et al*	1994	Ag, Au and Pt metal purification system	Hg^0 y Hg^{2+}	0.02-200 $\mu g\ m^{-3\ a}$	gas	[169]
3	US5322628A	Method for simultaneously drying and removing metallic and organic mercury from fluids	Tsoung Y. Yan	1994	Ag compound Hg drying and removal system	Hg^0, Hg^{2+}	250 $\mu g\ m^{-3\ a}$	gas	[170]
4	US5523067A	Removal of mercury from naturally occurring streams containing entrained mineral particles	Markovs J.	1996	Hg removal system in hydrocarbons with metals such as Au, Ag, Pt, and Pd	Hg^0, Hg^{2+}	0.02-300 $\mu g\ m^{-3\ a}$	Combustion gas	[171]

It is evident that the inventions are focused on industrial processes in which the fixed beds constituted by selective mercury adsorbent materials are recurrent for the purification of industrial streams. Markovs [169] presents several adsorption systems that provide an alternative for the removal of mercury in its different phases. The inventor indicates that mercury can be treated by means of two beds in the following way: The stream contaminated with mercury enters the first bed (regenerable bed), which is made up of materials such as gold, palladium, platinum, and silver. The mechanism allows the mercury in the feed stream to amalgamate the metals in the bed; this generates the separation of the contaminant from the feed stream. The purified stream passes to a second bed that is made up of activated carbon that, due to its superficial property, traps the mercury remains that have not amalgamated. This system allows mercury concentrations of less than 0.01 µg m^{-3} to be obtained from industrial streams. Additionally, they also presented a natural gas and hydrocarbon purification system that uses the same principle of metal amalgamation, but with hydrocarbon streams in the liquid and gaseous phase [171].

The use of new adsorbents for fixed beds improves mercury removal capabilities. Activated carbon is considered a profitable and abundant raw material; many adsorption systems are designed with activated carbon as a contaminant removal material [172]. Although it has adequate surface properties for mercury adsorption, these can be enhanced by the presence of certain metals, such as silver. Tsoung Y. [168] presents a silver adsorbent supported on activated carbon to remove mercury in natural gas and liquid hydrocarbons. The objective of the development of new materials for the absorption of mercury in the field of hydrocarbons lies in the limitation of the oil refining process by having mercury in the feed stream. For this reason, silver supported by activated carbon acts as a mercury remover through amalgamation; this purifies the stream and increases the quality of the crude oil. The use of activated carbon as a support also benefits the adsorbent recycling processes because it can be easily separated from the contaminated medium [170].

CONCLUSION

The use of AgNPs as colorimetric sensors and adsorbent to detect and remove Hg was confirmed. AgNPs, as colorimetric sensors, have a wide field of application due to their speed, profitability, and versatility in working in different wavelength ranges. As adsorbents, they are very versatile. With a removal rate of over 90%, they can be anchored to different support materials, which allows for easy removal and subsequent recycling. According to the patents analyzed, the applicability of AgNPs as colorimetric sensors and adsorbents has a great implication on an industrial scale. The use of AgNPs for the detection and removal of Hg still seems to be a promising area of study.

CONSENT FOR PUBLICATION

Not applicable.

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