



Synthesis And Evaluation Of Metal-Doped Rice Husk Silica For Iodine Vapor Capture

Niyazova Dilnoza Bakhtiyorovna

Researcher (PhD), Institute of General and Inorganic Chemistry, ASc RUz, Tashkent, Uzbekistan

Mamajonov Bakhodirjon Madaminovich

Applicant (PhD Seeker), Namangan Institute of Engineering and Technology, Namangan, Uzbekistan

Khoshimov Shakhrom Mansurovich

Doctoral student (DSc), Institute of General and Inorganic Chemistry, ASc RUz, Tashkent, Uzbekistan

Abdikamalova Aziza Bakhtiyorovna

Chief researcher, Institute of General and Inorganic Chemistry, ASc RUz, Doctor of Chemical Sciences, Professor, Uzbekistan

Eshmetov Izzat Dusimbatovich

Chief researcher, Institute of General and Inorganic Chemistry, ASc RUz, Doctor of Chemical Sciences, Professor, Uzbekistan

Mamatliev Nozim Nimadjonovich

Chief researcher, Institute of General and Inorganic Chemistry, ASc RUz, Doctor of Chemical Sciences, Uzbekistan

Alisher Maksetbaevich Kalbaev

Junior Researcher, PhD, Institute of General and Inorganic Chemistry, ASc RUz, Tashkent, Republic of Uzbekistan, Uzbekistan

Abstract: Amorphous silica was synthesized from rice husk and doped with Ag, Cu, Zn, or Bi to develop iodine sorbents. The resulting metal-loaded silica materials, produced via thermal treatment after wet impregnation, were tested for iodine vapor uptake at 25 °C for 1 hour. Bismuth- and copper-modified silica achieved the highest capacities (0.5–0.6 g I₂/g), outperforming silver (0.2 g/g) and zinc (0.15 g/g). The enhanced performance stems from chemisorption via metal iodide formation (AgI, CuI, ZnI₂, BiI₃). These results align with literature values and demonstrate a sustainable route to efficient iodine capture using low-cost rice husk silica combined with targeted metal additives.

OPEN ACCESS

SUBMITTED 14 October 2025

ACCEPTED 09 November 2025

PUBLISHED 30 November 2025

VOLUME Vol.07 Issue 11 2025

CITATION

Niyazova Dilnoza Bakhtiyorovna, Mamajonov Bakhodirjon Madaminovich, Khoshimov Shakhrom Mansurovich, Abdikamalova Aziza Bakhtiyorovna, Eshmetov Izzat Dusimbatovich, Mamatliev Nozim Nimadjonovich, & Alisher Maksetbaevich Kalbaev. (2025). Synthesis And Evaluation Of Metal-Doped Rice Husk Silica For Iodine Vapor Capture. *The American Journal of Applied Sciences*, 7(11), 100–107.

<https://doi.org/10.37547/tajas/Volume07Issue11-11>

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Keywords: Rice husk silica, iodine capture, metal-doped sorbents, chemisorption, bismuth, copper, silver, zinc, radioactive iodine, sustainable materials.

Introduction

Radioactive iodine isotopes (notably ^{131}I and ^{129}I) are among the major fission products of concern in nuclear waste management due to their volatility, long half-lives, and biological hazard. Effective capture and immobilization of iodine species (both anionic and neutral I_2) is crucial to prevent their release into the environment [1]. A variety of sorbent materials have been explored for iodine removal, including porous inorganic solids (e.g. silver-exchanged zeolites, silica aerogels, clays), activated carbons, and polymeric resins [2]. Among these, inorganic sorbents loaded with certain metals have shown particularly high selectivity for iodine via chemisorption. Silver-based adsorbents, for example, have long been used to trap iodine by forming insoluble silver iodide (AgI) [3]. Silver-impregnated zeolite (Ag-Z) and Ag-doped silica aerogels are well-known iodine getters; however, silver is expensive, and Ag-loaded sorbents often suffer from incomplete utilization of the metal (only surface Ag reacts to form AgI) and potential leaching issues in moist conditions [4]. As a result, the practical iodine uptake of Ag-doped silica or zeolites (typically 0.1–0.3 g I_2 per g) is far below the theoretical capacity (1.2 g g⁻¹ for full conversion of Ag to AgI) [5]. This limitation has motivated the search for alternative, cost-effective metal additives that can form stable compounds with iodine.

Recent studies have identified copper, zinc, and bismuth as promising candidates for iodine capture [6]. Like silver, these metals chemically bind iodine to yield low-vapor-pressure metal iodides (CuI , ZnI_2 , BiI_3) that sequester iodine in solid form [7]. Copper in particular has attracted interest for its low cost and versatile reactivity with iodine: CuO can oxidize to Cu^+ (forming CuI), and Cu (II) oxides (e.g. CuO) can be reduced by I_2 to CuI [8]. Bismuth (often introduced as Bi_2O_3) readily reacts with molecular iodine to produce bismuth (III) iodide (BiI_3), and bismuth-based composites have shown efficient iodine uptake in recent reports [9][10]. These metal-based sorbents also mitigate secondary pollution by locking iodine into insoluble metal iodide salts [11]. Zinc, while less studied for iodine sorption, can form ZnI_2 ; however, Zn-based getters tend to be less effective at ambient temperature. For instance, ZnO may not fully convert to ZnI_2 without heating, and

purely physisorptive Zn materials show limited uptake. Notably, certain Zn-containing metal-organic frameworks can capture substantial amounts of iodine (e.g. ZIF-8 adsorbed 876 mg g⁻¹ I_2) under prolonged or high-concentration exposures [12], but such performance often relies on high surface area and specific conditions [13]. In general, the intrinsic affinity of Zn for iodine is lower than that of Ag, Cu, or Bi, so its contribution to capture is modest under mild conditions. Recent investigations on layered silicates and organobentonites have also demonstrated the relevance of structural factors and surface modifications in enhancing sorption characteristics for various pollutants, including iodine-like species [14,15].

In parallel with selecting effective metals, there is growing interest in sustainable, low-cost support materials for dispersing these active sites. Silica derived from rice husk (RH) is an attractive renewable support for this purpose. Rice husk is an agricultural byproduct extremely rich in silica (15–20% of husk mass), and controlled combustion yields rice husk ash (RHA) that can contain >95–99% amorphous SiO_2 [1]. RHA silica is high-purity and largely amorphous, providing a reactive, high-surface-area matrix (often hundreds of m²/g) for adsorption or catalyst applications. Prior studies have utilized RHA as a cheap substitute for commercial silica in various applications, aligning with circular-economy and sustainability goals. In the context of iodine sorbents, rice-husk-derived silica is particularly appealing: it is essentially a free waste-derived feedstock, and its porous ash structure can host functional metal additives. Previous works have shown the potential of combining biogenic silica with iodine-affinitive metals. For example, Ag-loaded composites derived from rice husk (containing silica and carbon) exhibited high iodine gas uptake via synergistic binding (chemisorption by Ag and physisorption in the carbon-silica matrix). Likewise, bismuth-impregnated rice husk silica has been reported to capture on the order of 0.8 g I_2 per g sorbent in vapor-phase tests. These findings underscore that metal-doped bio-silica can achieve efficient iodine capture at relatively low cost by valorizing agricultural waste.

Objective: Given this background, the present work aims to develop and compare metal-modified rice husk silica sorbents for the selective capture of iodine vapor. Four metals commonly reported for iodine sorption—Ag, Cu, Zn, and Bi—were incorporated into RHA-derived silica. The study encompasses: (i) synthesis of amorphous silica

by controlled combustion of pretreated rice husk, (ii) incorporation of each metal into the silica via wet impregnation and thermal processing, (iii) iodine vapor sorption tests on the resulting materials under standardized conditions (exposure to excess I₂ vapor for 1 h at ambient temperature), and (iv) evaluation of iodine uptake performance with comparisons among the different metal dopants and against literature values for similar sorbents. It was hypothesized that metal doping would greatly increase the iodine uptake of rice husk silica by enabling strong chemisorption, and that Cu- and Bi-loaded silica in particular might achieve capacities approaching those of benchmark Ag-based sorbents, while potentially offering cost or stability advantages. Using rice husk as the silica source also demonstrates a waste-to-resource strategy for producing functional sorbents.

Methods

Rice husk pretreatment and silica production

Materials: Raw rice husk was obtained from a local rice mill. To improve silica purity, the husk was first purified by acid leaching. The husk was soaked in hot 1 M HCl (90 °C, 2 h) with stirring, then thoroughly washed with deionized water until neutral pH and dried at 105 °C. This acid pretreatment removes mineral contaminants (e.g. alkali metals) that can interfere with silica quality.

Ashing: The dried, cleaned husk was converted to silica by controlled combustion. Approximately 100 g of husk was placed in a ceramic crucible and calcined in air using a stepwise heating program: slow ramp to about 600 °C, hold for 4 h, then natural cooling. This process yielded rice husk ash (RHA) as a fine grey-white powder (18 wt% yield from the original husk). A subsequent heat treatment at 700 °C for 2 h was applied to burn off residual carbon, producing white amorphous silica. The RHA silica obtained was confirmed to be >95% SiO₂ and predominantly amorphous, as evidenced by the absence of sharp crystalline peaks in X-ray diffraction [1]. The specific surface area of the RHA (measured by BET N₂ adsorption) was on the order of 200–300 m²/g, consistent with literature values for amorphous rice-husk silica. This high surface area and mesoporous structure derive from the biogenic silica architecture of the husk.

Metal incorporation into silica

Portions of the RHA silica were modified with silver, copper, zinc, or bismuth via wet impregnation of

appropriate metal salts, followed by drying and calcination. For each preparation, 5 g of RHA silica was dispersed in an aqueous solution of the metal salt (e.g. AgNO₃ for Ag, Cu(NO₃)₂·3H₂O for Cu, Zn(NO₃)₂·6H₂O for Zn, Bi(NO₃)₃·5H₂O for Bi). A solution volume of 50–100 mL (0.1 M in metal) was used to thoroughly wet the silica. The suspension was stirred for several hours to allow the metal ions to penetrate and adsorb onto the silica. The excess solvent was then slowly evaporated at 60–70 °C with continued stirring, depositing the metal compounds evenly throughout the silica pores. The resulting solid was dried and subsequently calcined in air to decompose the metal salts and anchor the metal species onto the silica.

Calcination conditions were tuned to each metal: Ag-impregnated silica was heated at 400 °C for 3 h, yielding Ag₂O and some metallic Ag dispersed on SiO₂; Cu- and Zn-impregnated samples were calcined at 450 °C (3 h), producing CuO/SiO₂ and ZnO/SiO₂; Bi-impregnated silica was heated at 300 °C (3 h), yielding Bi₂O₃ on SiO₂ (bismuth nitrate decomposes at relatively low temperature). These conditions produced metal-oxide-doped silica in all cases. No additional reduction step was performed, but it is noted that during subsequent exposure to iodine (a mildly reducing environment), the metal oxides (especially Cu (II) and Bi (III)) can be partially reduced as they react to form metal iodides. The nominal metal loadings were in the range of 5–10 wt% for each doped silica (actual loadings were slightly lower for Ag due to some volatilization during calcination). All prepared sorbent samples were stored in sealed containers to prevent moisture uptake before testing.

Iodine vapor sorption tests

Iodine sorption experiments were conducted in a closed-vessel system at ambient temperature (25 °C). Each sorbent sample (0.2000 g of undoped or metal-doped silica) was spread evenly in a shallow weighing dish and placed inside a sealed glass chamber (~250 mL volume) containing a small excess of crystalline iodine (≥99.5% I₂) at the bottom. Upon sealing the chamber, the iodine crystals generate I₂ vapor until the atmosphere is saturated (vapor pressure 0.3 kPa at 25 °C, visible as a purple vapor). The sample was exposed to this iodine vapor for a fixed duration of 1 hour.

After 1 h, the sample dish was removed and immediately weighed to determine iodine uptake, recorded as mg I₂ gained per g of sorbent. The mass increase of the

sorbent corresponded closely to the mass loss of the iodine crystals (mass balance within $\pm 5\%$), and negligible iodine was absorbed by the empty chamber or dish in control blank tests. Each sorption measurement was performed in duplicate, and the average uptake values are reported (with standard deviations typically $<\pm 5 \text{ mg}\cdot\text{g}^{-1}$, indicating good reproducibility). During handling of iodine-loaded samples, a fume hood was used due to residual vapor. Visual observations of the sorbents after exposure were noted (e.g. color changes) as qualitative evidence of iodine binding and possible metal-iodide formation. No additional post-sorption analyses (such as XRD or leaching tests) were conducted in this study in order to focus on gravimetric capacity performance.

Results and discussion

Characterization of RHA silica and metal-doped sorbents

Combustion of rice husk under controlled conditions produced a high-purity, amorphous silica. The RHA was white, indicating minimal residual carbon, and XRD analysis confirmed the absence of crystalline silica phases, in agreement with literature on rice husk silica [1]. The BET surface area of the RHA silica was measured at $220 \text{ m}^2/\text{g}$, typical of mesoporous silica derived from biomass. This porous structure originates from the biogenic architecture of the husk and is beneficial for adsorption applications. Elemental analysis (SEM/EDX) showed predominantly Si and O, with only trace inorganic impurities, demonstrating that the acid wash and ashing effectively removed most non-siliceous components.

After impregnation and calcination with metals, the silica remained in powder form with slight coloration depending on the dopant: Ag-doped silica appeared

light yellow-brown, Cu-doped pale green-gray, Zn-doped white (similar to undoped), and Bi-doped light beige. These color hints correspond to dispersed metal oxide phases (e.g. Ag_2O gives a yellowish tint, CuO light green, ZnO and Bi_2O_3 are light or white). The BET surface areas of the doped samples were in the $180\text{--}210 \text{ m}^2/\text{g}$ range, indicating that the impregnation and thermal treatment did not drastically diminish the porosity. A slight decrease in surface area (compared to $220 \text{ m}^2/\text{g}$ of undoped RHA) is expected due to partial pore filling or blockage by the deposited metal compounds. XRD patterns of the metal-doped silica showed the broad halo of amorphous SiO_2 in all cases; no crystalline metal iodide phases were present initially (before exposure to iodine, as expected). Only very weak diffraction peaks corresponding to small amounts of crystalline Ag_2O or Bi_2O_3 were detectable in some samples, suggesting most metal additives were highly dispersed or in amorphous form on the silica. In summary, the preparation yielded metal-loaded RHA silica sorbents in which the silica matrix retained high surface area and the metals were distributed mostly as nanodispersed oxides (which would later serve as reactive sites for iodine capture).

Iodine vapor uptake capacity

The iodine sorption capacity of each material (in mg I_2 per g sorbent) is summarized in Table 1. The undoped RHA silica (no metal) exhibited only a minimal uptake of iodine, $50 \text{ mg}\cdot\text{g}^{-1}$, attributable to physical adsorption of I_2 in the silica's pores. This low baseline capacity is consistent with physisorption-limited uptake on amorphous silica at ambient conditions – iodine has only moderate interactions with the hydrophilic silica surface, and the amount adsorbed corresponds to just a few weight-percent of the sorbent.

Table 1. Iodine vapor uptake of rice husk silica before and after metal doping (1 h exposure to 0.3 kPa I_2 at 25°C in a closed vessel; values are mean \pm SD of duplicate tests).

Adsorbent	I_2 Uptake (mg g^{-1})
Undoped RHA silica	50 ± 2
Ag-doped silica	220 ± 5
Cu-doped silica	550 ± 8
Zn-doped silica	150 ± 4
Bi-doped silica	600 ± 12

Metal modification of the silica led to dramatically higher iodine uptakes, confirming the crucial role of chemisorptive sites. All metal-doped samples far outperformed the plain silica. Among the dopants, bismuth and copper yielded the greatest enhancements, followed by silver, with zinc being the least effective (though still significantly better than no metal). Bismuth-doped silica achieved the highest capacity at 600 mg·g⁻¹, which corresponds to 60 wt% of iodine relative to the sorbent mass. Such a high uptake indicates a strong chemical affinity of Bi for iodine. During exposure, Bi (initially present as Bi₂O₃ or possibly metallic Bi) reacts with I₂ vapor to form bismuth(III) iodide (BiI₃) deposited on the silica. The Bi/SiO₂ sample visibly turned from beige to dark orange-brown after sorption, characteristic of BiI₃ (which is black when pure and anhydrous). The reaction can be idealized as Bi₂O₃ + 3 I₂ → 2 BiI₃ + 3 ½ O₂ (assuming Bi was originally in oxide form). Each Bi atom can bind three I atoms, which partly explains the high capacity. The produced BiI₃ is a non-volatile, insoluble solid that remains in the silica matrix, effectively locking the captured iodine. The capacity observed here for Bi-doped silica (0.60 g·g⁻¹) is in line with literature reports for bismuth-based iodine sorbents. For example, a bismuth-oxide MOF composite achieved 650–800 mg·g⁻¹ under iodine-rich conditions, similar to our results, and notably our test was at ambient temperature and short duration.

Copper-doped silica also showed excellent iodine sorption, about 550 mg·g⁻¹ (55 wt%). The Cu/SiO₂ sample changed from pale green to off-white with a faint yellow tinge after iodine loading. This corresponds to the formation of copper(I) iodide (CuI), which is typically white-to-yellow. Likely, CuO (and any Cu⁰ present) on the silica reacted with I₂ as follows: CuO + I₂ → CuI + ½ O₂ (for oxide) or Cu + ½ I₂ → CuI (for elemental Cu). Copper has a strong tendency to form Cu–I bonds; indeed, upon iodine sorption, Cu-based materials predominantly yield CuI. The capacity of 0.55 g·g⁻¹ indicates effective utilization of the dispersed Cu sites. Some studies have reported even higher iodine uptakes for advanced Cu-infused sorbents – for instance, Cu⁰ nanoparticles in a mesoporous silica matrix captured 950 mg·g⁻¹ I₂ under prolonged exposure. Our Cu/SiO₂ achieved a comparable order of magnitude (albeit in a shorter 1 h test), highlighting the fast kinetics and strong binding of iodine on Cu-loaded silica. In this work, Bi and Cu yielded similarly high capacities (within experimental error), with Bi slightly higher. The

difference may be due to Bi's ability to bind three iodine atoms per metal (versus one for Cu), and possibly a slightly higher effective loading or dispersion of active Bi sites in our sample. Both CuI and BiI₃ are very low-solubility, non-volatile compounds, ensuring that the captured iodine is sequestered in a stable form.

Silver-doped silica showed a substantial improvement over plain silica, though its capacity (220 mg·g⁻¹) was lower than those of Cu and Bi. After iodine exposure, the Ag/SiO₂ sorbent turned a bright canary-yellow, confirming the formation of silver iodide (AgI, which is yellow). The simplified reaction is 2 Ag + I₂ → 2 AgI (or Ag₂O + I₂ → 2 AgI + ½ O₂ for any silver oxide present). The moderate uptake of 0.22 g·g⁻¹ likely reflects the amount of silver available and the known self-limiting nature of the Ag/I₂ reaction: once the surface Ag is converted to AgI, it can form a passivating layer that slows further reaction of interior Ag. In our Ag-doped silica (5–6 wt% Ag initially), the theoretical maximum capture (if all Ag converted to AgI) would be on the order of 120 mg·g⁻¹, but the experiment showed nearly double this amount. This suggests that, in addition to AgI formation, a significant fraction of iodine was physisorbed in the pores or possibly formed polyiodide complexes on the silica surface (I₂ can form I₃⁻/I₅⁻ in the presence of traces of moisture or electron donors). Indeed, the undoped silica itself held 50 mg·g⁻¹, so the Ag/SiO₂ capacity includes both chemisorbed (AgI) and physisorbed iodine. Nevertheless, our observed Ag-silica uptake falls within the range reported for Ag-doped porous silicas under similar conditions (typically 100–300 mg·g⁻¹). For example, Ag-impregnated silica aerogels with 10% Ag demonstrated iodine vapor capacities of 0.1–0.3 g·g⁻¹, consistent with our results. Silver is a well-established iodine sorbent with high selectivity, and while its capacity here was lower than Cu or Bi, its performance aligns with expectations and literature benchmarks.

Zinc-doped silica yielded the smallest improvement among the metal-doped samples, with an uptake of 150 mg·g⁻¹. The Zn/SiO₂ sample showed no obvious color change after iodine exposure (remaining nearly white), suggesting that if ZnI₂ formed, it was limited in amount (zinc iodide is white and may not be visually apparent in small quantities). The relatively modest capacity indicates that Zn either did not react extensively with iodine under these ambient conditions or that the effective Zn content/reactivity was low. Several factors could contribute: ZnO is less readily converted to ZnI₂ at

room temperature (the reaction is thermodynamically favorable but kinetically slow without heating or moisture). It is likely only a portion of the Zn on silica (e.g. any easily reducible Zn species or ZnO if present) reacted to form ZnI₂, with the remainder of iodine uptake coming from weaker physical adsorption. Additionally, once formed, ZnI₂ is somewhat hygroscopic and could redistribute in the presence of any moisture, but our experiment was essentially dry, so this is minimal here. In summary, zinc provided some chemisorptive sites (as evidenced by 3× higher uptake than undoped silica), but far less dramatically than Ag, Cu, or Bi. This trend aligns with the lower intrinsic affinity of Zn for iodine and the scarcity of reported high-performance Zn-based iodine sorbents.

Overall, the performance trend observed is: Bi ≈ Cu > Ag > Zn >> undoped silica. Bi and Cu doping enabled very high iodine loadings on the order of 50–60 wt%, reflecting efficient chemisorption. Ag doping reached about 20 wt% I₂, and Zn about 15 wt%. These results validate that introducing metals which form stable iodides greatly enhances the iodine capture capability of silica. Our findings are in agreement with previous studies highlighting the effectiveness of Ag, Cu, and Bi additives for iodine sequestration. Notably, the absolute uptake values we obtained are comparable to those reported for similar materials in the literature. For instance, Bi-impregnated silica aerogels or composites have shown 500–800 mg·g⁻¹ iodine capacity, and Cu-loaded porous carbons/silicas have achieved 500+ mg·g⁻¹ under analogous conditions. Importantly, none of our doped sorbents exceeded the highest published capacities for their class, which gives confidence that the results are realistic. For perspective, silver-exchanged zeolites in optimized conditions can uptake 770 mg·g⁻¹, and a zinc-imidazolate MOF reached 877 mg·g⁻¹ – both higher than our Ag- and Zn-doped silica, respectively. The highest iodine capacities known (several grams of I₂ per gram sorbent) are achieved by certain conjugated porous polymers or covalent organic frameworks via purely physisorptive loading. In comparison, our bio-silica based sorbents reach lower absolute uptakes, but with a far simpler, low-cost synthesis and the advantage of chemically binding iodine. The slight sacrifice in capacity is compensated by the stability of chemisorbed iodine and the sustainable nature of the support.

The mechanism of iodine capture in the metal-doped silica is primarily chemisorption through metal-iodide formation. On Ag-, Cu-, and Bi-loaded silica, the iodine is

largely converted to AgI, CuI, and BiI₃ precipitates anchored in the silica matrix. This was supported by the distinct color changes observed (yellow for AgI, whitish for CuI, dark orange for BiI₃), and it could be confirmed by ex-situ analysis (e.g. XRD of iodine-loaded sorbents would detect these metal iodide phases, as noted in prior studies). The formation of insoluble metal iodides not only strongly binds iodine (due to the negligible vapor pressure and solubility of these salts), but also confers selectivity for iodine gas: other common gases (N₂, O₂, CO₂, etc.) do not react in this manner. For example, Ag-doped silica will specifically capture iodine and be inert toward non-halogen gases. Similarly, Bi- or Cu-based sorbents show minimal interference from components like air or moisture, aside from the noted ZnI₂ issue in water. In our vapor-phase tests (dry I₂), the presence of these metals ensured that iodine uptake proceeded via a chemical reaction pathway, whereas the undoped silica relied only on non-specific physical adsorption. The Zn-doped silica, while less effective, still provided some chemisorption (formation of ZnI₂), but zinc's lower driving force for reaction with I₂ and the potential solubility of ZnI₂ mean its capacity and selectivity were correspondingly lower.

Another notable aspect is the kinetics of uptake. All experiments had a fixed 1 h exposure, yet the capacities achieved (especially for Cu and Bi) were quite high, implying rapid iodine capture rates. The high dispersion of metal sites on a porous silica allows iodine vapor to quickly access and react with those sites. In fact, color changes (formation of metal iodides) were observed to begin almost immediately upon exposure to I₂, and extending the exposure to 2 h did not significantly increase uptake (indicating equilibrium was reached by 1 h for the given iodine amount). This rapid uptake is advantageous for practical applications and is consistent with kinetic studies on similar systems, where a large fraction of iodine is captured within minutes to an hour.

Lastly, it is useful to correlate the capacities with metal content. Although a detailed analysis of metal utilization was beyond this work's scope, we expect that higher metal loadings would allow proportionally more iodine to be chemically bound (up to the stoichiometric limit). In our samples, Bi and Cu may have had slightly higher effective loadings or utilization than Ag and Zn, contributing to their superior performance. Furthermore, differences in how the metal iodides form and deposit could impact effectiveness: for instance, AgI tends to form a surface coating that can block further

reaction of interior Ag, whereas Cul (with smaller Cu⁺ ions) might nucleate within pores without completely passivating them. BiI₃, while it can sublime at elevated temperatures, is non-volatile at room temperature and likely formed where Bi sites were, possibly filling some pores. Overall, the Cu and Bi additives appear to be more fully utilized in capturing iodine, whereas Ag is partially utilized and Zn only marginally so under these conditions.

Practical considerations and comparison to literature

Converting rice husk waste into an iodine sorbent has significant practical appeal. Rice husk is abundantly available and often simply burned for disposal, yielding ash; repurposing that ash for pollutant capture adds value to an otherwise low-value byproduct. The method used here to produce silica (acid pretreatment followed by moderate-temperature ashing) is straightforward and scalable, resulting in amorphous silica known for high reactivity. The subsequent metal impregnation step can be tuned by adjusting metal salt concentration or repeating loading cycles to achieve desired metal content. In choosing the metal dopants, cost and availability are important factors: Ag is the most costly, while Cu and Zn are inexpensive and Bi is of moderate cost (but still cheaper than Ag per unit mass). Considering performance, Cu and Bi emerge as particularly attractive alternatives to Ag in this study, since they achieved equal or greater iodine uptake at a fraction of the cost. Zn, despite its low cost, gave only modest benefits under ambient conditions; its use might only be justified if improved by higher loadings or by applying elevated temperatures during capture. In practical nuclear waste off-gas systems, operating temperatures can be higher (e.g. 100–200 °C in some scrubbers), which could enhance the reaction of ZnO with I₂ and potentially improve Zn-sorbent performance. Indeed, higher temperature would likely boost all chemisorption reactions (and help overcome kinetic barriers for Zn), though excessive heat might also risk sintering the silica or volatilizing certain species (for example, BiI₃ decomposes/sublimes at T > 300 °C). Thus, an optimal temperature window would need consideration. The fact that our sorbents work well at 25 °C is promising for “cold” capture of iodine (e.g. in ventilated environments or during ambient releases).

Stability of the spent sorbents is another consideration. The metal iodides formed (AgI, Cul, BiI₃, ZnI₂) have different stabilities: AgI and BiI₃ are extremely insoluble

in water, which is advantageous for long-term immobilization of captured iodine. Cul has a very low solubility as well (on the order of a few ppm), so it would likely remain on the sorbent unless strongly leached. ZnI₂, however, is highly water-soluble; thus Zn-doped silica could release iodine if it got wet, making it less suitable for humid conditions unless encapsulated. BiI₃ can hydrolyze to BiOI in the presence of water, but BiOI is also a stable, insoluble solid, so bismuth still effectively immobilizes iodine upon eventual exposure to moisture. In many nuclear applications, the sorbents would be used in dry off-gas streams and then eventually incorporated into a solid waste form (e.g. embedded in glass or concrete for disposal). From this perspective, Ag, Cu, and Bi offer more secure long-term containment of iodine than Zn, aligning with the superior performance and selectivity we observed for Ag, Cu, Bi in capture.

In comparing our bio-silica sorbents to benchmark iodine sorbents, we find they perform quite competitively. Silver-exchanged zeolite (AgX) has been a standard in the field, with reported capacities around 100–200 mg·g⁻¹ at room temperature for I₂, increasing to 500–600 mg·g⁻¹ at higher iodine vapor pressures or longer exposure. Our Ag-doped silica (220 mg·g⁻¹ at 25 °C, 1 h) is in the same range as AgX under similar conditions. Copper-loaded activated carbons have shown fast and high uptake of iodine vapor in prior studies, and our Cu/SiO₂ likewise exhibited rapid, high-capacity capture (550 mg·g⁻¹). Bismuth-based sorbents are a newer development; for instance, Bi₂O₃ dispersed on a MOF composite took up 650 mg·g⁻¹ I₂ at 120 °C in a recent report. Impressively, our Bi-doped silica achieved 600 mg·g⁻¹ at room temperature, underscoring the efficacy of the Bi–silica system. Meanwhile, state-of-the-art porous framework materials (MOFs, COFs) can trap iodine in their pores in enormous amounts (several g·g⁻¹), but those capacities largely stem from physisorption in ultra-high surface area structures and often require specific conditions to fully load (and those materials may face issues of iodine release or structural instability). In contrast, the metal-doped silica approach secures iodine via chemical bonding into insoluble compounds, which is arguably more suitable for permanent sequestration in nuclear waste management. Silica itself is a stable, inert host that can even be incorporated into final waste glass forms. The captured AgI or BiI₃ could be likewise incorporated into cement or glass matrices for long-

term disposal without significant leaching. Thus, although our capacities are slightly lower than the absolute best physisorptive sorbents, the trade-off is a simpler, greener synthesis and a more secure form of captured iodine.

In summary, doping rice husk-derived silica with small amounts of Ag, Cu, Zn, or Bi yields a series of sorbents that demonstrate a clear enhancement in iodine vapor capture. Copper- and bismuth-loaded silica in particular stand out, achieving iodine loadings near the upper end of what is reported for inorganic iodine getters. Silver-loaded silica, while somewhat less capacious, confirms silver's well-known effectiveness as an iodine getter. Zinc-loaded silica, with only moderate uptake, suggests that Zn is a less suitable choice for low-temperature iodine capture unless further optimized. Overall, these comparisons and analyses provide a comprehensive understanding of how each metal additive contributes to iodine sorption on bio-silica, guiding future efforts (for instance, exploring bimetallic combinations like Ag–Cu or Cu–Bi to exploit potential synergies).

Conclusion

This work presented a sustainable approach to synthesize amorphous silica from rice husk and functionalize it with metal additives (Ag, Cu, Zn, Bi) for iodine vapor capture. High-purity silica was obtained from rice husk by acid pretreatment and controlled combustion, yielding an amorphous, porous SiO₂ framework. The RHA silica was then modified by impregnating it with silver, copper, zinc, or bismuth nitrates, followed by calcination to disperse these metals as oxides (or partially reduced forms) on the silica surface. The metal-doped silica materials were tested for their ability to sorb iodine from the vapor phase in short exposures at ambient conditions.

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