



Research article

The formation of gold in woody biomass combustion ashes

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ARTICLE INFO

Keywords:

Biomass
Combustion
Ash
Gold
Leaching

ABSTRACT

This paper investigates the enrichment of gold through combustion and ash-leaching techniques utilizing woody biomass as a fuel source. It delves into the formation of gold in ashes derived from the fixed grate combustion of pelletized woody biomass containing noble metals, conducted at a pilot-scale boiler. The biomass sample was gathered from a brownfield land at an abandoned mining area, avoiding induced phytoextraction. The fuel contained <0.05 mg/kg gold, while the bottom ash, after heat exchanger ash, deposited ash, and fly ash contained 1.52 mg/kg, 1.99 mg/kg, 2.64 mg/kg, and 3.52 mg/kg of gold, respectively. Although the amount of fly ash is lower compared to bottom ash, the concentration of gold is the highest in fly ash, which follows the after heat exchanger ash and bottom ash. The concentration of gold was enriched by a three-stage procedure of water leaching, acid leaching (10 % HCl), and alkaline leaching (5 % NaOH), after which 12.1 mg/kg and 12.6 mg/kg gold was found in the residues obtained from leached bottom ash and deposited ash, respectively. SEM was utilized to depict the morphology of gold, which appears in bottom ash as individual neat particles with a purity higher than 98 %. Pure gold particles in the size of 1–2 μm are presented in the after heat exchanger ash; meanwhile, gold in fly ash is primarily associated with potassium, sodium, sulfur, and oxygen. The findings in this study pave the way for reclaiming gold from bio-ores as well as assist in better understanding the formation of this precious metal in these secondary resources.

1. Introduction

Gold (Au) is renowned as a precious metal that has garnered significant human interest for centuries. This element possesses distinct physical and chemical properties such as exceptional conductivity, malleability, resistance to corrosion and oxidation, and aesthetic appeal [1]. Its applications are diverse across multiple disciplines ranging from jewelry and coinage (used as currency and investment) to medicine, electronics, or advanced technologies. Gold is scanty and dispersed in the earth's crust at a very low concentration of 0.004 mg/kg [2]. This precious metal is supremely valuable, its price, supply, and demand in the past years are illustrated in Fig. 1. The gold market experienced a significant decline in 2020 as a result of the COVID-19 pandemic, but it is currently rebounding. Despite the rising necessity of recycled gold, this source of production still represents a relatively small fraction of the total supply, approximately 25 %.

In addition to traditional mining, urban mining or reclaiming gold from secondary resources [4–6] such as electronic wastes [7–12], waste catalysts [13], ashes [7,14–18], tailings [19–21], or slag [22,23] is being explored. The urban mining approach strengthens the circular economy concept while it generates economic value [24]. Together with soil decontamination, bio-ores

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exploitation is an important contribution to the urban mining concept [25,26]. Woody biomass used for phytoextraction is a potential material for the recovery of gold [27]. Nevertheless, the current proportion of gold recycled from unconventional sources remains relatively low, primarily focusing on the recovery of gold from electronic waste [28].

Phytomining has emerged as an innovative technique to reclaim gold from low-grade minerals [29,30]. The holistic phytomining concept integrates three scientific domains: i) Phytoextraction serves as the initial stage, using plants to accumulate gold from contaminated soils [31–33]; ii) Subsequently, the precious metal is concentrated within solid remains known as bio-ores through the enrichment process of combustion [34,35]; iii) Ultimately, the extraction of gold from the bio-ores concludes the entire phytomining pathway [36,37].

One of the first recovery approaches covering all three stages in the gold reclaiming route was introduced by Lamb et al. (2001) [36]. In this route, the harvested plants were ashed and leached in a leaching agent of 2 M HCl. Following that, a solvent extraction process utilizing MIBK (methyl isobutyl ketone) was employed. Subsequently, the organic layer was subjected to the addition of NaBH₄ (sodium borohydrate) as a reducing agent, forming a black precipitate at the boundary between the layers. This precipitate was then heated at 800 °C, ultimately yielding metallic gold. However, this technique encountered various challenges. Specifically, the highly reactive reducing agent produced gas during the reaction, potentially degrading the solvent. The use of solvents entailed significant costs and raised environmental concerns. Separating the precipitate from the boundary layer proved to be difficult. Moreover, employing both thermal reduction and a reducing agent required a two-step process. Consequently, the authors worked on these issues and made certain advancements to propose an economically viable approach. In the subsequent procedure, 30 g of biomass containing 30 mg/kg Au was incinerated at 550 °C, and the resulting ash was leached in 300 mL of HCl. The aqueous phase was then extracted into 50 mL of MIBK and contacted with an equal volume of ascorbic acid (C₆H₈O₆). After 3.5 h, the solution was filtered, recovering 85 % of the gold in the precipitate. Although this extraction pathway was simplified to save the reagents and lower the costs, it still posed environmental limitations in terms of acid waste generation and high cost. Therefore, further research and development are necessary before this method becomes feasible for large-scale implementation.

Another technique for reclaiming gold from woody biomass was proposed in Ref. [37]. In this method, dried tobacco with a gold content of 1.2 mg/kg was subjected to an ashing process at around 300 °C. Borax (Na₂B₄O₇·10H₂O) and silver used as collector metal were added to the biomass ash and smelted at a temperature greater than 1000 °C to extract gold from the ash. Gold presented in the tobacco ash was recovered into the ultimate smelted product referred to as bullion. Although the outcomes were inconclusive, this study has provided a technically feasible approach for extracting gold from biomass ash.

Gold nanoparticles (AuNPs) synthesized from plant materials are gaining remarkable attention, where various parts or even the whole plant are utilized as source material. Recently, Akintelu et al. [38] published a comprehensive review focusing on various methods of synthesizing AuNPs, highlighting the influencing parameters, and describing the techniques used for characterization.

Although the phytoextraction of gold was thoroughly investigated in several studies mostly involving induced accumulation, where NH₄SCN [39,40], KCN [41,42], NaCN [37,43–45] or (NH₄)₂S₂O₃ [46,47] were used to treat the soil due to these chemicals increase the bioaccumulation factor (BF), studies representing the non-induced accumulation of gold in plants are being sparse possibly because of the lower BF. Additionally, less information is available regarding gold enrichment via combustion, while, to the best of the authors' knowledge, the appearance and formation of gold in the combustion ashes are not thoroughly investigated. Therefore, this study investigates the enrichment of gold by combustion and ash leaching techniques utilizing pelletized woody biomass used as fuel, while describing the behavior and depicting the formation of gold in the combustion system is supported by ICP and SEM methodologies.

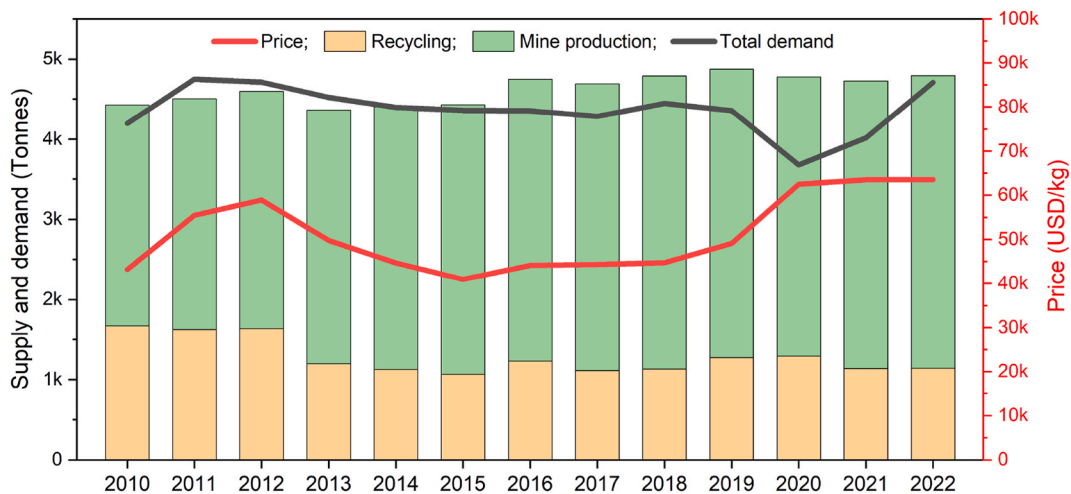


Fig. 1. Recent price, supply, and demand of gold, data obtained from Ref. [3]. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

2. Materials and methods

Biomass utilized in this research is harvested from contaminated land located in Gyöngyösoroszi, Hungary, where industrial mining operated until 1986. The major contaminants of the abandoned area are zinc, lead, copper, and cadmium. The plant species living in that brownfield include walnut, wattle, poplar, oak, pine, birch, and bushes. All parts of plants comprising trunk, branches, and leaves were collected and left in a laboratory for a few weeks for air drying. Afterward, the plant parts were shredded individually and then blended in the portion of 9 % leaf, 16 % branches, and 75 % trunk, correlating with a real tree mass ratio. The biomass blend was pelletized to the required dimensions for the boiler operation. The pellet was 10–30 mm long and 6 mm in diameter; its general properties are presented in Table 1.

The biomass pellet was incinerated in a fixed-grate pilot-scale boiler with continuous operation to enrich Au concentration from the biomass into ashes. The boiler performance varied between 10 and 30 kW_{th}. The average values of combustion temperature and flue gas temperature were 819 °C and 113 °C, respectively. Combustion solid remains were captured from divergent positions in the burning system, illustrated in Fig. 2, along with the measurement setup. Bottom ash was taken from the ashtray located below the combustion chamber, meanwhile, after heat exchanger ash was sampled from the chamber after the water heat exchanger. The deposited ash was collected from the surface of the air heat exchanger. The fly ash collection was implemented by an isokinetic fly ash sampling system utilizing Dekati® PM10 three-stage cascades impactor, and the average concentration of fly ash in the flue gas was 362 mg/Nm³. The impactor located outside the stack is heated up to the sample temperature by a heating jacket. The flow velocity inside the nozzle of the impactor is set to be equal to the velocity of the flue gas flow, aiming to isokinetically capture fly ash particles that pass through the nozzle without disturbing their paths. The sampling process was commenced after the boiler reached steady-state operational conditions, and the capturing method met the regulations of the ISO23210 standard. A Horiba PG350 flue gas analyzer analyzed the composition of flue gas. The flue gas's typical CO, O₂, and CO₂ concentrations were 2945 ppm, 6.7 vol%, and 12.9 vol%, respectively. The high-temperature flue gas from the combustion chamber first passes through a water heat exchanger and then an air heat exchanger for transferring thermal energy. Subsequently, the fly ash particles present in the flue gas are captured by an isokinetic fly ash sampling system prior to emission into the environment. During the combustion process, pelletized biomass from an attached fuel tank is fed automatically to the burner by a screw conveyor. The thermal input was regulated by the rotation speed of the fuel screw conveyor, while the combustion air was regulated based on the oxygen content of dry flue gas. A variable frequency drive connected to the stack fan controlled the flue gas pressure. A typical experiment lasted 8 h of operation (after the initial heat-up period).

Following the combustion process, leaching experiments were conducted as the first step to reclaim gold from biomass-derived ashes. The leaching process removes impurity substances and enriches gold in the leaching residues. The flow chart of the leaching procedure is depicted in Fig. 3, including three major stages: water leaching, acid leaching (10 % HCl), and alkaline leaching (5 % NaOH). As a result of the leaching process, a significant amount of impurities was eliminated accounting for more than 95 % of ash samples. Gold is expected to be enriched in the obtained leaching residues.

The woody biomass (WB), bottom ash (BA), after heat exchanger ash (HXA), deposited ash (DA), fly ash (FA), and leaching residues derived from leaching of bottom ash (LB), leaching of after heat exchanger ash (LHX), and leaching of deposited ash (LD) were chemically analyzed by ICP (Inductively Coupled Plasma) spectrometry. PerkinElmer Avio 200 inductively coupled plasma-optical emission spectrometer (ICP-OES) and ICP mass spectrometry (ICP-MS) were utilized. The samples were prepared based on the Hungarian standard MSZ EN 13346:2000.

Biomass combustion solid remains and leaching residues were characterized using SEM (scanning electron microscopy) and EDS (energy dispersive spectroscopy). SEM examinations of biomass-derived ashes aim to investigate the formation of gold in the ashes as well as to find evidence for the ICP measurement results. The SEM examination was performed by employing a ZEISS EVO MA10 scanning electron microscope that is equipped with backscattered and secondary electron detectors combined with EDS. In preparation for the SEM-EDS examination, representative sections of the solid samples were suspended onto aluminum plates.

3. Results and discussion

Although the concentration of gold in the fuel was below the detection limit (<0.05 mg/kg), the concentrations of gold in the BA,

Table 1
Ultimate, proximate, HHV, and density analysis of the biomass pellet used for combustion.

Ultimate, % by weight	N _{db}	0.40
	C _{db}	46.75
	H _{db}	6.05
	S _{db}	0.01
	O _{db}	33.43
Proximate, % by weight	Fixed carbon	34.10
	Volatile _{db}	52.53
	Moisture _{db}	11.07
	Ash _{db}	2.30
HHV _{db} , MJ/kg		18.2
Density, kg/m ³		422

HHV: higher heating value, db: dry basis, ^a: by difference.

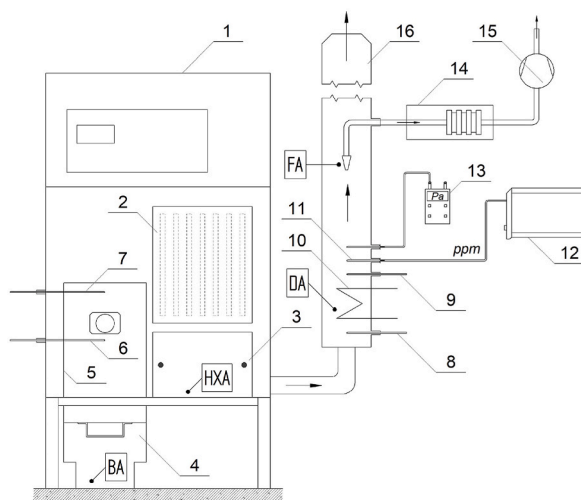


Fig. 2. Schematic illustration of the measurement setup: (1) boiler body, (2) water heat exchanger, (3) chamber after the water heat exchanger, (4) ashtray, (5) combustion chamber door, (6, 7, 8, and 9) thermocouples, (10) air heat exchanger, (11) gas sampling probe, (12) portable flue gas analyzer, (13) manometer, (14) impactor, (15) pump, and (16) to VFD controlled flue gas fan. Ash samples: BA – bottom ash, HXA – after heat exchanger ash, DA – deposited ash, FA – fly ash.

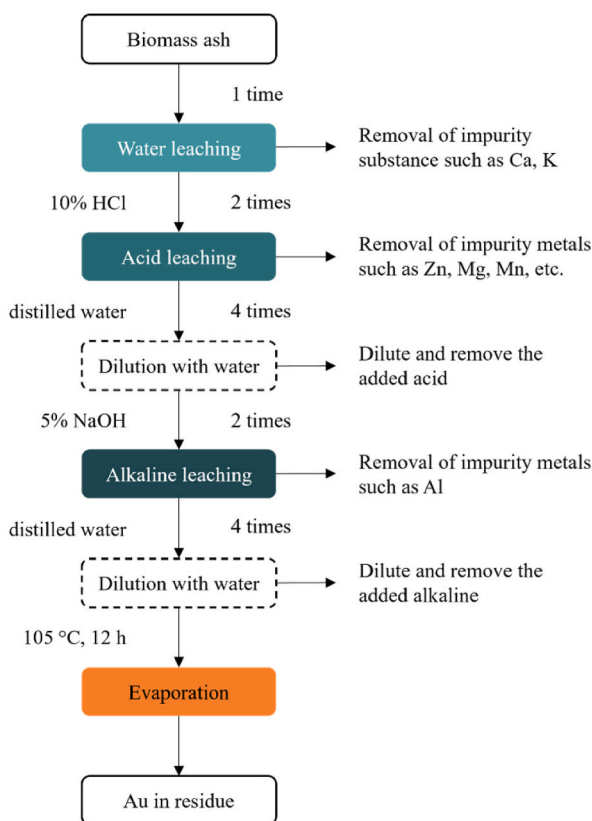


Fig. 3. Flow chart of leaching procedure of combustion ashes.

HXA, DA, and FA were 1.52 mg/kg, 1.99 mg/kg, 2.64 mg/kg, and 3.52 mg/kg, respectively, meaning that gold concentrations are greater moving forward in the combustion and flue gas system. The minimum quotients of gold level in ashes to that in biomass (calculated based on the detection limit) are 30, 40, 53, and 70 for bottom ash, after heat exchanger ash, deposited ash, and fly ash, respectively. These relatively high ratios demonstrate the efficacy of the combustion process in heightening gold concentration from

the biomass into combustion solid remains. The residues obtained from the leaching of bottom ash and deposited ash contain 12.1 and 12.6 mg/kg Au, respectively, which are 8 and 5 times higher than the Au concentration in the corresponding ashes. The greater levels of gold in the leaching residuals indicate that the leaching process effectively enriches this noble metal. The integration of the combustion and leaching approach substantially enhances the gold content. Gold concentrations in the leaching residuals such as the bottom ash-derived residue and the deposited ash-derived residue fold at least 242 and 252 times, respectively, compared to its levels in the biomass. The leached ash samples also have the benefit of having more discoverable gold particles during the SEM analysis as most of the ballast material is removed.

Girling and Peterson [48] performed experiments regarding gold uptake of various plants grown in solutions containing radioactive gold chloride or gold cyanide. Diffuse gold distribution with special accumulations in the leaf tips was shown by radioautography, while they forecasted the possibility of metallic form. Additionally, the localization of gold within the leaf was less specific in the case of gold chloride treatment. Based on the analysis performed in the recent study, the metallic gold formation within the plant was further supported by SEM. Fig. 4a shows an example of a gold structure found in the leached bottom (LB) ash sample. The surface of this particle was analyzed by EDS, which shows that the particle is evidently in neat gold form containing >98 % Au (Fig. 4b). The shape and the lamellar structure suggest that the particle was present in the plant in the same or similar form as seen in the picture. It is hypothesized that the particle falls down into the ashtray in a relatively short period of time due to its size and dense manner, meaning that the residence time of this particle in the heating zone is small; therefore, signs of heat treatment cannot be seen.

An example of another scenario can be seen in Fig. 5a, where the size and shape of the particle reveal that it was subjected to heat enough to partially melt the gold, which practically eliminates the lamellar structure but is insufficient to create droplet-like forms. Based on EDS analysis, this particle also consists of gold only (>99 % Au, Fig. 5b). Figs. 4 and 5 typically show the two borders of the overall appearance spectrum. Anything in between is the most general in the bottom ash samples, i.e. partial heat treatment can be observed on such particles combined with significantly smaller particles attached to the body surface (Fig. 6). The shape of these particles can be described as porous, irregular and rounded with low sphericity. In comparison, they have only minor elemental associations as the examined BA and LB gold particles consist of more than 98 % Au.

While the most significant gold particles by size appear in the bottom ash, a considerable amount of Au escapes the combustion chamber with the upward flowing flue gas, which has the ability of catching and transporting particles by its aerodynamical properties. It is supposed that Au particle sizes bigger than that found in the bottom ash won't appear in any subsequent parts of the combustion system. Therefore, the gold appearing in the sampling point of after heat exchanger ash should contain only smaller gold particles. This fact is supported by the SEM analysis, an example is shown in Fig. 7, where the tiny gold particle is attached to the surface of a bigger fly ash particle (Fig. 7a) or appears in neat form independently (Fig. 7b). Various scenarios describe the origin of such gold particles: 1) the particle is originated from the plant as is, however, the shape of the particle may vary depending on the residence time in the firing zone together with the temperature of combustion; 2) a bigger gold particle was broken into smaller fragments due to the lamellar structure and resulting in a smaller sized gold particle enough for escaping the combustion chamber; 3) the gold particle can melt as it flies through the high temperature zone due to the relatively small size resulting near droplet shaped forms; 4) the combination of the 1–3 scenarios.

The next sampling point in the combustion system is the wall of the air heat exchanger, which is considered as a source of deposited ash. Although individual 1–2 μm gold particles were found in the HXA, the results show that DA contained Au associated with other compounds indicating that the gold rather appears as a part of solid particles. Fig. 8 shows an example of such finding, including the SEM image (Fig. 8a) and the EDS spectrum (Fig. 8b), where mostly K (21.6 %), S (4.5 %), O (20.6 %), and Al (9.4 %) elements were also detected. The SEM analysis of fly ash suggests similar behavior to the deposited ash, as the gold is typically associated with K, Na, S, and O elements in all the cases during fly ash examination. One example is shown in Fig. 9a, where number 1 (Fig. 9b) highlighted in

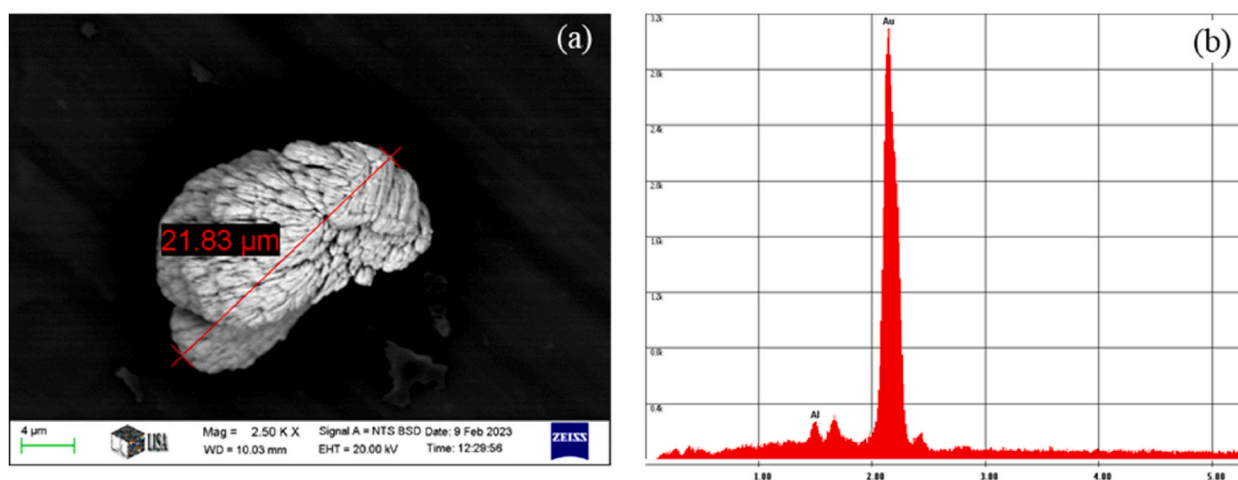


Fig. 4. Appearance of gold in the leached bottom ash sample. a) SEM picture, b) EDS spectrum, >98 % Au. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

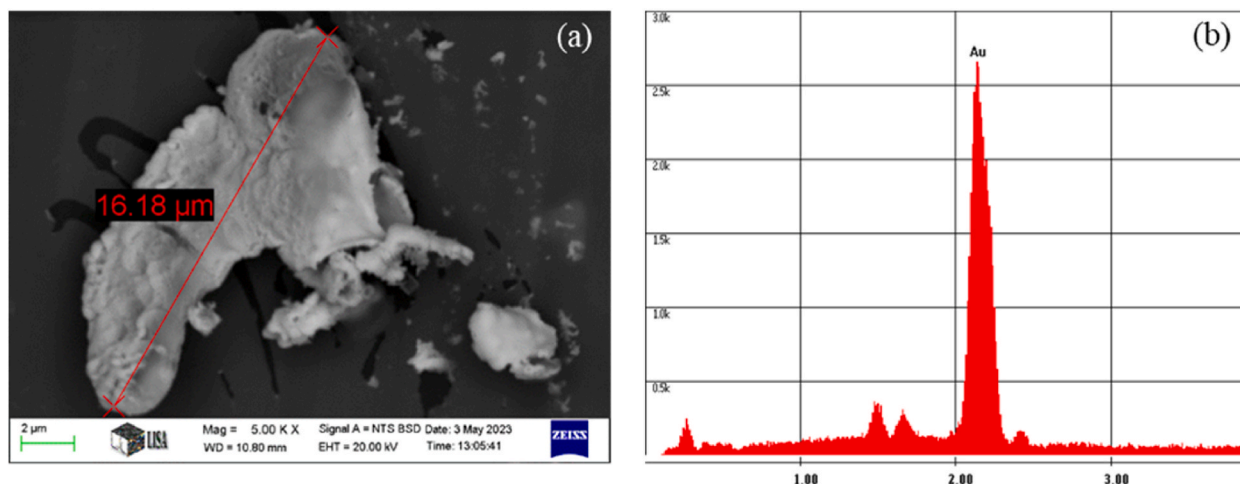


Fig. 5. Appearance of gold in the leached bottom ash sample. a) SEM picture, b) EDS spectrum, >99 % Au. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

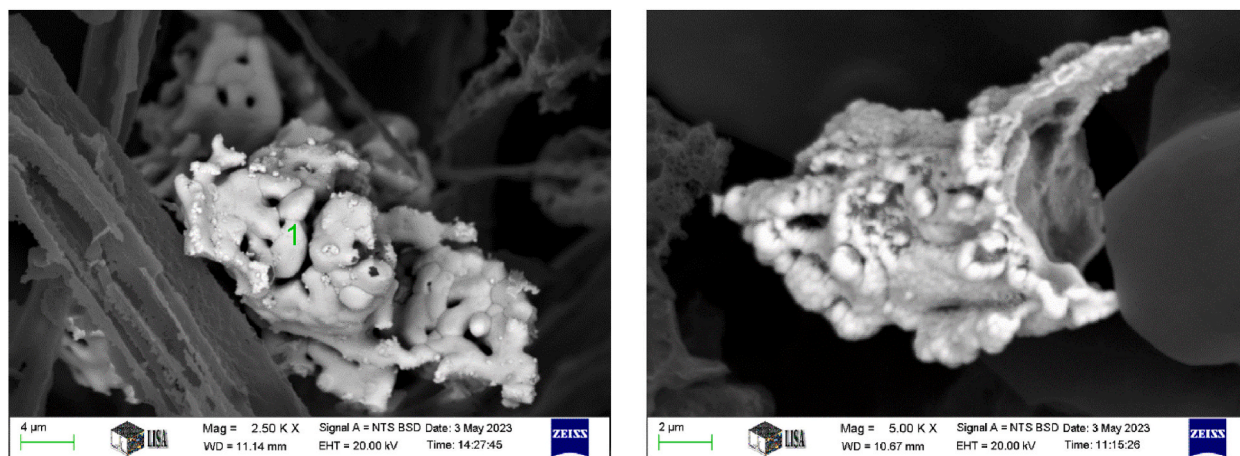


Fig. 6. Appearance of gold in the leached bottom ash sample. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

the SEM image denotes the background primarily containing carbon. Meanwhile, number 2 (Fig. 9c) (containing 17.9 % Au and 25.5 % K, 3.4 % Na, 4.2 % S, 21.3 % O) and number 3 (Fig. 9d) (consisting of 30.9 % Au and 20.2 % K, 1.4 % Na, 6.8 % S, 21.6 % O) represent the occurrence of Au in the fly ash particles. The association structure suggests that the gold in melted or partially melted form present in the combustion chamber should hit another appropriate particle, during which the gold becomes a part of the other particle. A certain amount of gold escapes the combustion chamber by this pathway, which can be considered as gold loss if fly ash is not treated. Additionally, since BA contains the most amount of gold, including particles in the tenth of microns by size, followed by the HXA with 1–2 μm particles, fly ash might also contain neat gold particles as well in the system utilized in this study, but supposingly at the nanometer scale.

4. Conclusions

The combustion of pelletized woody biomass harvested from a brownfield land was investigated from gold behavior point of view, which is a potential pathway in gold phytomining. Au concentrations measured by ICP in bottom ash, after heat exchanger ash, deposited ash, and fly ash were 1.52 mg/kg, 1.99 mg/kg, 2.64 mg/kg, and 3.52 mg/kg Au, respectively. The collected ashes were subsequently subjected to a three-stage leaching approach of water leaching, acid leaching (10 % HCl), and alkaline leaching (5 % NaOH), resulting in 12.1 and 12.6 mg/kg of gold in as the bottom ash-derived leaching residue and the deposited ash-derived leaching residue, respectively. The integration of the combustion and leaching process could significantly enrich the gold concentration from the woody biomass into the leaching residual.

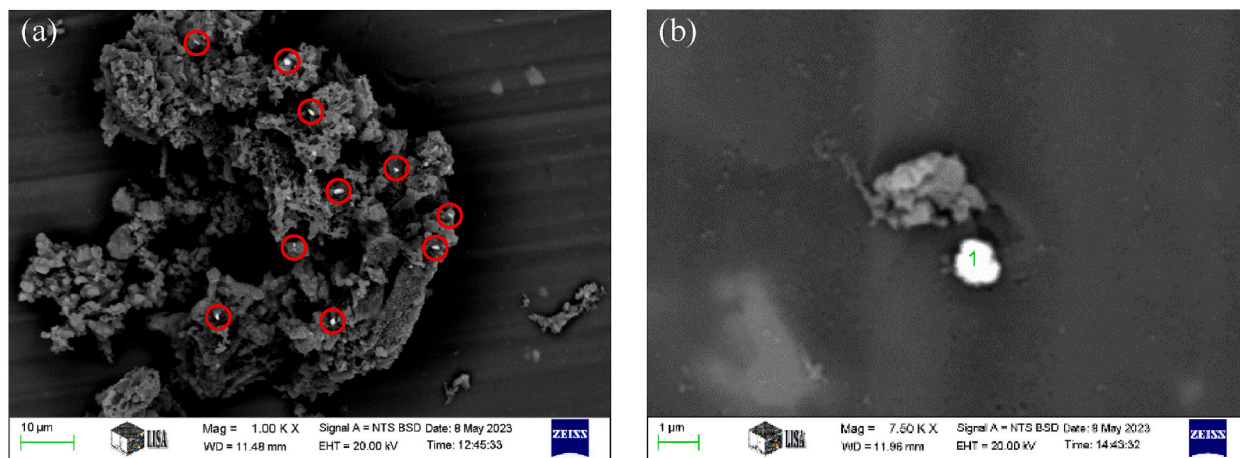


Fig. 7. SEM image examples of Au in after heat exchanger ash. a) the gold is attached to a bigger ash particle (indicated by the circles), b) the gold appears in neat form independently. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

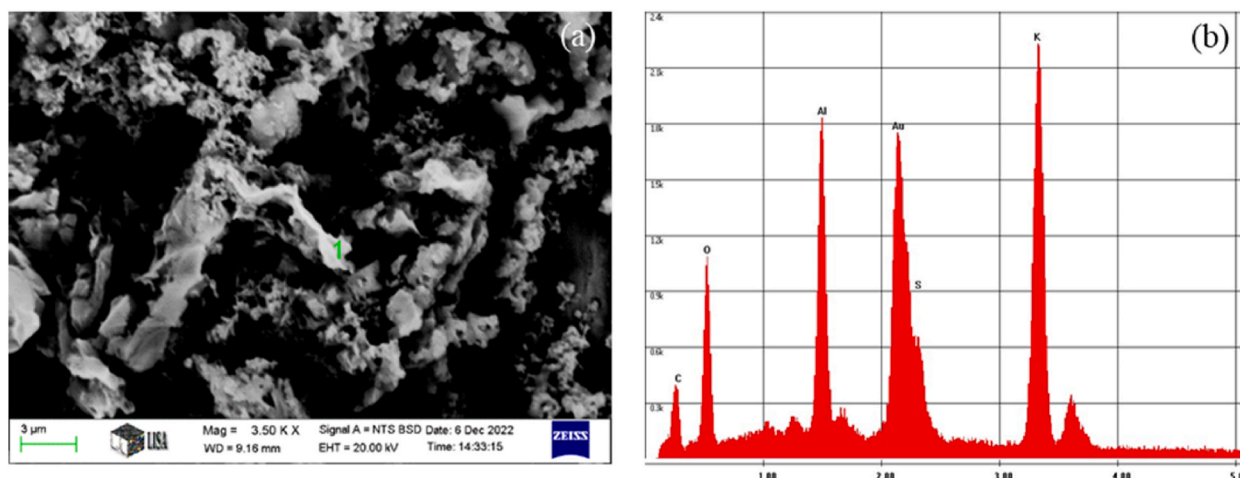


Fig. 8. Example of Au in deposited ash. (a) SEM image, (b) EDS spectrum.

The SEM-EDS outcomes are consistent with the ICP analyses as gold was found in all the combustion ashes, but the formation associated with gold depends on the location of sampling. The gold particles are presented in neat form in the bottom ash with the purity of higher than 98 %. Additionally, the size of a gold particle is significantly higher in bottom ash compared to other solid residues, which is elucidated by the fact that the relatively heavy gold particles cannot be transported out of the combustion chamber by the flue gas flow. Pure gold particles in the size of 1–2 μm were observed in the ash collected in the chamber after the heat exchanger, meanwhile, in fly ash and deposited ash, this noble metal is predominantly associated with potassium, sodium, sulfur, and oxygen. The SEM findings assist in better understanding the formation of gold and strengthen the feasibility of recovering this valuable element from unconventional resources.

Data availability statement

The authors confirm that data supporting the findings of this study are available within the article.

CRediT authorship contribution statement

Truong Dinh: Writing – original draft, Visualization, Software, Methodology, Investigation. **Helga Kovács:** Writing – review & editing, Validation, Resources. **Zsolt Dobó:** Writing – original draft, Validation, Supervision, Conceptualization.

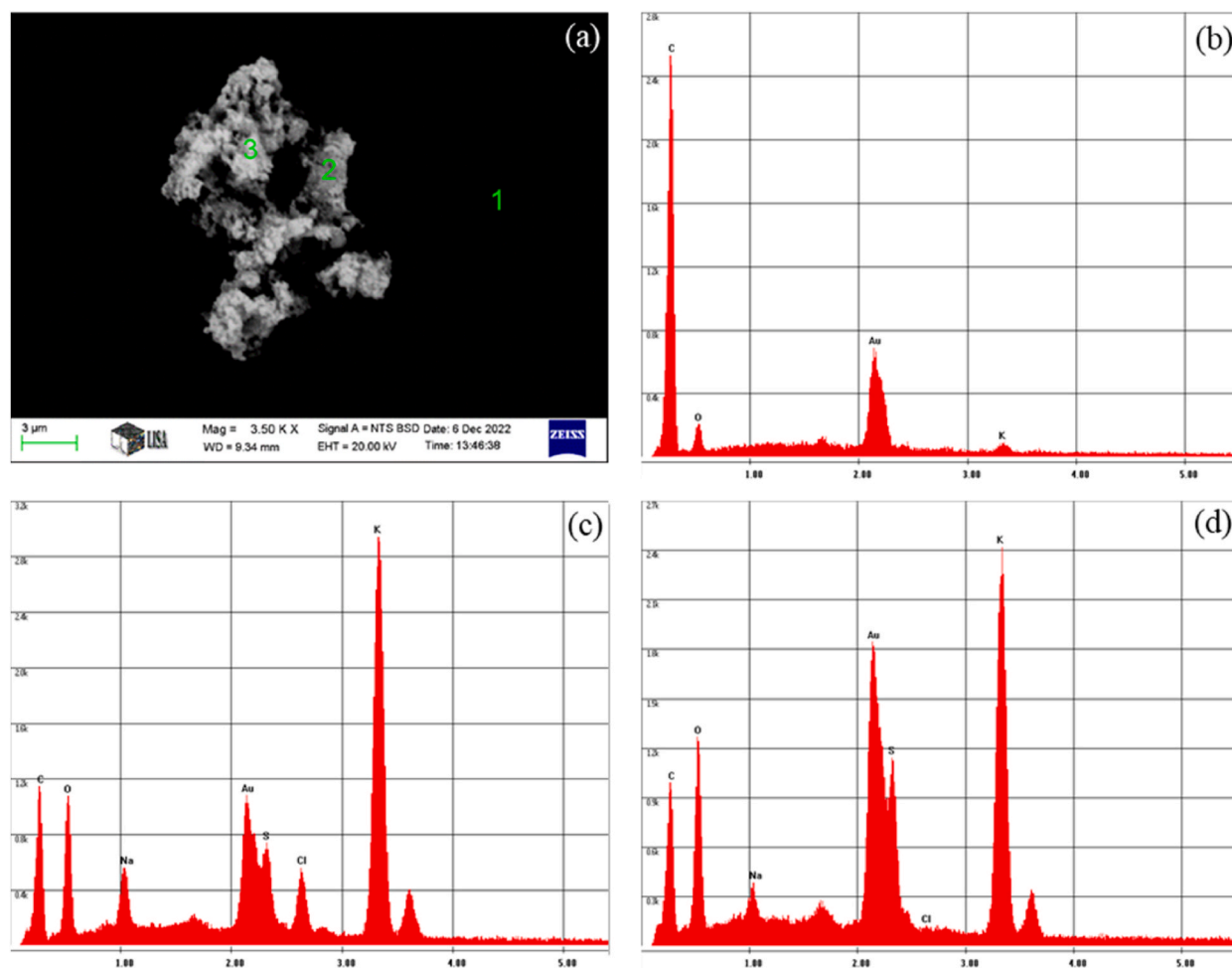


Fig. 9. Example of Au in fly ash. (a) SEM image, (b) EDS spectrum point 1, (c) EDS spectrum point 2, (d) EDS spectrum point 3.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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