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Toward environmentally favorable nano-sensing by production of reusable gold nanoparticles from gold nano-waste: life cycle and nanocircular economy implications†

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The environmental impacts of gold nanoparticle (AuNP)-based sensing were investigated using a cradle-to-grave life cycle assessment (LCA). The LCA model considered AuNP synthesis, surface functionalization, an AuNP-based detection assay, and disposal. Additionally, the model incorporated two potential Au nano-waste reuse strategies reliant upon α -cyclodextrin (α -CD) or Triton X-114. The results show that, across ten midpoint categories that >80.4% of the environmental impacts arise from AuNP synthesis thus demonstrating the benefit of reuse of Au nano-wastes. Importantly, the two different reuse strategies enhance the environmental sustainability of the sensing application. Gold recovery contributed to a significant reduction in the amount of pristine Au³⁺ initially required despite the additional chemical and electrical demands of the reuse processes. Sensitivity analysis focused on two variables (*i.e.*, recovery efficiency and the number of reuse cycles) indicated that the environmental favorability of the sensing application is dominated by recovery efficiency. Finally, the reuse of Au nano-waste reduces the energy demand of nano-sensing and the total cost of AuNP-based industries, thus illustrating energy and circular economy implications.

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Environmental significance

The manufacture and application of gold nanoparticles (AuNPs) have consistently expanded across many fields. However, the finite resource of gold ore, or lack thereof, continues to raise questions about the environmental sustainability of AuNP applications. There is limited knowledge of the environmental impacts of AuNP applications that must be addressed if practitioners want to support nanosustainability. Life cycle assessment (LCA) showed reduction of environmental burden of AuNP-based sensing through the reuse of Au nano-waste and illuminated greener alternatives to current AuNP production and use practices, thus illustrating energy and circular economy implications.

Introduction

The production of nanomaterials has been increasing in accordance with the surge in the development of nanotechnology-based techniques. Nonetheless, exploration of the environmental impacts of these techniques is relatively limited. It is imperative to rigorously assess the environmental sustainability of nanomaterial production to prevent unintended negative consequences.¹ Additionally, it is important to evaluate the environmental impacts of the nanomaterial use stage. Life cycle assessment (LCA) is a rigorous tool that allows practitioners to investigate the characteristics of a particular technique prior to field application.^{2–4} To date, LCA has been extensively used to evaluate the environmental impacts of the manufacture of a range of nanomaterials (*e.g.*, Au, Ag, TiO₂, FeO_x).^{5–10}

Gold nanoparticles (AuNPs) hold promise in many applications covering medical diagnostics and imaging for

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† Electronic supplementary information (ESI) available: (1) Experimental details; (2) detection of ssDNA using aptamer-functionalized AuNPs; (3) simulation details; (4) Tables S1–S5: inputs of synthesis, functionalization, detection, recovery, and recycling processes; (5) Table S6: 1 mg of customized chemicals from available databases; (6) the stoichiometry of the products and reactants for customization; (7) uncertainty analysis of environmental impacts (ozone depletion, smog, acidification, eutrophication, respiratory effect; (8) sensitive analysis of environmental impacts (global warming, carcinogenic, non-carcinogenic, ecotoxicity, fossil fuel depletion); (9) sensitive analysis of cumulative energy demand (CED). See DOI: <https://doi.org/10.1039/d3en00505d>



health care and dentistry, the electronics industry of inks in photovoltaics, and sensors.¹¹ The U.S. market value of AuNPs has reached ≈ 1180 million USD.¹² Free electrons on the AuNP surface oscillate when excited by incident light and generate unique localized surface plasmon resonance (LSPR).¹³ AuNP-based sensing has been widely applied owing to their unique plasmonic properties and their high surface-to-volume ratio.^{14,15}

While prior studies have evaluated the environmental impacts of AuNP synthesis methods,^{5,6,10} the environmental impacts of specific AuNP-based applications have not been explicitly explored. The acceleration in widespread synthetic production of AuNPs has led to increasing amounts of Au nano-waste. We previously reported that the disposal of Au nano-waste results in negative environmental impacts.¹⁶ Further, the declining amount and quality of natural gold ore grades remains a substantial challenge if Au-based nanotechnologies are to be sustainable.¹⁷ The nanotechnology field has recently been inspired by the 'circular economy' concept, a sustainable design framework to return materials at the end of their service life into a circular product ecosystem.¹⁸ This aspirational 'nanocircular economy' endeavors to build a closed-loop system involving the generation and reuse of nanomaterials that differs from the conventional 'make, use, dispose' linear model.¹⁹ Under these circumstances, the reuse of Au nano-waste has received attention as a promising strategy to reduce environmental burdens and promote sustainability.

In this study, we evaluated the environmental impacts of AuNP-based optical sensing. Furthermore, we investigated the environmental sustainability of the reuse of Au nano-waste as a means to advance the nanocircular economy. We used LCA as a quantitative framework to evaluate the environmental impacts of AuNP-based sensing across the stages of synthesis, functionalization, detection assay, disposal, and reuse (as applicable). As an exemplary optical sensing approach, we examined an aptamer-functionalized AuNP-based colorimetric detection method. In the presence of target ssDNA, the color of an aptamer-functionalized AuNP suspension changes due to hybridization-induced aggregation, resulting in an absorbance peak shift to longer wavelength (*i.e.*, red-shift). The application of aptamer-functionalized AuNPs enables rapid and sensitive colorimetric detection with the potential for low-cost and simple operation.^{20,21}

Materials and method

LCA framework

Herein, the environmental impacts of aptamer-functionalized AuNP-based sensing were assessed using LCA. As suggested by the International Organization for Standardization (ISO) this LCA included the following four steps: 1) goal and scope, 2) life cycle inventory (LCI) analysis, 3) life cycle impact assessment (LCIA), and 4) interpretation.²

Goal and scope. The goals of this study were 1) to assess the environmental impacts of a colorimetric detection method that uses aptamer-functionalized AuNPs; and 2) to investigate the environmental favorability of two Au nano-waste reuse strategies. The system boundaries included cradle-to-grave processes with the closed-loop of Au nano-waste reuse (Fig. 1). The system consists of AuNP synthesis, functionalization, detection assay, reuse (if applicable), and disposal. The functional unit employed was 100 femtomoles of functionalized AuNPs – this amount is equivalent to that used in a typical detection assay.²² In this study, aptamer-functionalized AuNPs were used for the detection of ssDNA (ESI†). The class 1 integron-integrase gene (*intI1*) was benchmarked as a model ssDNA target. It has been recognized as a genetic marker for anthropogenic pollution and is commonly linked to genes conferring resistance to antibiotics, heavy metals, and disinfectants.²³

Scenario 1 included all processes except a reuse step, representing the conventional 'make, use, dispose' linear model. Scenarios 2 and 3 examined two separate Au nano-waste reuse processes, representing a closed-loop circular model with reuse of Au nano-waste. At the grave stage, we simulated two recovery methods followed by strong acid-mediated recycling of Au nano-waste. Prior studies have reported several successful approaches to recovery from diverse nanomaterial wastes.^{10,24–31} We selected two recovery pathways for Au nano-waste that are either Au-specific or solution-based recovery methods since the approach considered here was aqueous sensing. It is noted that the choice of recovery methods would impact the environmental burden of the process due to different chemical and electricity demands and recovery rates. First, we simulated the use of α -cyclodextrin (α -CD) which interacts with the square planar complex AuBr_4^- , after acid dissolution of Au nano-waste.¹⁰ Second, we simulated the cloud point extraction (CPE) method that involves the thermo-reversible liquid-liquid phase transition of Au nano-waste using a nonionic surfactant (Triton X-114).^{25,26} In the literature, the α -CD and Triton X-114 based recovery methods have reported ~ 60 – 80% (ref. 10) and $>90\%$ (ref. 32) mass recovery efficiencies, respectively. The efficiency presumably varies due to uncertainties in the experimental conditions.

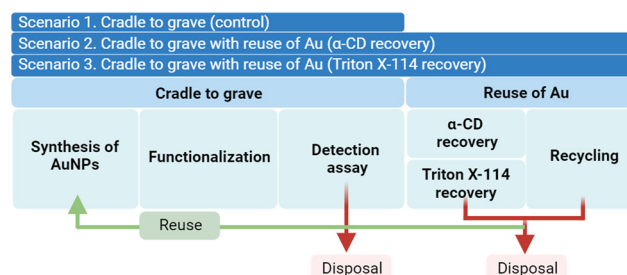


Fig. 1 System boundary of AuNP-based sensing with three different scenarios: control (without reuse) and two Au nano-waste reuse strategies.



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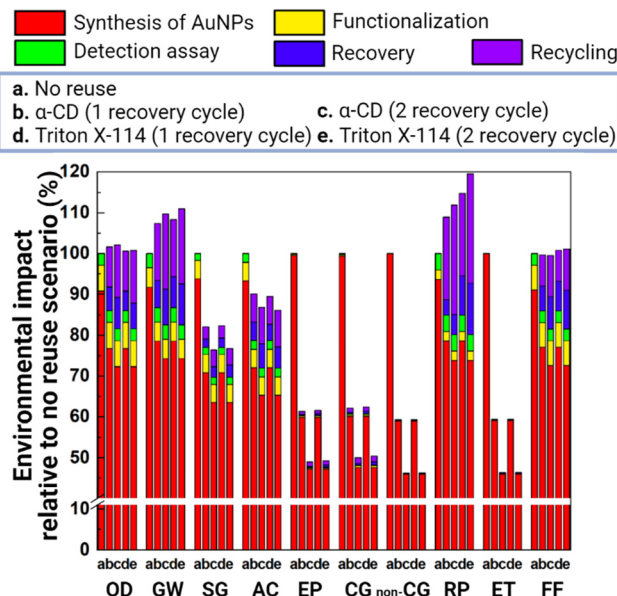


Fig. 2 Ten categorized relative environmental impacts of the sum of all processes (synthesis, functionalization, detection assay, recovery, and recycle) in the system boundary of the AuNP-based sensing application. No-reuse scenario (a) and two recovery method-mediated reuse scenarios (b and c: α -CD and d and e: Triton X-114) assuming a recovery efficiency of 0.7 and either 1 or 2 reuse cycles. For comparative purposes, all scenarios were normalized to the no-reuse scenario. Impact categories: ozone depletion (OD), global warming (GW), smog (SG), acidification (AC), eutrophication (EP), carcinogenic (CG), non-carcinogenic (non-CG), respiratory effect (RP), ecotoxicity (ET), fossil fuel depletion (FF).

LCA results, we expect that methodological variations in functionalization and the detection assay would not significantly affect the overall environmental impacts.

The relative environmental favorability of scenarios incorporating reuse was investigated (indicated in Fig. 2 as “b–e”) and were compared to the no-reuse scenario. Higher recovery efficiency and multiple reuse cycles were expected to minimize environmental impacts owing to increased reuse of Au nano-waste. Based on the reported recovery rates of both recovery methods,^{10,32} a recovery efficiency was conservatively assumed to be 0.7 with either one or two reuse cycles for simulation. We found that the relative environmental impacts were primarily favorable except for the global warming (GW) and respiratory effect (RP) impact categories. In particular, the environmental impacts of the two reuse-incorporating scenarios decreased by ~ 40 and $\sim 54\%$ for eutrophication (EP), carcinogenic (CG), non-carcinogenic (non-CG), and ecotoxicity (ET) for either one or two reuse cycles. Two reuse cycles showed more favorable impacts than one, implying the potential utility of multiple reuse cycles. Both recovery methods predict negative impacts for GW and RP, showing impacts of $>106\%$. This means there was a greater environmental impact burden when there was a reuse scenario than the no-reuse one. For each of these categories, two reuse cycles had greater environmental burdens than one reuse cycle. This result can be attributed to the significant

burden on GW and RP of the chemical and electricity demands required for recycling. The uncertainty analysis provides the statistical significance of the differences from the scenarios (Fig. S3†).

The results imply the environmentally favorable benefits through a closed-loop circular model of AuNP-based sensing application compared to a conventional linear one without reuse scenario. It was found that the environmental burden of AuNPs-based sensing applications can be more than halved for some categories which depend on the recovery rate and reuse cycle. It is worth noting that shifting the nanomaterial application paradigm from linear to circular models could significantly reduce environmental impacts. For better support to nanosustainability, the environmental favorability of other nanomaterial applications in the circular model should be investigated as well.

Impacts of extended reuse

To illustrate the relative contribution of synthesis and recovery/recycling processes with more extended reuse cycles, two representative categories that each had favorable (SG) and non-favorable (RP) environmental impact of reuse were further investigated. Fig. 3 indicates that smog (SG) and respiratory effect (RP) of synthesis, recovery, and recycling processes as a function of the number of reuse cycles assuming a recovery efficiency of 0.6. A lower recovery efficiency of 0.6 was used in this case to simulate different scenarios with underperformed recovery. As expected, with an increase in the number of reuse cycles, the synthesis process had decreased impacts in SG and RP owing to the reduction in $[\text{Au}^{3+}]_{\text{pristine}}$, while there were increased impacts for the recovery and recycling processes due to their additional chemical and electrical demands. Both the increasing/decreasing trade-offs plateaued with a higher number of reuse cycles because $[\text{Au}^{3+}]_{\text{reuse}}$ decreased. Such trends were the same across all categories. However, the overall environmental impacts were more mixed because the contribution of each reuse cycle to the categories was different. In the case of SG, the scale of the impact of the synthesis process was $\sim 10\times$ larger than that for the combined

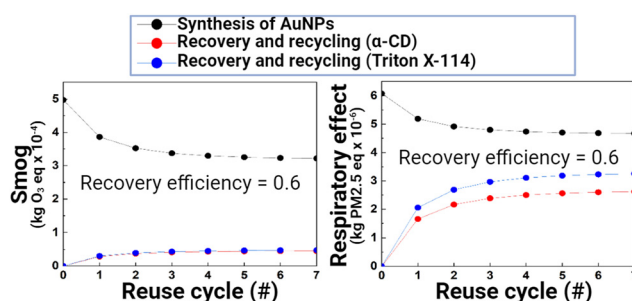
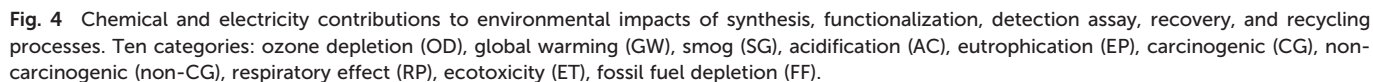


Fig. 3 Smog (SG) and respiratory effect (RP) of synthesis of AuNPs and two recovery/recycling processes with varying number of reuse cycles (#). The recovery efficiency was set to 0.6.



For the functionalization and detection assay, the use of a phosphate-buffered solution with 0.1% Tween-20 (PBS-T20) and electricity were dominant contributors, respectively. PBS-T20 was required to wash the nanoprobe after functionalization. The minimal impacts of the functionalization process support our prior assessment that alternative functionalization methods should not dramatically alter the overall environmental impacts. Similarly, the electricity required for the detection assay is an essential step required to denature the nanoprobe before hybridization occurred. Thus, any other hybridization buffer

Fig. 4 summarizes the chemical and electricity contributions to the environmental impacts of each process. For AuNP synthesis, electricity and HAuCl₄ (*i.e.*, [Au³⁺]_{pristine}) dominated the environmental impacts. In particular, HAuCl₄ constituted >95% of the impact for EP, CG, non-CG, and ET. These



recipes would not increase the environmental impacts significantly.

For α -CD and Triton X-114 based recovery methods, the major environmental impact contributors were HBr (50–75%) and electricity (40–90%) across all categories, respectively. Bromine is a corrosive and highly toxic chemical that is expected to have high environmental impacts across all categories.⁴⁰ The main difference between the two methods was the amount of electricity required. The α -CD-based method requires electricity only for sonication (0.24 kJ), while the Triton X-114-based CPE protocol needs electrical energy for heating (1.67 kJ) to recover 100 femtomoles of AuNPs (Table S4†).

For recycling, electricity dominated the impact due to the required HNO_3 boil-off step. To recycle 100 femtomoles of AuNPs, 3.88 kJ of energy were required to remove residual HNO_3 . This value is comparably high since the synthesis process required 4.44 kJ for heating (Tables S1 and S5†). Future research should focus on improving the efficiency of HNO_3 removal.

Sensitivity analysis of environmental impacts

The sensitivity of the environmental impacts of AuNP-based sensing was evaluated. Two variables (recovery efficiency and reuse cycle) were simulated and their concomitant environmental impacts were calculated. Recovery efficiency was the key factor determining the reduction in the amount of pristine HAuCl_4 required to make 100 femtomoles of

functionalized AuNPs. Higher recovery efficiencies have a decreased environmental impact and reflect the promise of reuse strategies. Multiple reuse cycles increase the amounts of chemicals used and the electricity required for recovery and recycling, but also reduce the amount of pristine HAuCl_4 required. Fig. 5 shows the environmental impacts of the reuse scenarios with α -CD (top row) and Triton X-114 (bottom row) mediated recovery methods in five selected categories with distinct patterns (OD, SG, AC, EP, RP). In this figure, all environmental impacts were normalized relative to the no reuse scenarios. We simulated recovery efficiency from 0 to 1.0 over seven cycles of reuse. The other five categories (GW, CG, non-CG, ET, FF), which show similar patterns, are illustrated in Fig. S4†. The required amounts of pristine HAuCl_4 and the additional chemicals and electricity for recovery and recycling were calculated using eqn (1) and (2). Simply, if recovery efficiency and reuse cycles lie on the contour line of <100%, reuse strategies indicated greater environmental favorability than the no reuse case and *vice versa*.

The overall sensitivity patterns for both reuse strategies were quite similar. OD (and FF) showed the least sensitivity to the two variables, while EP (and CG, non-CG, ET) showed the most. In the range of the two variables in this study, the relative impacts were 90–125% for OD and 17–93% for EP. Overall, the many horizontal contour lines imply that the reuse cycles were less sensitive to environmental impacts than recovery efficiency for most cases. For the OD category, both reuse scenarios turned out to be more environmentally

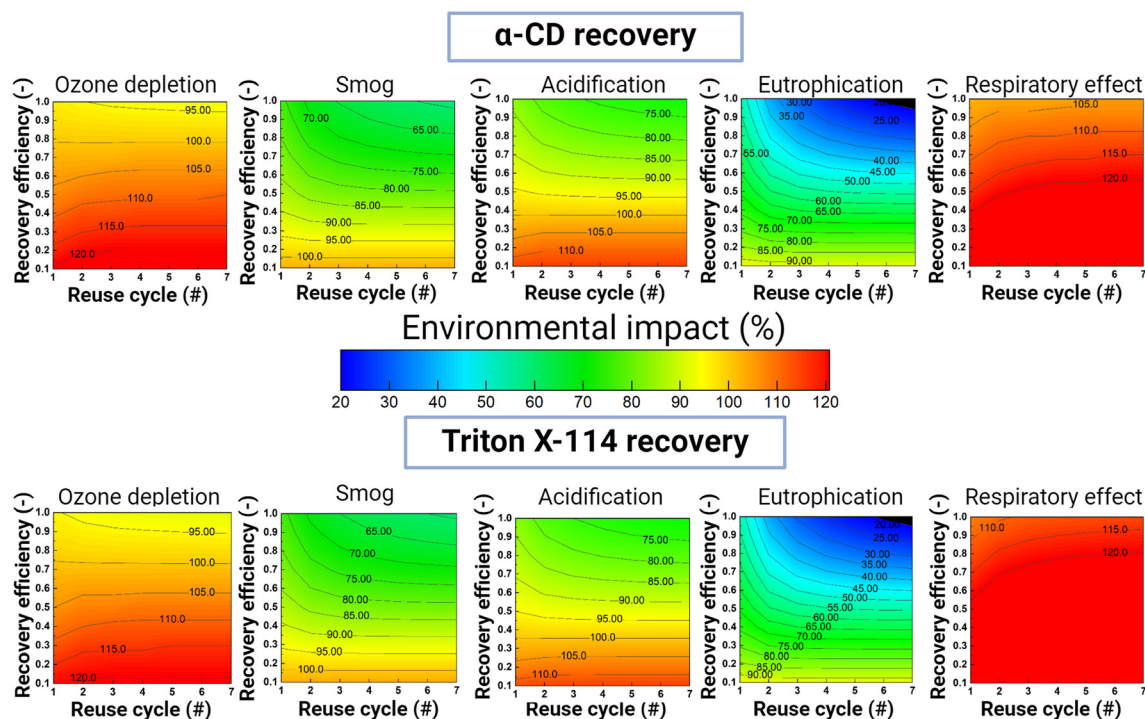


Fig. 5 Sensitive analysis of environmental impacts (ozone depletion, smog, acidification, eutrophication, respiratory effect) with varying recovery efficiency (0.0 to 1.0) and the number of reuse cycles (#). The environmental impacts were normalized by the no-reuse scenario.



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