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A Critical Study of Nanostructured Gold and Silver Plasmonic Devices with Ultra-Sensitive Localized Surface Plasmon Resonance for Biosensing Applications

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ABSTRACT

The critical analysis of nanostructured plasmonic devices based on ultra-sensitive localised surface plasmon resonance on nanostructured gold and silver and their use in bio-sensing has been presented in this paper. The paper investigates the physical mechanism of LSPR and studies the impact of the choice of material, geometries of nanoparticles, surface chemistry, and designs of hybrid on sensing performance. Gold nanospheres, nanorods, nanotriangles, nanorings, nanostars, silver nanoparticles and Au-Ag core-shell systems are considered in particular, and compared in terms of sensitivity in refractive index, figure of merit, capability to detect, selectivity and stability of operations. As it can be seen in the review, anisotropic and sharp-featured nanostructures tend to offer superior local confinement of electromagnetic fields and increased sensing sensitivity compared to plain spherical particles. Devices based on gold have demonstrated obvious benefits in chemical stability, biocompatibility, and biomolecular functionalisation whereas silver-based systems typically have sharper plasmonic resonances and have a greater capacity to enhance fields which makes them very popular in ultra-sensitive detection. Nevertheless, the oxidation, unsteadiness of the environment, and issues of reproducibility continue to make the practical application of silver very scarce. Hybrid AuAg systems could be viewed as the potential solutions as they are a combination of the high response of silver to light and enrichment of the strength and surface chemistry of gold. It is found in the study that the future viability of LSPR biosensors does not lie in the ability to attain high sensitivity in ideal conditions, but in the ability to ensure reproducibility, fouling resistance, stability in complicated biological conditions, as well as in fabricatable device structures. In general, nanostructured gold and silver plasmonic platforms have a great promise in being used as the next-generation label-free biosensing systems in medical diagnostics and environmental and point-of-care detection.

Introduction

Localized surface plasmon resonance (LSPR), and surface plasmids, in particular, have become a significant field of study in nanophotonics due to the ability of the collective oscillation of metallic nanoparticles conduction electrons to cause strong optical extinction, intense near-field enhancement, and high sensitivity to the surrounding dielectric environment [Anker *et al.*, 2011]. Unlike the propagation of surface plasmon resonance on a continuous film of metal, LSPR is limited to nanoscale dimensions, and is therefore of particular interest to miniaturised, labelling free, and low-volume sensing devices. It is these features that have made LSPR of much interest in biosensing, molecular diagnostics, chemical detection and applications in optical transduction [Haes and Van 2002, Unser *et al.*, 2015]. Gold (Au) and silver (Ag) are still the most significant plasmonic materials due to the ability to tune their optical activity to the visible and near-infrared spectrums by size, shape, composition, and local environment [Kelly *et al.*, 2003, Anker *et al.*, 2008, Mayer and Hafner 2011, Larsson *et al.*, 2007]. Gold is extensively preferred in biosensing due to its chemical stability, biocompatibility and established surface functionalisation chemistry (particularly by self-assembled monolayers using thiols). Silver, in turn, tends to have a reduced temperature resonance, smaller optical losses and high field enhancement, and can enhance the refractive-index sensitivity and spectral resolution; but is more susceptible to oxidation, corrosion and long time instability in actual operating

conditions [Mayer and Hafner 2011, Unser *et al.*, 2015, Loiseau *et al.*, 2019– Preston *et al.*, 2021]. This graceful yet somewhat hot-tempered manner of behaviour is not the only reason why silver-based devices usually work exemplarily in the lab and grump in more severe situations unless covered with coated or hybrid architectures [Mayer and Hafner 2011, Loiseau *et al.*, 2019, Preston *et al.*, 2021]. It is well established over many studies that sensing of LSPR devices is not only determined by the metal material but also by the geometry of nanoparticles and interfacial design. Compared to simple nanospheres, shape-controlled nanostructures, including triangular silver nanoparticles, gold nanoring, gold nanotriangles, gold nanostars, and Au-Ag core-shell nanoparticles exhibit greater field localisation and better sensitivity to refractive-index [Haes and Van 2002, Larsson *et al.*, 2007, Loiseau *et al.*, 2019]. It has been demonstrated by representative studies that gold nanorings can reach sensitivities to bulk refractive-index of 880 nm/RIU and above and that the gold nanotriangles have been able to reach about 468 nm/RIU as well as selective DNA hybridisation detection at room temperature [Larsson *et al.*, 2007, Soares *et al.*, 2014]. Similarly, 5-armed gold nanostars have been utilized in miniature optical-fibre sensing devices and Au-Ag core-shell nanoparticles have been employed to perform toxin biosensing in small but detectable LSPR shifts [Cennamo *et al.*, 2013, Loiseau *et al.*, 2019]. These data help realize that hotspots engineering, anisotropic morphology, and controlled coupling of plasmids are contributing factors to ultrasensitive biosensing instead of the selection of a

metal [Larsson *et al.*, 2007, Loiseau *et al.*, 2019]. This paper will critically discuss nanostructured gold and silver plasmonic structures with ultra-sensitive LSPR to be used in biosensing. The debate is on the effect of material selection, nanostructure geometry, bimetallic design/nanoengineer, surface functionalisation and device geometry on sensitivity, selectivity, figure of merit and practical detection limits. Simultaneously, the paper assesses the main obstacles that still impede wider biosensing implementation, which are spectral expansion, silver oxidation, signal loss in non-isolated systems, re-productibility of nanofabrication, and the challenge of transferring highly sensitive lab-scale systems into point-of-care systems [Mayer and Hafner 2011, Unser *et al.*, 2015, Loiseau *et al.*, 2019, Preston *et al.*, 2021].

Methodological section

Literature search and study identification-As the current paper is a critical study, rather than an experimental article in the laboratory, the approach to the methodology was to identify, screen, classify, and critically compare published works on nanostructured gold and silver plasmonic devices based on the localized surface plasmon resonance (LSPR) in the application of biosensing. The review design was based on the logic of transparent selection and reporting of evidence suggested in the PRISMA 2020 statement, especially when it comes to identifying studies, screening, assessing their eligibility, and planning their synthesis [Page *et al.*, 2021]. The search in the literature was conducted with the help of such major academic databases as Web of science, Scopus, PubMed and Google Scholar as these sources represent a wide scope of peer-reviewed articles in the field of nanophotonics, plasmonics, materials science, physics and biosensor engineering [Page *et al.*, 2021, Unser *et al.*, 2015]. The search terms were constructed to focus on the combinations of the following words localized surface plasmon resonance, LSPR biosensor, gold nanoparticles, silver nanoparticles, plasmonic nanostructures, nanorods, nanostars, nanotriangles, core-shell nanoparticles, refractive index sensitivity, and biosensing applications. Classical research was preserved where it was deemed essential to gain insight into the physical mechanism of plasmonic resonance, the influence of size and shape of nanoparticles, and the significance of dielectric environment in spectral response determination [Kelly *et al.*, 2003, Mayer *et al.*, 2011]. The core analysis was restricted to peer-reviewed journal articles with a direct approach to the subject of Au-, Ag-, or Au-Ag-based LSPR-based biosensing platforms. The priority was on original research papers that have reported the measurable sensing results including resonance wavelength shift, sensitivity to refractive index, linewidth, figure of merit, limit of detection, surface functionalisation strategy or direct biosensing performance [Anker *et al.*, 2008, Loiseau *et al.*, 2019]. Articles that contained purely propagation of surface plasmon resonance of continuous thin films were also not considered part of the core set of comparisons unless they had conceptual interest in nanoscale localised plasmonic sensing. In the same manner, the studies that do not provide quantitative optical or biosensing data were not included in the ultimate pool of analysis. This sieving was significant since plasmonics is among those areas where beautiful pictures and virtuous words occasionally precede sensory data that can be reproduced. The current research hence concentrated on reports enabling to make a meaningful comparison between optical performance and biosensing utility [Mayer *et al.*, 2011, Unser *et al.*, 2015].

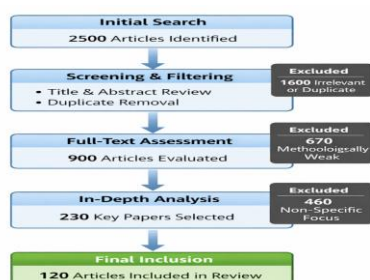


Fig.1. Schematic showing the literature selection pathway used in the present critical study.

Classification of nanostructured plasmonic devices- Screening followed with the identification of selected studies which were sorted into three general material groups of gold nanostructures, silver nanostructures, and hybrid or bimetallic Au-Ag systems. In each category further classification was done on the basis of morphology that included nanospheres, nanorods, nanotriangles, nanorings, nanostars, nanobipyramids, and core-shell structures. The need to classify them was due to the clear previous studies that optical response of plasmonic nanoparticles is highly dependent on size, aspect ratio, shape anisotropy, sharpness of edges, interparticle coupling and local dielectric environment [Kelly *et al.*, 2003, Larsson *et al.*, 2002]. They have been actively employed as nanostructures of gold due to their chemical stability, biocompatibility and established surface chemistry of immobilisation of biomolecules, and also silver nanostructures due to their reduced inherent optical losses at visible frequencies. Simultaneously, silver is more susceptible to oxidation and deterioration in the environment, which is why direct comparison between Au and Ag systems in particular is particularly significant in biosensing studies [Unser *et al.*, 2015, Loiseau *et al.*, 2019]. The chosen articles were further categorised based on mode of sensing and its intended use, i.e. label-free refractive-index sensing, DNA hybridisation detection, protein-recognition, toxin sensing, and bioaffinity analysis. This was a step towards differentiating between the studies which showed only the optical sensitivity in model solvents and the ones that had advanced to the real biological detection. Previous experiments by Haes and Van Duyne demonstrated that triangular silver nanoparticles are sensitive to local dielectric variation, and can be utilized as nanoscale affinity biosensors, and Larsson *et al.* have demonstrated that gold nanorings have shape-dependent sensitivity and are appropriate in ultrasensitive biosensing [Haes and Van 2002, Larsson *et al.*, 2002]. The studies in particular are particularly good benchmarks as they relate the geometry of nanoparticles directly to sensing behaviour as opposed to considering the shape of nanostructures as the nanotechnology wallpaper wall [Haes and Van 2002, Larsson *et al.*, 2002].

Parameters used for critical evaluation-The primary parameters of analysis that were obtained in each of the chosen studies were the LSPR peak position, spectral shift, sensitivity of the bulk refractive-index component in nm/RIU, full width at half maximum (FWHM), figure of merit (FOM), limit of detection (LOD), surface functionalisation chemistry, and reported stability of operation [Anker *et al.*, 2008, Unser *et al.*, 2015]. Additional data on repeatability, regeneration, anti-fouling behaviour, compatibility with aqueous or biological media as well as long-term signal reliability was collected where available. This was required since in optimal optical conditions an LSPR device can exhibit excellent peak shifting behaviour, but in real biosensing conditions the nanostructure is unstable, chemically reactive or hard to functionalise thus still performing poorly [Mayer *et al.*, 2011, Unser *et al.*, 2015, Loiseau *et al.*, 2019]. Sensitivity in the current paper was determined as $S = 8G/1/1$ (01 8), where 8G2 is the resonance wavelength change and 8G1 8 is the local refractive index change, and figure of merit was viewed as the ratio of sensitivity to spectral line width [Mayer *et al.*, 2011]. It is also through the comparative analysis that the broader aspects of physical and practical consequences such as the effect of nanoparticle morphology on local field confinement, the trade-off between optical sharpness and chemical robustness, bimetallic design, and the combination of favourable properties of both Au and Ag were also studied [Kelly *et al.*, 2003, Loiseau *et al.*, 2019]. Gold typically has better flexibility of bioconjugation and environmental stability but silver typically has greater plasmonic enhancement and reduced spectral characteristics. Hybrid systems, such as Au-Ag, core-shell and others, were thus viewed as intermediate structures to maintain high sensitivity and enhance stability and biosensing feasibility [Unser *et al.*, 2015, Loiseau *et al.*, 2019]. The arrangement of the analysis so will bring the section beyond mere description and establish a framework on which materials, device geometries, and real sensing usefulness can be critically compared. Small nanostructures can be

used to produce large electric fields, yet a biosensor has to endure chemistry, noise, fouling and the actual world.

Results and discussion

Comparative analysis of the chosen articles reveals that nanostructure geometry, localisation of the field, and spectral linewidth of plasmonic nanostructured devices are the primary factors that control the sensing behaviour of nanostructured metal as opposed to spectral linewidth selection. The reported sensitivity of refractive indices is 44-703 nmRIU across 7 shapes of gold nanoparticles with a clear progression of simple spheres to anisotropic and branched forms [Chen *et al.*, 2008]. Gold nanorings were some of the best examples with reported bulk refractive-index sensitivities of up to 880 nm/RIU, several times larger than that of larger nanodisks of the same size [Larsson *et al.*, 2007]. These results can be well used to argue that ultrasensitive LSPR biosensing can be realized when the electromagnetic field is confined to small areas by plasmonic structures like edges, voids, tips, and ring interiors. To be a bit more direct, the more acute and alien the particle, the more viciously it senses its environment. Another group of nanorods, which are anisotropic structures made out of gold nanorods, is also highly sensitive and usable in practical biosensing formats. Chip-based gold nanorod sensor A chip-based gold nanorod sensor with label-free gold nanorod detection in serum has a wavelength sensitivity of 252 nm/RIU [Marinakos *et al.*, 2007], although geometry-selection experiments on gold nanorods later showed that with careful aspect-ratio optimisation bulk sensitivities could be optimised to as high as 600 nm/RIU [Nusz *et al.*, 2009]. Nanotriangles that were made of gold also performed well in nucleic-acid biosensing with a reported limit of 468 nm/RIU, LSPR shifts up to 35 nm on complementary 50-mer targets, and shifts over 20 nm on clinical PCR products of approximately 350 bp, with non-complementary targets falling within a range of about +1 nm [Soares *et al.*, 2014]. Gold nanostars went a step further as its ability of single particle label-free biosensing allowed streptavidin binding to be detected by changes in the tip- and core-tip plasmon resonances [Dondapati *et al.*, 2010]. This was also demonstrated by five-branched gold nanostars that were embedded on plastic optic fibre platforms that demonstrated how branched nanostructures can be scaled to miniature sensor devices [Cennamo *et al.*, 2013]. These findings, combined, demonstrate that gold is the most flexible biosensing platform in cases when high sensitivity has to be achieved in conjunction with bioconjugation chemistry and environmental stability.

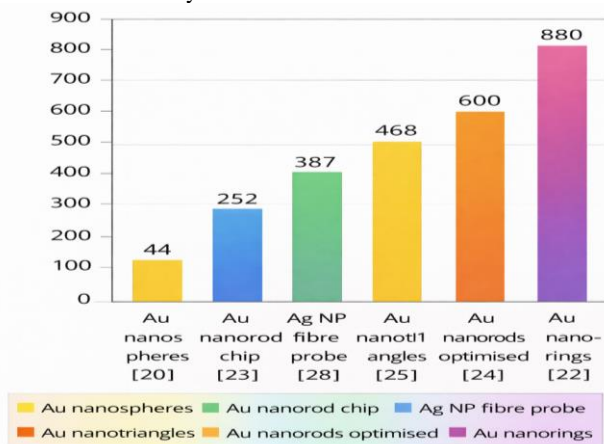


Fig. 2. Reported refractive-index sensitivities of representative LSPR nanostructures discussed in the present critical study. Plasmonic devices based on silver, on the other hand, still have a real optical edge. Triangular silver nanoparticles were one of the first LSPR biosensing studies in which it was determined that local refractive-index variations due to the molecular recognition could be translated into spectral shifts that were readable [Haes and Van 2002]. Subsequent studies identified that the theoretical sensitivity to refractive index of 5-50 nm diameter spherical particle as Ag nanoparticles increases 153 to 265 nm/RIU, versus 128 to 233 nm/RIU in the case of Au nanoparticles of comparable size [Loiseau *et al.*, 2019]. A reflective optical fibre biosensor using silver

nanoparticles has been demonstrated to perform well in the form of an experimental device with a refractive-index sensitivity of 387 nm/RIU and resolution of 9.04×10^{-4} RIU with reproducibility, stability and clear spectral changes with biomolecular modification [Chen *et al.*, 2015]. It not only validates the conventional plasmonics trade-off that silver tends to provide the sharper and stronger optical response, but only when it's chemical instability is placed on a leash. The primary cause of reason why silver has not just come in and taken over the entire kingdom of biosensing is oxidation, tarnishing of the surface and long-term drift.

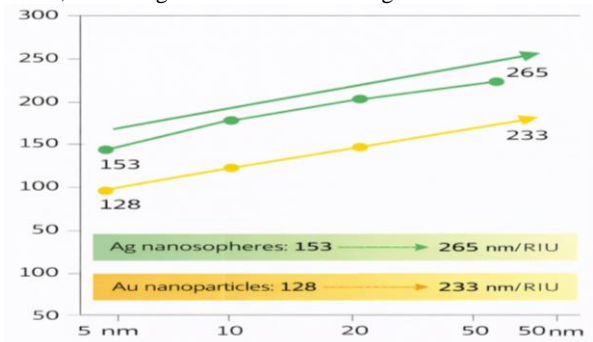


Fig. 3. Theoretical refractive-index sensitivity trend for spherical Ag and Au nanoparticles in the 5-50 nm size range.

Fig. 3 explains why silver remains an area of focus as one of the ultra-sensitive sensing materials: even before one introduces sharp corners, dimers, or core-shell fettsish, even bare silver tends to show a more compelling refractive-index response than gold in similar spherical geometries [Loiseau *et al.*, 2019].

Hybrid and bimetallic systems have become a reasonable compromise due to the imperfection of both metals. Specifically, naked-eye toxin biosensing using Au-Ag core-shell nanoparticles has been realized by exploiting the high response of silver to LSPR and the improved biomolecular interface chemistry offered by gold [Loiseau *et al.*, 2019]. An associated Au nanoparticle LSPR immunosensor to staphylococcal enterotoxin A (SEA) had an estimated limit of detection of 5 ng/mL, and retained its analytical capability with one year of storage at 4 C which is an unusually useful reminder that stability is no minor consideration but a fundamental analytical variable [Ben *et al.*, 2017]. These researches indicate that not the biosensor with the largest attention-grabbing single sensitivity figure, but the biosensor with capability to maintain plasmonic performance at the same time, resistance to storage, surface modification, and actual sample matrices like milk or serum is the best. There is where most glamorous nanoparticles come in contact with the ugly paperwork of reality.

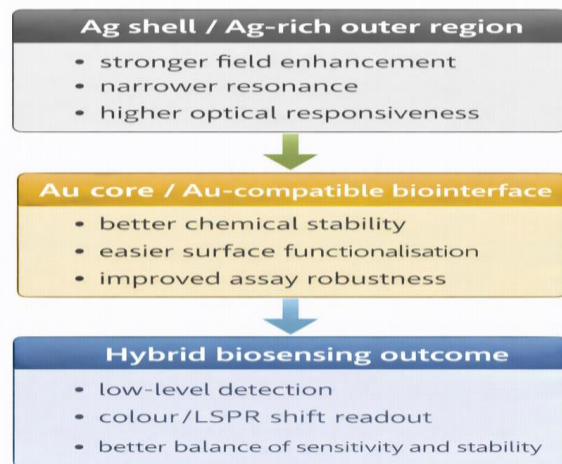


Fig. 4. Schematic showing the performance logic of hybrid Au-Ag plasmonic biosensors.

The general trend that is followed of the literature reviewed is that there is no universal winner of gold versus silver as LSPR biosensing. Applications In general, gold tends to be preferable to the use of other sensing platforms in situations where the sensing platform needs to endure intricate media, repeated

biofunctionalisation, and long-term storage. Silver is much more desirable when the ideal is maximizing the field, more sharp resonances or high signal-noise behaviour. Hybrid Au-Ag systems are particularly promising as they can partially overcome this gap, yet, they remain highly dependent on the shell thickness, uniformity of the interfaces, oxidation resistance and reproducibility of fabrication [Chen *et al.*, 2015, Ben *et al.*, 2017]. The fact that LSPR can be sensitive is no longer the problem to which the field is the major challenge, then, in a critical perspective. That part is already done. The more difficult is the creation of nano structured plasmonic devices that are sensitive, selective, stable, reproducible and manufacturable simultaneously.

Conclusions

This is a critical study that analysed nanostructured gold and silver plasmon devices that use ultra-sensitive localised surface plasmon resonance-based biosensing. It has been revealed in the review that LSPR biosensing performance is not dictated solely by the type of noble metal used, but rather the collective influence of nanostructure geometry, spectral linewidth, surface chemistry, local field confinement and device stability. In the studied literature, the anisotropic and sharp-featured nanostructures, namely, nanorods, nanotriangles, nanorings, nanostars, and their hybrid analogs, always performed better than a simple nanosphere due to this property: these nanostructures formed a stronger electromagnetic hotspots and higher resonance shifts with a local refractive-index change. The comparative analysis also shows that gold and silver have different complementary benefits. Gold-based nanostructures have continued to be of interest to biosensing due to their high chemical stability, good biocompatibility as well as strong surface functionalisation to immobilise biomolecules into surfaces. Silver-based structures, however, tend to have sharper plasmonic resonances, enhanced field enhancements as well as optical responsiveness and have been particularly attractive as ultra-sensitive detectors. Nonetheless, the fact that silver is more prone to oxidation and environmental degradation remains a major limitation in the direct application of silver in robust sensing environments. This is why the approach of hybrid and core-shell Au-Ag systems seems to have a very promising perspective, as they can be used to combine the powerful plasmonic activity of silver and advantage of stability and interfaces of gold. One of the key findings of this research is that the future of LSPR biosensing is not solely based on whether or not better values of sensitivity can be attained on paper. A more challenging aspect is to be able to produce devices that are sensitive, selective, reproducible, stable, and manufacturable under real biological conditions. Numerous plasmonic designs are also doing well in controlled optical experiments, but only a much smaller number have been shown to be stable on long-term platforms, resistant to contaminants, compatible with complex environments, and can be easily integrated into miniature-scale sensing systems. Hence, the field is currently transitioning to the proof-of-concept elegance to engineering maturity. Comprehensively, nanostructured gold and silver plasmonic devices have proven themselves to be of great importance as label-free biosensing. The fact that they have the potential to transduce nanoscale biochemical interactions to detectable optical signals provides them with good potential applications in medical diagnostics, environmental analysis, food safety analyses, and point-of-care assays. The reviewed evidence in this paper indicates that the future of LSPR is likely to brighten up on engineered geometries, hybrid material systems, enhanced surface chemistry, and reproducible fabrication strategies that can push LSPR out of the laboratory excellence to real-life, reliable biosensing applications.

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