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Research Article

Solvent effects on the localized surface plasmon resonance of Ag nanoparticles

Minh-Kha Nguyen[1,2,*], Quoc-An Phan[1,2], Thi-Thanh-Thuy Tran[1,2], Khanh-Binh Vo[1,2]

ABSTRACT

Ag, Au, and Cu are common metals which exhibit strong optical absorption in the visible region. Notably, Ag has demonstrated the most effective light-driven chemical reactions. The characteristics of the solution play a direct role in influencing the localized surface plasmon resonance (LSPR) and stability of Ag nanoparticles (AgNPs) in various applications, thereby regulating the efficiency of the reaction. Nevertheless, to date, there has been a lack of systematic research on the impacts of solvents on the surface plasmon resonance of the AgNPs. Herein, AgNPs were synthesized using sodium citrate as a reducing agent, and the effect of the solvent on the excitation energy at the surface interface to the LSPR was investigated. The determination was made that the surface plasmon band of AgNPs is significantly impacted by the characteristics of the solvent including pH, polarity, and the lengths of alcohol chains. The influence of ethanol was most pronounced with alcohol, while in polar solvents, the level of polarity in the solvent correlated with the magnitude of the LSPR effect on AgNPs. The effects of solvents on the LSPR of AgNPs also determine that water is a suitable solvent for storage. The results are valuable information for improving the activity and sensitivity of the LSPR in plasmonic applications.

Key words: Surface plasmon, Ag nanoparticle, Stability, Optical sensing, Solvent effect

[1]*Faculty of Chemical Engineering, Ho Chi Minh City University of Technology (HCMUT), 268 Ly Thuong Kiet Street, Dien Hong Ward, Ho Chi Minh City, Vietnam*

[2]*Vietnam National University Ho Chi Minh City, Linh Xuan Ward, Ho Chi Minh City, Vietnam*

Correspondence

Minh-Kha Nguyen, Faculty of Chemical Engineering, Ho Chi Minh City University of Technology (HCMUT), 268 Ly Thuong Kiet Street, Dien Hong Ward, Ho Chi Minh City, Vietnam

Vietnam National University Ho Chi Minh City, Linh Xuan Ward, Ho Chi Minh City, Vietnam

Email: nmkha@hcmut.edu.vn

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INTRODUCTION

The localized surface plasmon resonance (LSPR) plays a significant role in various applications in science and life, e.g., photocatalysts, biosensors, solar cells, anticancer therapeutics, and nanophotonic devices[1–3]. Only three noble metals (Au, Ag, and Cu) could provide large enhancement in the visible region of the electromagnetic spectrum. The extent of plasmonic enhancement in LSPR is often related to nanoparticle (NP) composition, size, and morphology[4]. Consequently, a wide array of NP shapes has been investigated, from spheres to more complex shapes like cubes and octahedrons, as well as nanorods and nanowires of varying lengths[5–7]. Moreover, due to a strong dependence of the electric field on the metal substrate, a plethora of metals providing different plasmon resonant frequencies and enhancement factors have been investigated. Au and Ag have been the most popular plasmonic metals, due to their surface plasmon resonance (SPR) bands located in the visible region, with Ag providing the highest enhancement due to its superior permittivity and quality factor[8,9]. Furthermore, Ag nanostructures exhibit a more intense and well-defined SPR across a wider range from ultraviolet (UV) to infrared (IR) regions compared to Au[9].

It has long been recognized that the devices from Ag are more sensitive than those from Au[10]. However, poor chemical and structural stability has been a serious issue, limiting the further practical applications of Ag nanostructures. Moreover, in the liquid phase, solvents also can alter the interfacial bonds between nanoplasmonic metal particles and molecules in solution, even participating directly in reactions[11]. The presence of solvent can cause light scattering, create different refractive indices, and control the metal particle surface charge density. The plasmon resonance energy of nanoparticles can also be dissipated into the solvent through electron-phonon coupling, but its kinetics are usually much slower than electron-electron scattering and charge recombination[12]. In addition, solvents such as methanol and solvent-soluble substances such as OH^- or O_2 can also interfere with charge transfer at the nanoplasmonic particle surface[13]. Meanwhile, most applications of AgNPs for photocatalysis or biosensing are performed in the liquid phase. These solvents have a direct influence on the LSPR properties of AgNPs and impact the effectiveness of the application. However, currently, many studies have not focused on the influence of solvents on the LSPR of AgNPs. The effect of the solvent on the excitation energy at the surface interface and the charge transfer between the plasmonic metal and the

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molecules in the solution still needs to be further elucidated. A suitable solvent for application fields related to AgNPs is crucial, but there is not much research on this topic.

In this study, AgNPs were produced through a chemical reduction technique utilizing sodium citrate as a reducing agent. An analysis was conducted on the responsive LSPR behavior of the synthesized AgNPs in different solvents under controlled pH levels, polarity variations, and storage durations. The investigation revealed a trend where the LSPR of AgNPs experiences a blueshift towards shorter wavelengths in solutions with higher pH values or greater solvent polarity, while the absorption spectrum undergoes a redshift towards longer wavelengths with increasing alcohol chain length. In terms of the endurance of LSPR characteristics in solvents under identical storage conditions, it was observed that water offers superior stability for AgNPs compared to alternative solvents such as propanol and ethanol. These findings not only enhance the comprehension of the excitation energy dynamics between AgNPs and solvents but also introduce a novel strategy for enhancing and optimizing the functionality of LSPR-based materials to attain elevated efficacy and specificity in applications such as biosensing and photocatalysis.

MATERIALS AND METHODS

Materials

Silver nitrate ($AgNO_3$, ≥99.9%), sodium citrate dihydrate ($Na_3C_6H_5O_7{\cdot}2H_2O$, 99%), ethanol ($C_2H_5OH$, 99.5%), 1-propanol ($C_3H_7OH$, 99%), 1-butanol ($C_4H_9OH$, 99%), and acetonitrile (C_2H_3N, 99%) were purchased from Thermo Scientific. Double-distilled water was used for all related experiments.

Methods

AgNPs were prepared via the reduction of $AgNO_3$ using sodium citrate as a reducing agent. Typically, 40 mL of $Na_3C_6H_5O_7$ solution with varying concentrations from 2 to 10 mM was heated to boiling temperature (~105 ^oC) under magnetic stirring and reflux. After the temperature reached constant for 5 min, 4 mL $AgNO_3$ solution (5 mM) was injected. The solution changed quickly from white to yellow. After 60 min, the growth was quenched by cooling the reaction flask to room temperature. The AgNPs were collected by centrifugation at 6000 rpm for 15 min and then washed with 30 mL of water three times before being redispersed in 10 mL of water.

To investigate the influence of solvents on the LSPR, the synthesized AgNPs were centrifuged and redispersed in a testing solvent such as water, ethanol, 1-propanol, 1-butanol, and acetonitrile for 15 min.

Characterization

The surface plasmon resonance of each AgNPs solution was analyzed by the V-730 Ultraviolet-visible spectroscopy (UV-Vis) from JASCO, in the range from 300 nm to 800 nm. The morphology of AgNPs was identified by TEM (transmission electron microscopy, JEM-2100), operating at 80 kV.

RESULTS

Synthesis of AgNPs

Effect of $Na_3C_6H_5O_7$ concentration

The UV-Vis absorption spectra of five AgNPs samples, which were synthesized under varying concentrations of $Na_3C_6H_5O_7$, exhibit a sharp peak at a wavelength of 416 nm (**Figure 1**). The absorbance increased as the $Na_3C_6H_5O_7$ concentration increased from 2 to 6 mM and then decreased at 8 and 10 mM concentrations. Increasing the concentration of the reducing agent from 2 to 10 mM also led to a broader spectrum. It is known that the absorption spectrum of AgNPs is strongly influenced by particle shape and size[14]. As the reducing agent concentration increases, large-sized nanoparticles predominate[15]. Large AgNPs scatter more strongly than small particles. Furthermore, as the sodium citrate concentration increases, the formation of AgNPs occurs more slowly[16]. With the same concentration of AgNPs, a higher absorbance illustrates more effective LSPR for applications. As a result, 6 mM $Na_3C_6H_5O_7$ solution was chosen as the reducing agent to investigate the effect of reaction time.

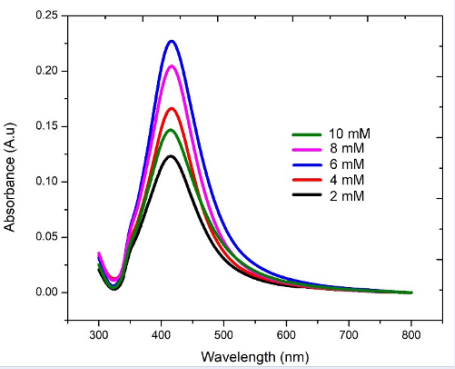

Figure 1: Absorption spectra of AgNPs synthesized under varying concentrations of $Na_3C_6H_5O_7$ solution. [Source: Authors]

Effect of reaction time

The intensity-based LSPR of AgNPs increased with the increasing reaction time from 30 to 60 min and

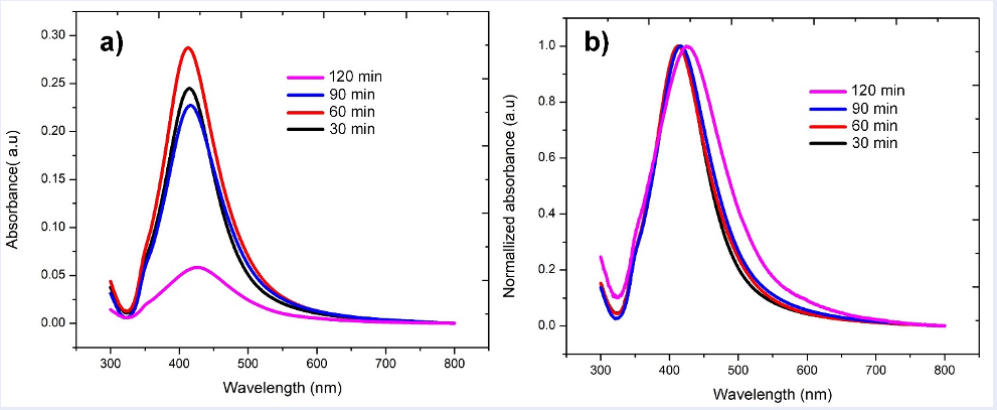

Figure 2: Absorption spectra of AgNPs with different reaction times a) Actual absorbance, b) Normalized absorbance. [Source: Authors]

decreased when the reaction time increased to 90 and 120 min (**Figure 2**). During the period of 30 to 60 min, there was a progressive increase in the formation of nanoparticles, leading to a corresponding rise in absorption. As the duration of the reaction progresses, the nanoparticles grow to generate larger particles. As a result, the LSPR spectroscopy is wider with a longer reaction time (**Figure 2b**). Particularly, the position of the LSPR peak has shifted to a larger wavelength with a reaction time of 120 min. 60 min was chosen as the valuable time for the formation of AgNPs.

Reproducibility of synthesis protocol

Five samples of AgNPs were prepared with a reducing agent concentration of 6 mM $Na_3C_6H_5O_7$ and 60 min in reaction time. **Figure 3** shows that the LSPR properties of AgNPs between the five synthesized samples are consistent in both actual absorbance and normalized absorbance. The normalized LSPR curves are the same and the wavelength of maximum absorbance of AgNPs from five samples is 416 nm. The result indicates that the protocol of synthesized AgNPs is well reproducible.

The TEM images of AgNPs, which were prepared with a reducing agent concentration of 6 mM and reaction time of 60 min, are shown in **Figure 4**. It clearly shows that more than 97% of AgNPs are spheres with an average diameter of ~35 nm.

The effect of solvents on the LSPR of AgNPs

Influence of pH of aqueous solution

When the pH is below 3, the AgNPs solution transitions from a yellow to dark green, signifying aggregation of the particles. The absorption spectra of AgNPs in the solution also show two broad and low-intensity peaks at the wavelength of ~418 nm and ~690 nm (**Figure 5a**). AgNPs synthesized utilizing citrate as a reducing agent exhibit notable stability and homogeneous distribution within a solvent. However, citrate becomes fully protonated at low pH levels, leading to a significant reduction in surface negative charges. The decline in negative charges on the surface prompts aggregation, consequently diminishing the stability of AgNPs. Conversely, at a pH higher than the pK_{a1} values of citric acid (pK_{a1} ~3), the aqueous nanoparticle solutions sustain the yellow color, indicating the absence of aggregation. This phenomenon is attributed to the complete deprotonation of citrate, resulting in an abundance of negative surface charges that foster repulsion among adjacent AgNPs, thereby inhibiting aggregation. As a result, when pH is higher than 3, the LSPR peak of AgNPs shifts to a shorter wavelength in the range of 412 to 415 nm. The pH of the solution has a great influence on the surface plasmon resonance of the AgNPs. Therefore, a neutral pH was chosen for all experiments concerning the aqueous solution of AgNPs.

Influence of polarity of solvents

It is known that solvents can modify the bonds between nanoplasmonic metal particles and molecules, affecting reactions and properties such as light scattering, refractive indices, and surface charge density[17]. To test the dissipation of plasmon resonance energy into the solvent, the impact of the polarity of alcohol based on chain length on the LSPR spectrum of AgNPs was examined. Various alcohols with differing

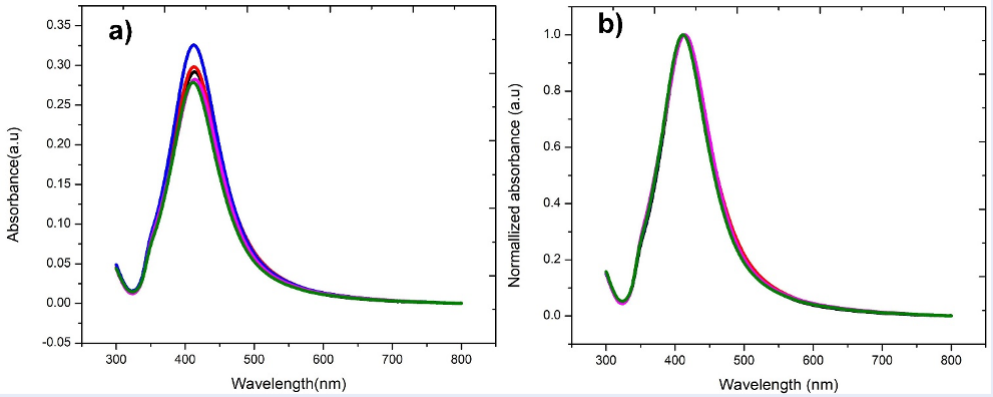

Figure 3: Absorbance spectra of five AgNPs samples synthesized under the same conditions. a) Actual absorbance, b) Normalized absorbance. [Source: Authors]

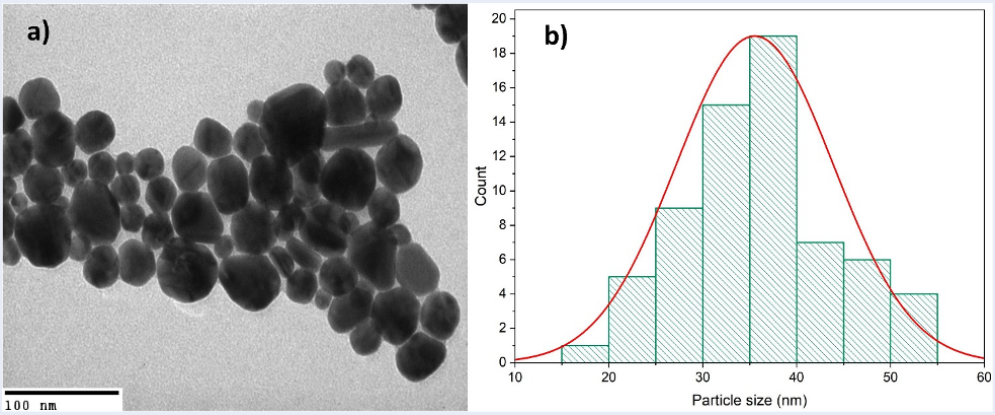

Figure 4: a) TEM image and b) particle size distribution of AgNPs. [Source: Authors]

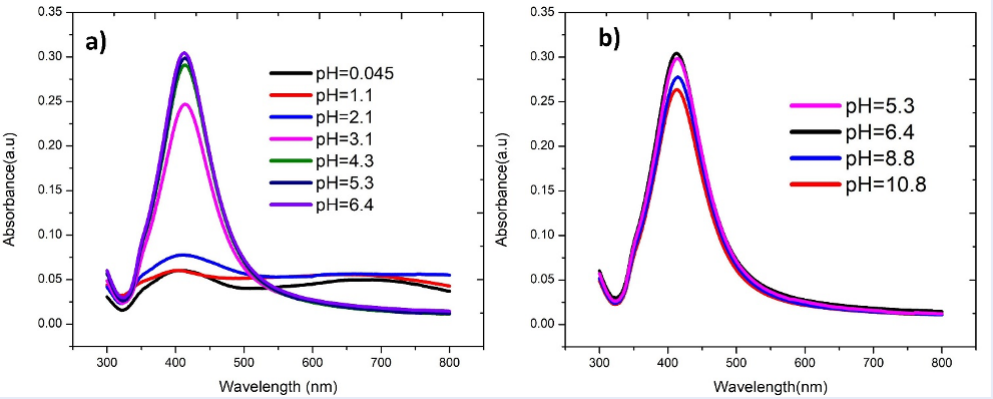

Figure 5: The LSPR of AgNPs in different pH environments: a) acidic environment and b) basic environment. [Source: Authors]

chain lengths denoted as $CH_3(CH_2)_xOH$ with x ranging from 1 to 3, were utilized. The mixture between AgNPs and each alcohol was well dispersed and exhibited pH levels between 6.0 and 6.2. It was observed that the chain length of the alcohol significantly affects the LSPR of AgNPs. **Figure 6a** shows that the peak of plasmon absorption in ethanol is detected at 417 nm and shifts towards the red region at ~420 nm and a broad shoulder at 650 nm in the propanol. Meanwhile, the alcohol chain length increases to x = 3, i.e., butanol, the LSPR properties of AgNPs tend to be almost straight line. The absorbance of AgNPs in ethanol is the highest intensity compared to the other two solvents. As the length of the carbon chain increases, the ability to donate electrons from the oxygen atom (–OH) in the alcohol molecule to AgNPs decreases [18]. A longer chain length exhibits a lower diffusion ability of the –OH group. When the alcohol's electron donation is less, it leads to a decrease in electron density on the nanoparticle surface, and at the same time, the surface plasmon resonance effect of AgNPs decreases. It means that the absorption maxima of AgNPs in these solvents follow the polarity of the alcohols increases from butanol to ethanol.

Besides that, when increasing the polarity of the solvent, the LSPR spectrum shifts to a shorter wavelength region (**Figure 6b**). As the polarity of the solvent increases, the absorption intensity of AgNPs also increases. Polar solvents directly interact with the surface of AgNPs through interaction and electron transfer to the surface of AgNPs, hence, changing electrons on the surface result in the variation of the λ_{max} of the LSPR [19]. The change in the peak of LSPR as a function of refractive index signifies a clear interaction with AgNPs owing to their polar characteristics [20]. This interaction is further reinforced by a noncovalent bond formed between solvent and citrate ions, resulting in a consistent blueshift observed when using shorter alcohol chains or more polar solvents.

Influence of storage time on LSPR of AgNPs in solvents

To investigate the stability of LSPR of AgNPs during storage time, the synthesized AgNPs were dispersed in various solvents such as water, ethanol, and propanol for 2 months. The mixture between AgNPs and each solvent was well dispersed and exhibited pH levels between 6.0 and 6.4. After two weeks, the LSPR of the material was checked. **Figure 7** shows that the decrease in absorbance of AgNPs in water is the slowest, followed by samples in ethanol and propanol. Furthermore, after a period of storage in solvents, the LSPR of AgNPs shifted to a larger wavelength range. The interaction between the citrate layer on AgNPs with solvent molecules is unstable in a low-polarity solvent. As a result, the Ostwald ripening process forms larger nanoparticles with a broad shoulder range in ethanol and propanol. The decrease in the absorption peak of AgNPs is might due to the amount of dissolved oxygen in the solvents. AgNPs are relatively sensitive, therefore in an environment with dissolved oxygen, AgNPs oxidize to form Ag_2O, which reduces absorption [21]. Under the same temperature and pressure conditions, oxygen solubility decreases as the solvent's polarity increases. On the other hand, the –OH group in water transfers electrons to the AgNPs surface via the interchain H-transfer stronger than that of ethanol and propanol due to its polarity [22,23]. With a higher electron density, the surface plasmon resonance of AgNPs in water is higher intensity and more stable than the other solvents. Hence, water is a valuable solvent for the storage of AgNPs.

DISCUSSION

The reduction of Ag^+ by sodium citrate is:

$4Ag + C_6H_5O_7Na_3 + 2H_2O \rightarrow$

$4Ag + C_6H_5O_7H_3 + 3Na^+ + H^+ + O_2$

40 mL of 2 mM citrate solution reduces enough to 4 mL of 5 mM $AgNO_3$. However, in reality, a higher concentration of citrate is needed to increase the stability of nanoparticles. The citrate compound serves as a reducing agent and a capping agent, effectively hindering the aggregation of particles stemming from the presence of negative surface charges. The electrostatic forces of repulsion between adjacent nanoparticles, induced by the negative surface charge of the citrate layer, contribute to their sustained dispersion in the solution. The pH solution significantly impacts the charge of the citrate layer, which greatly influences the surface plasmon resonance of AgNPs.

In the case of alcohols, the change in the LSPR wavelength with chain lengths suggests direct interaction with AgNPs due to the polar nature of alcohols. While AgNPs are stabilized by noncovalent bonds with a citrate layer, the barrier to prevent polar alcohol molecules from penetrating the surface is not strong enough, thus affecting the LSPR of AgNPs. The redshift observed with longer alcohol chains highlights their specific interaction with AgNPs and indicates a decrease in electron density donation to AgNPs as chain length increases. It highlights the importance of chemical interactions between AgNPs and solvents, leading to the consistency between LSPR variations and alcohol refractive indices.

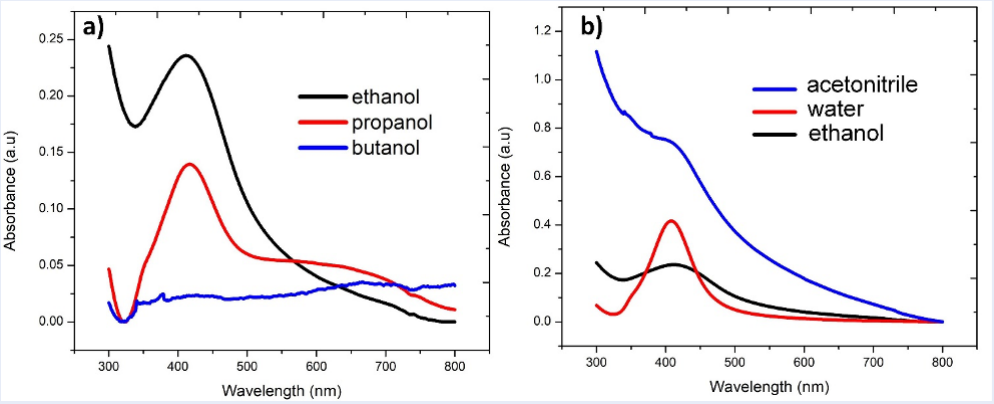

Figure 6: The LSPR of AgNPs in solvents: a) with different alcoholchain lengths and b) with different polarizations. [Source: Authors]

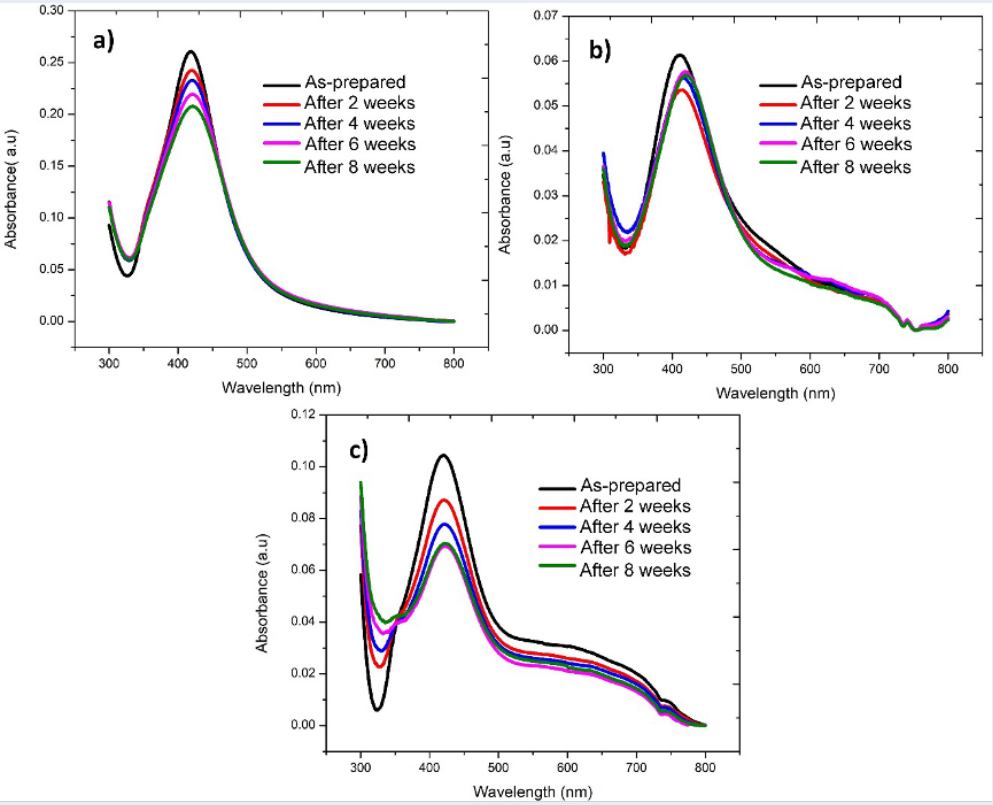

Figure 7: The LSPR of AgNPs in solvents over time: a) in water, b) in ethanol, and c) in propanol. [Source: Authors]

Table 1: The influence of solvent on LSPR of AgNPs [Source: Authors]

		Dipole moment (D)	Refractive index (n)	λmax (nm)
Polar solvents	Acetonitrile	3.92	1.3441	410
	Water	1.84	1.3330	416
	Ethanol	1.69	1.3614	417
	Propanol	1.68	1.3829	418
	Butanol	1.66	1.3856	N.A.
Non-polar solvent	Toluene	0.31	1.4486	N.A.
	Cyclohexane	0.00	1.4262	N.A.
	Carbon tetrachloride	0.00	1.4600	N.A.

Note: N.A. – Notavailable

To evaluate the sensitivity of LSPR of AgNPs in different environments, a group of polar solvents and non-polar solvents was selected for testing (as shown in **Table 1**). Non-polar solvents have a layer separation phenomenon with the AgNPs solution, so the maximum absorption wavelength is not observed. The maximum wavelength of the LSPR of AgNPs in these solvents also shows a gradual redshift as the solvent refractive index increases.

Besides, compared with other coating agents such as polyethylene glycol and polyvinylpyrrolidone, the stability of citrate is much poorer [8,18]. Therefore, the AgNPs in solvents with a low polarity index were not well protected, and this led to a decrease in absorbance and a shift to a larger wavelength region. Polar solvents can form complexes with Ag surfaces through charge-transfer interactions, with varying electron injection capabilities. The coordinatively unsaturated Ag atoms at the particle surface can attract electrons from solvent molecules, leading to a notable shift in plasmon band position as per Mie theory. The differences in available electrons from various solvent molecules affect the LSPR of AgNPs in polar solvent systems, stabilizing the charged particles and preventing aggregation due to repulsive forces mediated by the solvent molecules.

CONCLUSION

AgNPs prepared and stabilized by citrate exhibit were clearly defined surface plasmon resonance and the formation of nanoparticles was monitored by tracking absorption profile adjustments during reduction. This straightforward synthetic method yields metal particles that disperse excellently in polar solvents. Two distinct mechanisms by which solvents affect the LSPR of AgNP are refractive index changes at the nanoparticle/bulk interface and complex formation with the nanoparticle surface. The LSPR of the metal colloids will be directly impacted by these complexation processes, which will cancel out the effects of the refractive index. The specificity of the surface complexation process with metal particles is indicated by the subsequent redshift that occurs with an increase of one carbon atom in the alcohol chain. Regarding durability under storage conditions in solvents, water is a valuable solvent to improve the LSPR of AgNPs. The results will enable the chemical synthesis of metallic nanoclusters with controlled size and shape in various solvents, which may have a broad range of uses in molecular microelectronics, chemical-biological sensors, and other applications requiring highly controllable optical properties.

COMPETING INTERESTS

The authors declare that there are no conflicts of interest regarding the publication of this paper.

ACKNOWLEDGEMENTS

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AUTHOR CONTRIBUTIONS

Minh-Kha Nguyen contributed to conceptualization, methodology, data analysis, manuscript writing, and editing. Quoc-An Phan performed the experiments and conducted data analysis. Thi-Thanh-Thuy Tran contributed to methodology and validation. Khanh-Binh Vo provided support for manuscript writing. All authors reviewed and approved the final version of the manuscript.

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Bài nghiên cứu

Ảnh hưởng của dung môi đến hiệu ứng cộng hưởng plasmon của hạt nano bạc

Nguyễn Minh Kha[1,2,*], Phan Quốc An[1,2], Trần Thị Thanh Thuý[1,2], Võ Khánh Bình[1,2]

TÓM TẮT

Ag, Au và Cu là những kim loại phổ biến có khả năng hấp thụ quang mạnh trong vùng ánh sáng khả kiến. Trong đó, Ag nổi bật với hiệu quả cao nhất trong các phản ứng hóa học được kích thích bởi ánh sáng. Trong nhiều ứng dụng, đặc tính của dung môi có ảnh hưởng trực tiếp đến hiệu ứng cộng hưởng plasmon bề mặt (LSPR) cũng như độ ổn định của các hạt nano bạc (AgNPs), từ đó ảnh hưởng đến hiệu suất phản ứng. Tuy nhiên, đến nay vẫn còn thiếu các nghiên cứu mang tính hệ thống về ảnh hưởng của dung môi đối với cộng hưởng plasmon bề mặt của AgNPs. Trong nghiên cứu này, AgNPs được tổng hợp dùng natri citrate làm chất khử, đồng thời khảo sát ảnh hưởng của dung môi đến tính chất LSPR. Kết quả cho thấy dải plasmon bề mặt của AgNPs chịu tác động rõ rệt bởi các đặc tính của dung môi như pH, độ phân cực và chiều dài mạch ancol. Trong nhóm ancol, ethanol thể hiện ảnh hưởng mạnh nhất; còn đối với các dung môi phân cực, độ phân cực càng cao thì mức độ ảnh hưởng đến LSPR của AgNPs càng lớn. Ngoài ra, các kết quả cũng chỉ ra rằng nước là dung môi phù hợp cho việc lưu trữ AgNPs. Những phát hiện này cung cấp cơ sở quan trọng để nâng cao hoạt tính và độ nhạy của LSPR trong các ứng dụng plasmon.

Từ khoá: plasmon bề mặt, nano bạc, độ ổn định, cảm biến quang học, ảnh hưởng của dung môi

[1]*Khoa Kỹ thuật Hóa học, Trường Đại học Bách khoa Tp. HCM, 268 Lý Thường Kiệt, Phường Diên Hồng, Thành phố Hồ Chí Minh, Việt Nam*

[2]*Đại học Quốc gia Thành phố Hồ Chí Minh, Phường Linh Xuân, Thành phố Hồ Chí Minh, Việt Nam*

Liên hệ

Nguyễn Minh Kha, Khoa Kỹ thuật Hóa học, Trường Đại học Bách khoa Tp. HCM, 268 Lý Thường Kiệt, Phường Diên Hồng, Thành phố Hồ Chí Minh, Việt Nam

Đại học Quốc gia Thành phố Hồ Chí Minh, Phường Linh Xuân, Thành phố Hồ Chí Minh, Việt Nam

Email: nmkha@hcmut.edu.vn

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