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Nastanek zlatih nanodelcev pri ultrazvočni razpršilni pirolizi

Formation of Gold Nanoparticles with Ultrasonic Spray Pyrolysis

Povzetek

Ultrazvočna razpršilna piroliza (USP) je poznana metoda za sintezo različnih finih prahov. V prejšnjem raziskovalnem delu smo z USP uspeli sintetizirati različne zlate nanodelce (AuNPs) brez natančnega poznavanja mehanizmov nastanka. Zato, da bi identificirali te mehanizme in imeli večji nadzor nad sintezo AuNPs, smo modificirali USP z ločenim območjem za izhlapevanje kapljic aerosola in z uvedbo reducirnega plina neposredno v reakcijsko peč. V takšnem sistemu smo z 2,5 MHz ultrazvočnim generatorjem ustvarili kapljice aerosola iz raztopine z $HAuCl_4$, kjer so bile koncentracije raztopljenega zlata med 0,5 in 5 g/l. Nosilni plin dušik (s pretokom med 1,5 do 4,5 l/min) je transportiral kapljice v cono izhlapevanja, kjer so bile testirane temperature med 50 in 100°C. Za redukcijo zlatega klorida v AuNPs smo uporabili plin vodik (s pretokom med 1.0 in 2.0 l/min). V reakcijski peči so bile testirane temperature med 300 in 400°C. Ugotovljeno je bilo, da AuNPs nastanejo s kombinacijo mehanizmov pretvorbe kapljic v delec (t.i. DTP mehanizem) in plina v delec (t.i. GTP mehanizem). Eksperimentalno smo potrdili, da so parametri, ki najbolj vplivajo na razmerje med tema mehanizmoma: koncentracija zlata v začetni raztopini in pretoka plinov. Ustrezno izbrani parametri sinteze so minimizirali nastanek AuNPs z DTP mehanizmom in omogočili prevladujočo sintezo z GTP mehanizmom, kar je posledično ustvarilo enotne oblike AuNPs, ki jih pred tem ni bilo možno doseči.

Ključne besede: ultrazvočna razpršilna piroliza (USP), zlati nanodelci (AuNPs), DTP in GTP mehanizem nastanka, presevna elektronska mikroskopija (TEM)

Abstract

Ultrasonic Spray Pyrolysis (USP) is a known method for synthesis of various fine powders. In our previous research work we synthesized different gold nanoparticles (AuNPs) with limited success, as the formation mechanisms were not known for AuNP synthesis with the USP. In order to identify the formation mechanisms and provide greater control over AuNP synthesis, we have modified the USP with a separate aerosol droplet evaporation zone and an introduction of the reduction gas directly into the reaction furnace. A 2.5 MHz ultrasound was used to create aerosol droplets of a solution with $HAuCl_4$, with gold concentrations from 0.5 to 5 g/l. Nitrogen was used as the carrier gas for droplet transportation into the heating zone (gas flow from 1.5 to 4.5 l/min). Hydrogen gas was used for reduction of the gold chloride into AuNPs (gas flow from 1.0 to 2.0 l/min). Heating temperatures were 50-100°C for the evaporation zone and 300-400°C for the reaction furnace. It was identified

that the AuNPs are formed from a combination of the Droplet-to-Particle mechanism, DTP and the Gas-to-Particle mechanism, GTP. The most influential parameters for affecting the ratio between these two formation mechanisms were determined: Gold concentration in the precursor solution and gas flows. Appropriate synthesis parameters have decreased the formation by the DTP mechanism; synthesizing AuNPs mainly by the GTP mechanism and producing more uniform AuNPs, which were not obtained previously.

Keywords: Ultrasonic Spray Pyrolysis (USP), gold nanoparticles, DTP and GTP formation mechanisms, Transmission Electron Microscopy (TEM)

Nastanek zlatih nanodelcev pri ultrazvočni razpršilni pirolizi

Nanomateriali (nanodelci, nanocevke, nanopiramide, itd., materiali z vsaj eno dimenzijo pod 100 nm) imajo različne lastnosti v primerjavi z materiali običajnih dimenzij. Njihove spremenjene fizikalne in kemijske lastnosti izhajajo iz velikega razmerja med površino in prostornino ter visoke površinske aktivnosti. Zaradi tega so uporabni na različnih področjih (elektronika, kemija, biotehnologija, medicina) [1]. Zlasti zanimivi so zlati nanodelci (AuNP), saj imajo dodatno lastnost, imenovano površinska plazmonska resonanca (Surface Plasmon Resonance – SPR) [2]. Ta s pomočjo vpadne svetlobe povzroča nihanje prevodnih elektronov na površini nanodelcev. AuNP imajo dobre fizikalne, kemične in optične lastnosti zaradi plazmonske resonance [3-5]. Običajno so AuNP biološko nereaktivni in so zato primerni za biomedicinsko slikanje in terapijo [6,7]. Takšni AuNP se lahko konjugirajo in funkcionalizirajo s peptidi in se tako lahko uporabljajo za diagnozo in zdravljenje raka [8-10]. Iz naših raziskav je bilo razvidno, da so sferični AuNP z velikostjo 50 nm najbolj primerni za biomedicinske aplikacije [11]. Zato je bil cilj naše raziskave sinteza sferičnih AuNP z ozko velikostno porazdelitvijo okoli 50 nm (70% vseh AuNP), z visoko vsebnostjo Au (vsaj 99,99 mas.% Au).

Znane so različne proizvodne metode za nanodelce; delijo se na pristop izdelave,

Formation of gold nanoparticles with Ultrasonic Spray Pyrolysis

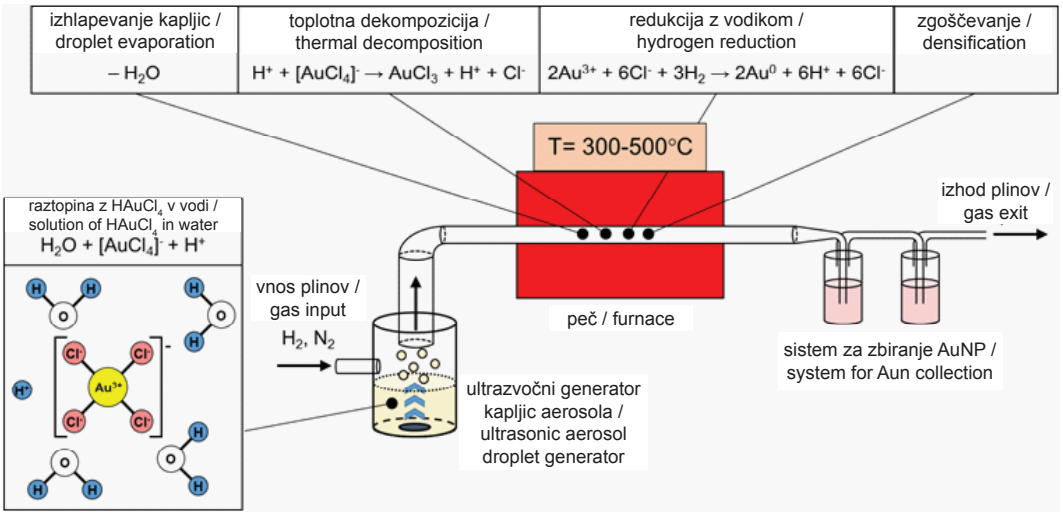
Nanomaterials (nanoparticles, nanotubes, nanopyramids, etc. with at least one dimension below 100 nm) have different properties compared to materials with ordinary dimensions. Their altered physical and chemical properties come from a large surface-to-volume ratio and a high surface activity. Because of this, they are useful in various fields (electronics, chemistry, biotechnology, medicine) [1]. Especially interesting are gold nanoparticles (AuNPs), as they have an additional property, called Surface Plasmon Resonance (SPR) [2]. This causes the oscillation of conduction electrons on the surface of the nanoparticles, stimulated by incident light. AuNPs have good physical, chemical and optical properties because of the Plasmon Resonance [3-5]. Usually, AuNPs are biologically unreactive and, as such, are suitable for biomedical imaging and therapy [6,7]. Such AuNPs can be conjugated and functionalized with peptides, medicine and can be used for diagnosis and cancer treatment [8-10]. From our research, it was shown that spherical AuNPs with sizes of 50 nm are the most optimal for biomedical applications [11]. As such, the aim of our research was the synthesis of spherical AuNPs with a narrow size distribution around 50 nm (70% of all AuNPs), with a high content of Au (at least 99.99 wt.% Au).

od spodaj navzgor (bottom-up) in od zgoraj navzdol (top-down). Primeri od spodaj navzgor so sol-gel, kemično naparjevanje, sinteza s plamenskim razprševanjem, različne pirolize in atomska ali molekularna kondenzacija [12-15]. Metode od zgoraj navzdol vključujejo lasersko ablacijo, nanolitografijo in visoko-energetsko mletje [16-17]. Te metode so trenutno primerne za proizvodnjo majhnih količin nanodelcev z večjimi odstopanji v oblikah in velikostih nanodelcev pri proizvodnji različnih serij. Metoda od spodaj navzgor, imenovana ultrazvočna razpršilna piroliza (Ultrasonic Spray Pyrolysis – USP), ima dober potencial za odpravo teh tehnoloških težav in za bolj nadzorovano sintezo nanodelcev [14,18]. Na splošno je piroliza proces kemične razgradnje različnih spojin pri povišanih temperaturah. Z metodo USP dodatno uvajamo ultrazvok za razprševanje začetne raztopine z želenim materialom v kapljice. Te kapljice so nato izpostavljene visoki temperaturi, tako da se material znotraj kapljic kemično razgradi s pirolizo in pridobijo nanodelci čistih elementov. Prednost metode USP je preprostost postavitve posameznih procesnih segmentov in spreminjanje njihove konfiguracije, neprekinjene sinteze nanodelcev in možnosti sinteze čistih nanodelcev iz različnih materialov. Pomanjkljivost je nizka učinkovitost metode pri uporabi neoptimizirane USP naprave, ki se uporablja za laboratorijske namene, zaradi izgub raztopljenega materiala na konstrukcijskih elementih naprave USP.

Glavni elementi standardne USP naprave so ultrazvočni generator, reaktorska peč in sistem za zbiranje nanodelcev (slika 1). Obstajajo različne surovine, ki jih je mogoče uporabiti za pripravo začetnih raztopin za sintezo AuNP (spojine, ki vsebujejo Au). V našem primeru je bila zaradi njene cene, razpoložljivosti in kemične stabilnosti izbrana tetrakloroavrična kislina $HAuCl_4$ (s),

Different production methods for nanoparticles are known; they are divided into bottom-up and top-down approaches. Bottom-up examples include sol-gel, chemical vapour deposition, flame spray synthesis, various pyrolysis and atomic or molecular condensation [12-15]. Top-down methods include laser ablation, nanolithography and high-energy milling [16-17]. Currently, these methods are suitable for production of small quantities of nanoparticles with major variations in shapes and sizes of the nanoparticles from the production of different batches. A bottom-up method, called Ultrasonic Spray Pyrolysis – USP, has good potential for removing these technological issues, for a more controlled nanoparticle synthesis [14,18]. Pyrolysis in general is a process of chemical decomposition of various compounds at elevated temperatures. With the USP method, we additionally introduce ultrasound for dispersing a precursor solution with our desired material into droplets. These droplets are then exposed to high temperature, such that the material inside the droplet is decomposed chemically via pyrolysis and nanoparticles of pure elements are obtained. The advantage of the USP method is the simplicity of setting up individual process segments and changing their configuration, continuous nanoparticle synthesis and the possibility of synthesizing pure nanoparticles from various materials. The disadvantage is the low efficiency of the method when using an un-optimized USP device used for laboratory purposes, due to losses of the dissolved material on the construction elements of the USP device.

The main elements of the standard USP device are the ultrasonic generator, the reactor furnace and a system for nanoparticle collection (Figure 1). There are various raw materials, which can be used for preparing precursor solutions for AuNP



Slika 1: Sinteza AuNP s konvencionalnim USP

Figure 1: AuNP synthesis with the conventional USP

ki smo jo uporabili tudi v prejšnjih raziskavah s sintezo USP. Začetno raztopino smo pripravili z raztapljanjem $HAuCl_4$ v vodi [19, 20].

Iz literature je znano, da so velikosti sintetiziranih AuNP odvisne od ultrazvočne frekvence [14,18,19,21], ki določa velikost kapljic aerosola in od koncentracije raztopljenega Au v kapljicah. Zaradi vibracij ultrazvoka pod površino raztopine, se kinetična energija molekul raztopine hitro poveča. To povzroči, da se majhne kapljice ločijo od površine raztopine s premagovanjem površinske napetosti. Z visokofrekvenčnim ultrazvokom (0,5-3 MHz) se kapljice ustvarjajo v velikostni porazdelitvi od 1 do 15 mikrometrov [22].

Z uporabo nizkih koncentracij raztopljenega zlata (0,5 g/l - 5,0 g/l Au) v začetni raztopini vsaka kapljica vsebuje tako količino materiala, da se po izhlapevanju in sušenju oblikujejo naslednje velikosti delcev: i) s premerom nekaj 10 nm pri 0,5 g/l Au in ii) s premerom več kot 100 nm pri 5,0 g/l Au v začetni raztopini.

synthesis (compounds containing Au), in our case, tetrachlororauric acid $HAuCl_4$(s) was selected, due to its price, availability and chemical stability, as shown in our previous research with USP synthesis. The precursor solution was prepared by dissolving $HAuCl_4$ in water [19,20].

From literature it is known that the sizes of the synthesized AuNPs depend on the ultrasound frequency [14,18,19,21], which determines the sizes of the aerosol droplets, and the concentration of the dissolved Au in the droplets. Due to vibrations of the ultrasound below the solution surface, the kinetic energy of the solution`s molecules is increased rapidly. This causes small droplets to overcome surface tension and break away from it. With a high-frequency ultrasound (0,5-3 MHz), droplets are created in a size distribution from 1 to 15 micrometers [22].

By using low concentrations of dissolved gold (0,5 g/l – 5.0 g/l Au) in the precursor solution, each droplet contains such an amount of material that, after evaporation

Kapljice začetne raztopine se prenesejo v peč z nosilnim plinom. V peči se AuNP oblikujejo v skladu s stopnjami sinteze:

1. izhlapevanje in krčenje kapljic ($HAuCl_4$ z vodo),
2. toplotna dekompozicija $HAuCl_4$ v $AuCl_3$,
3. redukcija $AuCl_3$ z vodikom in tvorba Au,
4. zgoščevanje delcev.

Naštete stopnje sinteze potekajo istočasno s konvencionalnim USP. Iz kapljic aerosola z manjšimi premeri (2r>1μm) nanodelci nastanejo veliko hitreje kot pri večjih kapljicah (2r<10μm). Tako imamo v USP hkrati majhne delce in kapljice, ter možnost nastanka nanodelcev različnih velikosti in oblik zaradi trčenja in združevanja kapljic z delci. To ni primerno za sintezo ciljnih AuNP.

Z običajnim USP smo sintetizirali AuNP z velikostmi od 10 do 300 nm, z različnimi oblikami, od sferičnih, nepravilnih, trikotnih in cilindričnih [19] (slika 2). Prisotno je bilo tudi veliko nečistoč. Takšni AuNP niso bili primerni.

Podrobnejše študije [14,18,21,23] so pokazale, da so oblike sintetiziranih AuNP odvisne od hitrosti izhlapevanja kapljic ter hitrosti difuzije ionov $[AuCl_4]$- in H+ znotraj kapljice. Hitrosti teh mehanizmov so pri USP odvisne od več dejavnikov: koncentracija začetne raztopine, velikosti kapljic, število kapljic in relativne vlažnosti v sistemu, hitrost prenosa kapljic v peč z nosilnim plinom, tlak v sistemu, dimenzije transportnih cevi in temperaturo v peči. Za nastavitev primernih parametrov (koncentracija začetne raztopine, pretok plinov, temperatura peči) so potrebne informacije o lastnostih začetne raztopine, kot so gostota in površinska napetost, značilnosti raztopljene $[AuCl_4]$- ter difuzije ionov v raztopini in rast AuNP.

Na podlagi predstavljene študije je bil cilj našega raziskovalnega dela postavitev modificirane USP naprave za sintezo

and drying, the following particle sizes are formed: i) with diameters of a few 10 nm at 0,5 g/l Au and ii) with diameters of more than 100 nm at 5.0 g/l Au in the precursor solution.

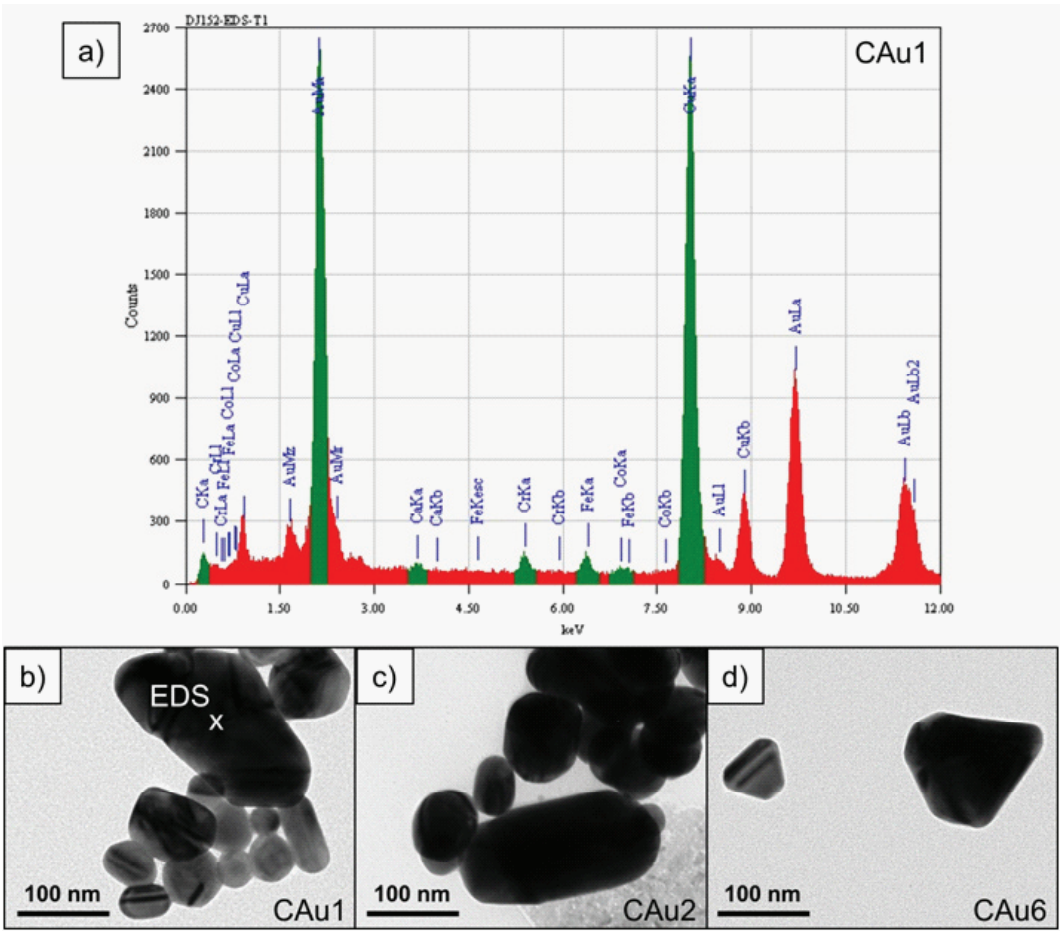
Droplets of the starting solution are transported into the furnace with a carrier gas. Inside the furnace, the AuNPs are formed according to the following synthesis stages:

1. Evaporation and droplet shrinkage ($HAuCl_4$ with water)
2. Thermal decomposition of $HAuCl_4$ into $AuCl_3$
3. Reduction of $AuCl_3$ with hydrogen and the formation of Au
4. Densification

The listed synthesis stages are taking place at the same time with the conventional USP. With smaller diameters of the aerosol droplets (2r>1μm), nanoparticles are formed much sooner than with larger droplets (2r<10μm). Therefore, nanoparticles of different sizes and shapes can be synthesized, due to droplet collisions and coagulation. This is not suitable for synthesizing the targeted AuNPs.

With the conventional USP, we have synthesized AuNPs with sizes from 10 to 300 nm, with different shapes, from spherical, irregular, triangular and cylindrical [19] (Figure 2). A lot of impurities were also present. Such AuNPs were not suitable.

More detailed studies [14,18,21,23] have shown, that the shapes of the synthesized AuNPs depend on the rate of droplet evaporation and the rate of ion diffusion $[AuCl_4]$- and H+ inside the droplet. These rates with USP synthesis depend on several factors: Precursor solution concentration, droplet sizes, number of droplets and relative humidity in the system, velocity of droplet transportation into the furnace with the carrier gas, pressure in the system, dimensions of the transport pipes,



Slika 2: a) EDS analiza AuNP, izdelanih s konvencionalnim USP; b) TEM slika okroglih in nepravilnih AuNP s temperaturo sinteze 260°C-500°C; c) prisotnost cilindričnih AuNP pri sintezi s temperaturo 280°C-500°C; d) prisotnost trikotnih AuNP pri sintezi s temperaturo 260°C. Vsebnost C in Cu v EDS analizi izhaja iz mrežice za TEM, na katero so bili naneseni analizirani AuNP

Figure 2: a) EDS analysis of AuNPs obtained with conventional USP; b) TEM image of spherical and irregular AuNPs at synthesis temperatures from 260°C-500°C; c) Presence of cylindrical AuNPs at synthesis temperatures from 280°C-500°C; d) Presence of triangular AuNPs at synthesis temperatures of 260°C. The C and Cu content in the EDS analyses comes from the TEM formvar grid, on which the AuNPs were examined

AuNP (slika 3). S to spremembo smo ločili izhlapevanje kapljic od preostalih stopenj sinteze in vnesli redukcijski plin neposredno v reakcijsko peč. Na ta način smo lažje nadzorovali izhlapevanje kapljic in kemične

and temperature inside the furnace. For setting up suitable parameters (precursor solution concentration, gas flow, furnace temperature), information is needed for the starting solution properties, such as density

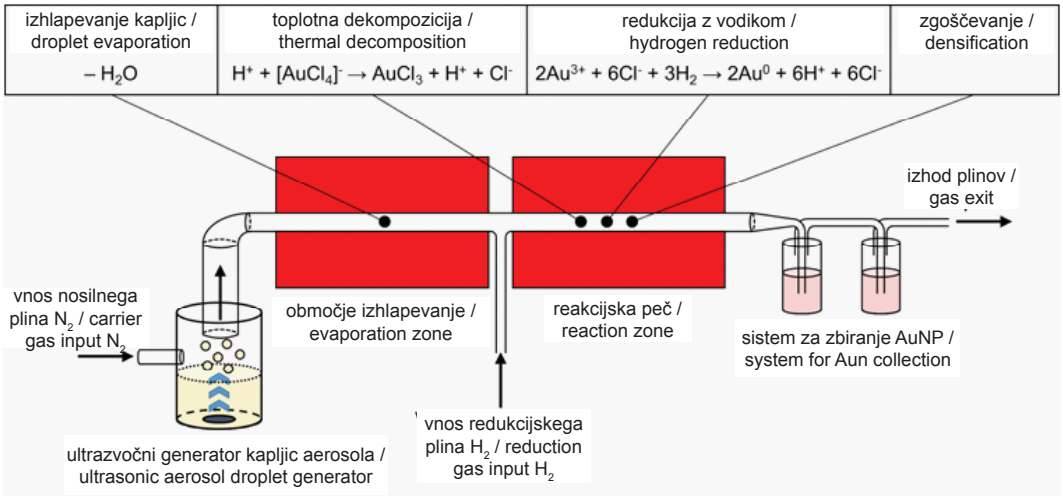
reakcije, da bi dosegli želene velikosti sferičnih AuNP. Predpostavili smo, da bo ločena stopnja izhlapevanja omogočala bolj optimalno difuzijo materiala znotraj kapljice ($[AuCl_4]$- in H+) in nastanek sferičnih AuNP. Mehanizmi sinteze AuNP iz $HAuCl_4$ z USP s tako zasnovo še niso pojasnjeni v literaturi.

Izvedli smo več poskusov z modificiranim USP z različnimi izbranimi vplivnimi parametri (ultrazvočna frekvenca 2,5 MHz, koncentracije zlata v raztopini $HAuCl_4$ od 0,5 do 5 g/l Au, pretok nosilnega plina dušika od 1,5 do 4,5 l/min, pretok redukcijskega plina vodika od 1,0 do 2,0 l/min, temperatura peči 50-100°C za območje izhlapevanja in 300-400°C za reakcijsko peč). Sintetizirane AuNP smo analizirali z različnimi metodami karakterizacije za prepoznavanje njihovih velikosti, oblik, kemične sestave in stopnje aglomeracije. Na podlagi teh rezultatov smo nato presodili glede vpliva posameznih parametrov na mehanizme nastanka AuNP. Ugotovili smo, da AuNP nastanejo iz kapljic in iz plinske faze. To pomeni, da so tvorjeni

and surface tension, and characteristics of the dissolved $[AuCl_4]$-, ion diffusion inside the solution and AuNP growth.

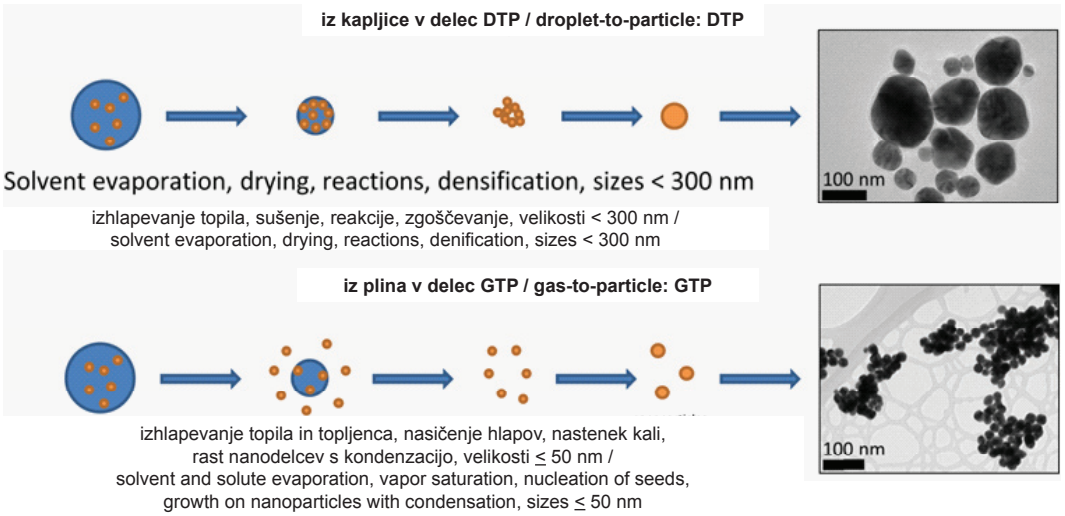
Based on the presented studies, the objective of our research work was setting up a modified USP device for the synthesis of AuNPs (Figure 3). With this modification, we have separated the droplet evaporation from the rest of the synthesis stages and introduced the reduction gas directly into the reaction furnace. In this way, we could control the evaporation and chemical reactions in order to achieve the desired sizes of spherical AuNPs. We presumed, that a separate evaporation stage would allow for a more optimal material diffusion inside the droplet ($[AuCl_4]$- and H+) and would result in the synthesis of spherical AuNPs. The mechanisms of AuNP synthesis from $HAuCl_4$ by USP with such a design are not yet clarified in the literature.

Several experiments were performed with the modified USP, with different selected influential parameters (2.5 MHz



Slika 3: Modifikacija konvencionalnega USP - postopka z ločenim območjem za izhlapevanje kapljic in z vnosom redukcijskega plina neposredno v reakcijsko peč

Figure 3: Modification of the conventional USP with a separate evaporation zone and reduction gas input directly into the reaction zone



Slika 4: DTP in GTP mehanizma pri USP, s pripadajočimi AuNP glede na mehanizem nastanka

Figure 4: DTP and GTP mechanisms in USP, with obtained AuNPs corresponding to the formation mechanisms

iz kombinacije mehanizmov nastanka DTP (Droplet-To-Particle, iz kapljice do delca) in GTP (Gas-To-Particle, iz plina v delce) [14] (slika 4). Naslednja ugotovitev je bila, da sta parametra z največjim vplivom na nastanek bila koncentracija Au v začetni raztopini in pretok plinov N_2 in H_2. Na podlagi teh ugotovitev smo postavili model nastanka, ki pojasnjuje, kako lahko spremenimo ta dva parametra in dosežemo prevlado GTP mehanizma v sistemu. Na ta način lahko dobimo želene AuNP. Model je bil potrjen s sintezo ciljnih, sferičnih AuNP z velikostno porazdelitvijo okoli 50 nm.

Za nadaljnje delo je potrebno oblikovati optimiziran sklop modificiranega USP z večjo učinkovitostjo, kjer bi preprečili nalaganje AuNP na stene transportnih cevi. To je možno z izgradnjo posameznih elementov USP s primernimi materiali za preprečevanje nalaganja. Za povečanje zmogljivosti izdelave večjih količin AuNP z USP sintezo in premik na industrijsko raven je potrebno preučiti in razviti nove

ultrasound frequency, gold concentrations in $HAuCl_4$ solution from 0.5 to 5 g/l, nitrogen carrier gas flow from 1.5 to 4.5 l/min, hydrogen reduction gas flow from 1.0 to 2.0 l/min, heating temperatures of 50-100°C for the evaporation zone and 300-400°C for the reaction furnace). The synthesized AuNPs were characterized with various characterization techniques for identification of their sizes, shapes, chemical composition and degree of agglomeration. Based on these results, we have surmised the influence of individual parameters on the AuNP formation mechanisms. We have found out, that the AuNPs are formed from droplets and from the gas phase. This means that they are formed from a combination of DTP (Droplet-To-Particle) and GTP (Gas-To-Particle) formation mechanisms [14] (Figure 4). The next finding was that the parameters with the most influence on formation were the Au concentration in the precursor solution and N_2 and H_2 gas flows. Based on these findings, we have set

komponente ter sklope USP na podlagi ugotovitev v tem raziskovalnem delu: od proizvodnje aerosola in transport kapljic, dimenzij transportnih cevi ter pogoji pretoka plinov, izhlapevanje/sušenje kapljic in grelni elementi (difuzijski sušilniki, elektrouporovne ali indukcijske peči, gretje z mikrovalovi), do zbiralnega sistema (zbiranje v suspenzijah ali v elektrostatičnih precipitatorjih, glede na uporabo končnih nanodelcev).

Za nadaljnje študije o mehanizmih nastanka AuNP in aplikacijah AuNP je treba sintezo preučiti tudi s drugimi začetnimi sestavinami, namesto $HAuCl_4$. Za sintezo AuNP v razponu od 10 do 50 nm je treba uporabiti tudi dodatne stabilizacijske snovi, ki so primerne za testiranje biokompatibilnosti in potencialno uporabo v biomedicinskih aplikacijah, kar je trenutno primarna uporaba AuNP.

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up a formation model, which explains how we can change these two parameters and achieve dominance of the GTP mechanism in the system. In this way we can obtain the desired AuNPs. The model was confirmed with synthesis of the targeted, spherical AuNPs with a size distribution around 50 nm.

For further work, an optimized assembly with increased efficiency of the modified USP should be designed, where the AuNP deposition would be prevented. This should be done by constructing the individual USP elements with suitable materials for deposition prevention. In order to upscale the USP synthesis of AuNPs to an industrial level, the following components of the USP should be revised and developed a new, based on the findings in this research work: Aerosol generation and droplet transport, transport tube dimensions and gas flow conditions, evaporation/drying and heating elements (diffusion driers, electroresistance or induction furnaces, microwaves), collection system (collection in suspensions or electrostatic precipitators, based on final application).

For further studies regarding AuNP formation mechanisms and applications of AuNPs, the synthesis should also be examined using precursors other than $HAuCl_4$. Additional stabilization agents should also be used for synthesis of AuNPs in the range of 10 – 50 nm, more suited for biocompatibility testing and potential use in biomedical applications, which is currently the primary application of AuNPs.

Acknowledgment

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AKTUALNO / CURRENT

Koledar livarskih prireditev 2018

Datum prireditve	Ime prireditve	Lokacija prireditve
16.-18. 01. 2018	EUROGUSS	Nuernberg, Nemčija
07.- 08.03. 2018	Aachener Giessereikolloquium	Aachen, Nemčija
26.- 27.04. 2018	Grosse Giessereitechnische Tagung (Osterreich, Schweiz, Deutschland)	Salzburg, Avstrija
23.- 25.05. 2018	17th International Foundrymen Conference	Opatija, Hrvaška
5. - 7. 06. 2018	Castforge	Stuttgart, Nemčija
12.- 14.09. 2018	58. mednarodno livarsko posvetovanje	Portorož, Slovenija
23.- 27.09. 2018	73rd World Foundry Congress »Creative Foundry«	Krakow, Poljska