PRELIMINARNA STUDIJA
PRELIMINARY REPORT

SKENING ELEKTRON-MIKROSKOPSKA ANALIZA OBLIKA I VELIČINE ČESTICA RAZLIČITIH AMALGAMSKIH LEGURA

SCANNING ELECTRON-MICROSCOPIC ANALYSIS OF SHAPE AND SIZE OF PARTICLES IN DIFFERENT AMALGAM ALLOYS

Jovanka Gašić, Jelena Daković, Goran Radičević, Milan Spasić

UNIVERZITET U NIŠU, MEDICINSKI FAKULTET, KLINIKA ZA STOMATOLOGIJU, ODELJENJE ZA BOLESTI ZUBA, SRBIJA, SRBIJA I CRNA GORA
UNIVERSITY OF NIŠ, MEDICAL FACULTY, CLINIC OF STOMATOLOGY, DEPARTMENT OF DENTAL PATHOLOGY SERBIA, SERBIA AND MONTENEGRO

Kratak sadržaj

Autori su postavili cilj da se ispita veličina i oblik čestica različitih amalgamskih legura pomoću skening elektronske mikroskopije. Korišćeno je sedam vrsta kapsuliranih amalgama različitih proizvođača (Dentam TM, Amalcap S. A. S., Extracap D, ANA 2000, Cavex non-gamma 2, Cavex Avalloy i Cavex Octight). Ustanovljeno je da se legure Extracap D, ANA 2000 i Cavex Avalloy sastoje samo od sečenih, iregularnih čestica, pri čemu sve tri legure pokazuju razlike u veličini partikula. Kod legure, ANA 2000, ta razlika je najizraženija. Legure Dentam TM, Amacap S.A.S. i Cavex non-gamma 2, predstavljaju kombinovane legure sa iregularnim i sfernim česticama. U pogledu odnosa sferne partikule: iregularne partikule, uzorak legure Dentam TM je pokazao najviši odnos u korist sfernih čestica u poređenju sa ostale dve legure. Legura Cavex Octight sadrži samo čestice pravilnog, sfernog oblika.

***Ključne reči**: Amalgamska legura, oblik i veličina partikula, SEM*

Abstract

The aim of the authors is to investigate the size and shape of particles in different amalgam alloys by using scanning electron microscopy (SEM). They have used seven types of capsulated amalgams made by different manufacturers (Dentam TM, Amalcap S.A.S., Extracap D, ANA 2000, Cavex non-gamma 2, Cavex Avalloy and Cavex Octight). It has been discovered that Extracap D and Cavex Avalloy alloys consist only of lathe-cut particles, where all of them show a great difference in particle size. The most evident difference is in the alloy ANA 2000. DentamTM, Amalcap S. A. S. and Cavex non-gamma 2 are admixed alloys with lathe-cut and spherical particles. Regarding spherical and lathe-cut particle relation, the sample of Dentam TM alloy contains the greatest number of spherical particles as compared to the remaining two alloys. The alloy Cavex Octight contains only regular – shaped spherical particles.

Key words: Amalgam Alloy, particles, SEM

Uvod

Amalgami za zubne ispune predstavljaju legure žive sa srebrom, kalajem i bakrom. Bez obzira na vrlo ekspanzivan razvoj estetske restaurativne stomatologije i usavršavanje ko-

Introduction

Amalgams for dental fillings are alloys of mercury mixed with silver, tin and copper. Regardless of a very expansive development of aesthetic restorative stomatology and the im-

mpozitnih materijala, dentalni amalgami još uvek zauzimaju značajno mesto u konzervativnoj stomatologiji.

Na osnovu sadržaja bakra, oblika partikula i glavnih elemenata prisutnih u sferičnim partikulama mešovitih legura, dentalne amalgamske legure i njihovi odgovarajući amalgami mogu biti klasifikovani u šest grupa[1]:

- Nisko-bakarni amalgami sa iregularnim partikulama (lathe-cut),
- Nisko-bakarni amalgami sa sfernim partikulama (spherical),
- Visoko-bakarni amalgami sa iregularnim partikulama,
- Visoko-bakarni amalgami sa sfernim partikulama,
- Visoko-bakarne mešane legure, Ag-Sn-Cu (admixed alloy),
- Visoko-bakarne mešane legure, Ag-Cu.

Prva dva tipa pripadaju "konvencionalnim" amalgamima i nazivaju se i "srebro-kalaj" legurama. Legura "jednostavnog sastava" ili jednostavna legura (*single composition alloy*) označava visoko bakarnu leguru sa sfernim česticama. Opšti naziv "mešana" legura (*admixed alloy*) podrazumeva leguru koja se sastoji od iregularnih i sfernih čestica, pri čemu sferne patrikule koje imaju visok sadržaj bakra, sadrže uglavnom Ag, Sn i Cu, ili Ag i Cu.[1,2]

Oblik i veličina čestica amalgamske legure imaju uticaja na reakciju vezivanja amalgama, dimenzionalne promene, kao i na neke mehaničke osobine vezanog amalgama.[1,2]

provement of composite materials, dental amalgams still occupy an important position in conservative stomatology.

According to the level of copper, the shape of particles and the main elements of spherical particles in admixed alloys, dental amalgam alloys and their corresponding amalgams can be classified into six groups[1]:

- amalgams with a low level of copper and lathe-cut particles
- amalgams with a low level of copper and spherical particles
- amalgams with a high level of copper and lathe-cut particles
- amalgams with a high level of copper and spherical particles
- admixed alloys with a high level of copper, Ag-Sn-Cu
- admixed alloys with a high level of copper, Ag-Cu.

The first two types belong to "conventional" amalgams and they are called "silver-tin" alloys. A single composition alloy is an alloy with a high level of copper and spherical particles. The common term "admixed alloy" is used for an alloy that is composed of lathe-cut and spherical particles, where spherical particles with a high level of copper contain mainly Ag, Sn and Cu, or Ag and Cu.[1,2]

The shape and size of particles in amalgam alloys influence the reaction of amalgam linking, dimensional changes and some mechanical features of a linked amalgam as well.[1,2]

Cilj istraživanja

U ovom radu je postavljen cilj da se na skening elektronskom mikroskopu analizira veličina i oblik čestica različitih amalgamskih legura.

The aim of the research

This research has for its aim to investigate the size and shape of particles in different amalgam alloys by using scanning electron microscope.

Materijal i metode

U istraživanju je korišćeno sedam vrsta kapsuliranih amalgama različitih proizvođača. Na tabeli 1 dat je prikaz naziva amalgama, njihovih proizvođača i odgovarajući sastav koji je sadržan u prospektu.

Material and methods

Seven types of capsulated amalgams of different manufacturers have been used in this research. Table 1 shows the names of amalgams, their manufacturers and the corresponding composition given in the brochure.

Tabela 1. Amalgamske legure i njihov sastav
Table 1. Amalgam alloys and their composition

	Ag%	Cu%	Sn%	Zn%
1. Dentam™, Scitem, London	45	24	31	
2. Amalcap S.A.S., Lek, Ljubljana	70,1	11,9	18,0	
3. Extracap D, Galenika, Beograd	70	3,3	25,7	
4. ANA 2000, N. D. AB, Angelholm, Sweden	43	25,4	29,6	
5. Cavex non-gamma 2, Cavex, Holland BV	69,2	11,9	18,6	0,3
6. Cavex Avalloy, Cavex, Holland BV	45	24	30,5	0,5
7. Cavex Octight, Cavex, Holland BV	60	12	28	

Uzorci amalgamskih legura dobijeni su iz originalnih kapsula pažljivim odvajanjem praha od žive koja se nalazi u blisteru. Standardni nosači uzoraka za SEM premazani su lepkom i preko njih je posut amalgamski prah. Uzorci različitih amalgamskih legura su konvencionalnom metodom pripremani za mikroskopiranje i posmatrani na skening elektronskom mikroskopu tipa JEOL-JSM-5300. Mikrografije su snimljene na uvećanju 150x.

The samples of amalgam alloys have been obtained from the original capsules by careful isolation of powder from mercury which is placed in a capsule. Standard samples for SEM are smeared all over with glue and then sprinkled with the amalgam powder. Using a conventional method, the samples of different amalgam alloys have been prepared for microscopy and examined under the scanning electron microscope, type JEOL-JSM-5300. The magnification of these microscopic images is 150 times the original size.

Rezultati

Dobijeni rezultati su prikazani na mikrografijama od 1 do 6.

Uočeno je da **Dentam™** amalgamska legura sadrži sferne i iregularne (lathe-cut-iveričasto sečene) partikule različite veličine. Većina sfernih partikula je malog dijametra koji ne prelazi 10 µm. Postoji i manji broj krupnijih, čiji je prečnik oko 30 µm a uočeno je nekoliko najkrupnijih sfernih partikula prečnika oko 60 µm. Osim pravilnih sfernih partikula, legura sadrži i elipsaste i kruškolike čestice. Čini se da su iregularne čestice zastupljene u manjem broju. Približan odnos dužine i širine čestica nepravilnog oblika iznosi 3:1 (Slika 1).

Leguru **Amalcap S.A.S.** čine krupne partikule prosečne dužine od 70 do 120 µm, širine od 40 do 60 µm (mada je prisutan i manji broj čestica dužine i do 200 µm), nepravilnog (poliedarskog) oblika čija je iregularnost jače izražena nego kod prethodnog uzorka. Uzorak legure sadrži malu količinu sfernih čestica malih dimenzija ($r \approx 10$ µm) i nekoliko većih ($r \approx 50$ µm) (Slika 2).

Results

The microscopic images 1 – 6 show the obtained results.

It has been observed that **Dentam™** amalgam alloy contains both spherical and lathe-cut particles of different size. The diameter of most of the spherical particles is very small, not more than 10 µm. There is also a number of bigger particles whose diameter is about 30 µm, as well as a number of the biggest spherical particles of about 60 µm in diameter. Beside regular spherical particles, the alloy is composed of both elliptical and pear-like particles. It seems that there is a small number of lathe-cut particles. The approximate proportion of the length and width of particles is 3:1 (Figure 1).

The alloy **Amalcap S.A.S.** is made up of big particles with irregular (polyhedral) shape, whose approximate length is 70 to 120 µm, and approximate width 40 to 60 µm (though there is a small number of particles with 200 µm in width), and whose irregularity is more evident than in the previous sample. The sample of the alloy contains a small amount of spherical particles of

Slika 1. ***Dentam*** *™. SEM amalgamske legure pokazuje mnoštvo sfernih i elipsastih partikula različite veličine i manji broj čestica iregularnog oblika*

Figure 1. ***Dentam*** *™. SEM image of the amalgam alloy shows a great number of spherical and elliptical particles of different size and a small number of lathe-cut particles*

Slika 2. ***Amalcap S.A.S.*** *Krupne partikule sa izraženom iregularnošću. U središtu mikrografije zapaža se "grozd" sferičnih čestica izuzetno malih dimenzija*

Figure 2. ***Amalcap S.A.S.*** *Bigger particles with the apparent irregularity. In the centre of the image a "grape" of spherical particles of extremely small dimensions can be observed*

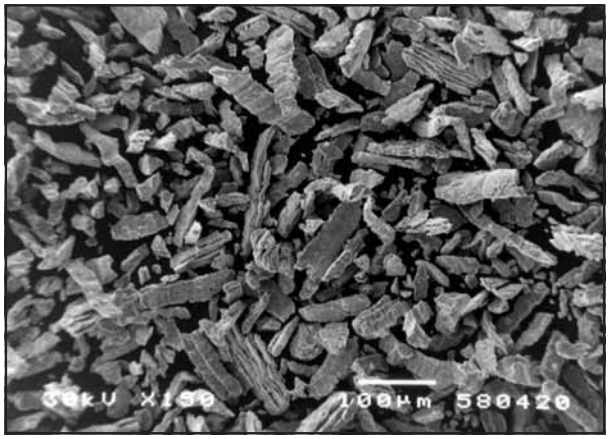
Extracap D legura se sastoji samo od "lathe-cut" čestica. Uzorak pokazuje manju nepravilnost oblika i velike razlike u dimenzijama partikula. Dužina malobrojnih sitnih partikula iznosi oko 20 µm, dok najkrupnije imaju dužinu i do 130 µm, a širinu između 10 i 40 µm (Slika 3).

Kod legure **ANA 2000** uočene su samo čestice nepravilnog oblika. Sitnije partikule imaju dužinu od oko 30 do 90 µm, a krupne i do 200 µm. Širina i sitnih i krupnih čestica veoma varira zbog izražene iregularnosti: neke čestice su oblika kocke, druge više podsećaju na kvadar ili prizmu. Oblik čestica je teško opisati zbog neravnomernih i često nazubljenih površina (Slika 4).

small dimensions (10 µm in diameter) and a few bigger ones (50 µm in diameter) (Figure 2).

Extracap D alloy consists only of lathe-cut particles. The sample shows a minor irregularity of shape and a big difference in the dimensions of particles. A number of tiny particles are about 20 µm in length, while the biggest ones are up to 130 µm in length and between 10-40 µm in width (Figure 3).

In the alloy **ANA 2000** only particles of irregular shape have been observed. Smaller particles are about 30-90µm in width, and bigger ones about 200µm. The width of both small and big particles varies a lot because of the obvious irregularity: some particles are cube-shaped,



Slika 3. ***Extracap D****. Amalgamska legura sastavljena samo od "lathe-cut" čestica. Postoji manja iregularnost oblika čestica, ali su izražene razlike u njihovoj veličini*

Figure 3. ***Extracap D****. Amalgam alloy made up only of lathe-cut particles. There is a minor irregularity in particle shape, but differences in their size are obvious*

Slika 4. ***ANA 2000.*** *Uzorak pokazuje leguru koja se sastoji samo od iregularnih čestica čiji oblik varira (kvadar ili poliedar sa neravnim ili nazubljenim ivicama). Postoji izrazita razlika u njihovoj veličini*

Figure 4. ***ANA 2000.*** *The sample shows an alloy which consists only of lathe-cut particles whose shape varies (rectangle or polyhedron with irregular or rough edges). There is a considerable difference in their size*

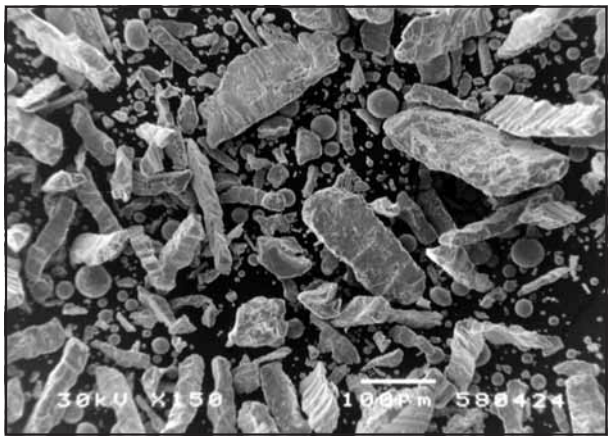
Cavex non-gamma 2 predstavlja kombinovanu leguru koja sadrži i sferne i iregularne čestice. Sferne partikule su malih dimenzija (od 7 do 50 µm). Među "lathe-cut" česticama postoje izuzetno krupne čija dužina iznosi do 250 µm, a širina približno 90 µm (Slika 5).

Legura **Cavex Avalloy** sadrži samo sečene iregularne čestice sa izraženom razlikom u njihovoj veličini. Dužina najsitnijih partikula je manja od 10 µm, dok najkrupnije imaju dužinu oko 120 µm. Razlika u veličini čestica je izraženija nego kod legure Ekstracap D, ali je manje uočljiva nego kod legure ANA 2000 (Slika 6).

others resemble a rectangle or a prism. The shape of particles is hard to describe because of irregular and often rough surfaces (Figure 4).

Cavex non-gamma 2 is an admixed alloy which contains both spherical and lathe-cut particles. Spherical particles are of small dimensions (7-50µm). Among lathe-cut particles there are those which are extremely big, up to 250 µm in length and approximately 90 µm in width (Figure 5).

The alloy **Cavex Avalloy** contains only lathe-cut particles which visibly differ in size. The smallest particles are less than 10µm in length while the length of the biggest ones is about 120µm. The difference in particle size is more evident than in Ekstracap D alloy, but less apparent than in ANA 2000 alloy (Figure 6).



*Slika 5. **Cavex non gamma-2.** SEM amalgamske legure pokazuje kombinovan sastav: sitne sferične partikule i izuzetno krupne iregularne čestice*

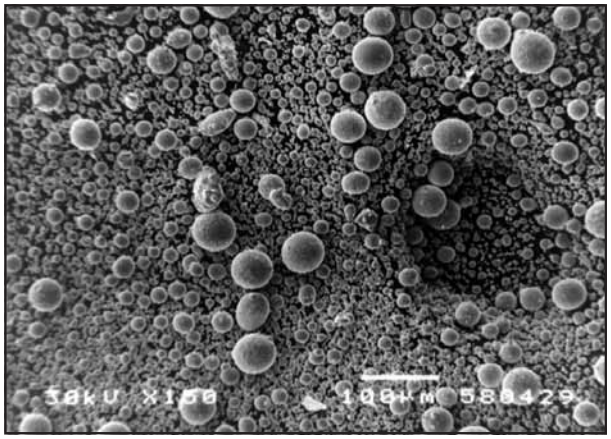
*Figure 5. **Cavex non gamma-2.** SEM image of the amalgam alloy shows a combined composition: tiny, spherical particles and extremely big lathe-cut particles*

*Slika 6. **Cavex Avalloy.** Amalgamska legura koja sadrži samo iregularne čestice. Postoji izrazita razlika u veličini čestica*

*Figure 6. **Cavex Avalloy.** The amalgam alloy which contains only lathe-cut particles. There is a considerable difference in particle size*

Cavex Octight legura sastoji se samo od sfernog praha. Dijametar čestica se kreće između 5 i 60 µm (Slika 7).

Cavex Octight alloy consists only of the spherical powder. Particles are between 5-60 µm in diameter (Figure 7).



*Slika 7. **Cavex Octight.** Sferni prah amalgamske legure. Čestice uglavnom pravilnog, kružnog oblika sa izraženom razlikom u dijametru*

*Figure 7. **Cavex Octight.** The spherical powder of amalgam alloy. Particles of mostly regular, bigger shape which obviously differ in diameter*

Diskusija

Oblik i veličina čestica amalgamske legure od značaja su za reakciju vezivanja i kvalitet dobijenog amalgama.[3,4] Podaci iz literature sugerišu da su manja zrna podesnija za reakciju vezivanja sa živom. Međutim, zbog tehnologije dobijanja amalgamskih partikula, izgleda da su razlike u veličini čestica neminovne. Smatra se da prosečna veličina sečenih partikula u legurama sa optimalnim osobinama treba da iznosi 25-35 µm.[2] Legura **ANA 2000**, kako je zapaženo u ovom ispitivanju, sadrži veći broj izuzetno sitnih i manji broj većih iregularnih partikula, tako da postoji izrazita razlika u njihovoj veličini. Kod legure **Cavex Avalloy** ta je razlika manje uočljiva. **Extracap D** legura sadrži manji broj sitnih čestica u odnosu na prethodne dve. Smatra se da ekstremno sitne partikule u legurama prouzrokuju veću stopu marginalnog preloma plombe.[5] Ako su čestice manje od 3µm, biće potrebno više žive tokom amalgamacije. Ukoliko su čestice veće, povećava se osetljivost na koroziju.[3,6]

Veličina i oblik čestica amalgamske legure utiče i na dimenzionalne promene. Manje čestice pravilnijeg (sfernog) oblika (zbog lakše reakcije sa živom) dovode do lakšeg i bržeg rastvaranja srebra i kalaja, što favorizuje kontrakciju.[7] Stariji klasični amalgami, koji su dobijani iz amalgamskih legura većih i nepravilnijih čestica i sa većom količinom žive u odnosu živa: legura, pokazivali su dominantnu ekspanziju u procesu vezivanja, dok se kod modernih amalgama, zapaža manja finalna ekspanzija.[2,8,9] U kombinovanim legurama (**Dentam™, Amalcap S.A.S. i Cavex non gamma 2**), kako je ovo istraživanje pokazalo, postojala je razlika i u veličini čestica određenog oblika kao i u odnosu sferne/iregularne partikule. Manje sferne čestice amalgamske legure daju veću čvrstoću i, uopšte, bolje mehaničke osobine vezanog amalgama nego čestice drugačijih, nepravilnijih oblika. Amalgam sa optimalnim mehaničkim osobinama dobija se sfernim česticama veličine 5-40 µm.[2,10] Sferni oblik čestica omogućava najpovoljnije pakovanje, usled čega se smanjuje slobodan prostor između njih, pri čemu je i ukupna zapremina amalgama manja.[2,10,11] Ovo istraživanje je pokazalo da legura **Cavex Octight** sadrži isključivo sferne čestice koje bi mogle da odgovaraju optimalnoj veličini.

Discussion

The size and shape of particles in the amalgam alloy are important for a linking reaction and the quality of a produced amalgam.[3,4]The literature data suggest that smaller grains (particles) are more suitable for a linking reaction with mercury. However, because of the technology of getting amalgam particles, it seems that the differences in particle size are inevitable. It has been considered that the average size of lathe-cut particles in alloys with optimal features should be 25-35 µm.[2] **ANA 2000** alloy, as it has been observed in this research, contains a large number of extremeny tiny, irregular particles and a small number of bigger ones, so that there is a significant difference in their size. In the alloy **Cavex Avalloy**, this difference is less evident. **Extracap D** alloy contains a small number of tiny particles in relation to the other two(previous ones). It is considered that the extremely small particles in alloys cause a higher rate of a marginal filling crack.[5] If the particles are less than 5µm in diameter, more mercury will be needed during amalgamation. If particles are bigger, sensitivity to corrosion is greate.[3,6]

The size and shape of particles in the amalgam alloy also affect dimensional changes. Smaller particles of more regular (spherical) shape (because of an easier reaction with mercury) lead to an easier and faster dissolving of silver and tin, which then leads to a contraction.[7] Former, typical amalgams, obtained from the amalgam alloys of bigger and more irregular particles and with a larger amount of mercury in the proportion mercury: alloy have shown a dominant expansion in the linking process, while, in modern amalgams, a smaller final expansion is observed.[2,8,9] In combined alloys, (**Dentam™, Amalcap S.A.S. i Cavex non gamma2**), according to this research, there has been a difference in the size of the particles of a certain shape, and in the spherical/irregular particle proportion as well. Smaller spherical particles in the amalgam alloy provide better solidity, and generally, better mechanical features of the linked amalgam than particles of different, more irregular shapes. The amalgam with optimal mechanical features is obtained from spherical particles, 5-40 µm in diameter.[2,10] The spherical shape of particles makes the most suitable package, which reduces a free space between them and as a result makes the total volume smaller.[2,10,11] This research has shown that **Cavex Octight** alloy contains only spherical particles which could correspond to an optimal size.

Za detaljnija ispitivanja oblika i veličine čestica amalgamskih legura potrebno je primeniti metodu poluautomatske obrade SEM grafija i stereološku analizu strukturnih karakteristika čestica amalgamske legure. Korišćenjem "Kontron-MOP Videoplan" digitalne table, koja nije bila dostupna autorima, moguće je odrediti faktore oblika (sferičnosti) i maksimalne prečnike čestica.

S obzirom da oblik i veličina čestica (tj. opseg u kome se kreće veličina čestica) dentalne amalgamske legure direktno utiču na osobine dobijenog amalgama, autori planiraju nastavak istraživanja u smislu ispitivanja kompaktnosti vezane legure i osetljivosti dentalnog amalgama na koroziju.

For a detailed research of the size and shape of particles in amalgam alloys it is necessary to apply a method of semiautomatic processing of SEM images, and stereological analysis of the structural characteristics of particles in the amalgam alloy. By using "Kontron-MOP Videoplan" digital table, which wasn't available to the authors, it is possible to determine the factors of shape (spherical) and the maximal diameters of particles.

Considering that the shape and size of particles (that is, the range of particle size) in dental amalgam alloys directly affect the features of the produced amalgam, the authors are planning to continue the research in order to investigate compatibility of a linked alloy and sensitivity of dental amalgam to corrosion.

Zaključak

• Legure *Extracap D*, *ANA 2000* i *Cavex Avalloy* sastoje se samo od sečenih, iregularnih čestica. Sve tri legure pokazuju razlike u veličini partikula, s tim što je kod legure ANA 2000, ta razlika najizraženija.

• Legure *Dentam*™, *Amalcap S.A.S.* i *Cavex non gamma 2* predstavljaju kombinovane legure sa iregularnim i sfernim česticama. Odnos sferne/iregularne partikule je kod legure *Dentam* ™ najveći u korist sfernih partikula, u poređenju sa ostale dve legure. Prečnici sfernih partikula u pojedinačnim legurama ne pokazuju velike razlike, dok se kod čestica nepravilnog oblika uočava znatna razlika u dimenzijama. Najkrupnije iregularne čestice sadrži legura **Cavex non gamma 2**.

• Legura **Cavex Octight** sadrži samo čestice pravilnog, sfernog oblika, čiji se dijametar kreće u onim granicama koje bi vezanom amalgamu mogle pružiti optimalne osobine.

Conclusion

• *Extracap D*, *ANA 2000* and *Cavex Avalloy* alloys consist only of lathe-cut particles. All of them show differences in particle size but the most evident difference is in *ANA 2000* alloy.

• *Dentam*™, *Amalcap S.A.S.* and *Cavex non gamma 2* alloys are combined alloys with lathe-cut and spherical particles. The biggest spherical/lathe-cut particle proportion is in the alloy *Dentam* ™ in favour of spherical particles, as compared to the remaining two alloys. The diameters of spherical particles in some alloys don't show significant differences, while diameters of irregular particles differ a lot. *Cavex non gamma 2* contains the biggest irregular particles.

• *Cavex Octight* alloy contains only particles of regular, spherical shape whose diameters would provide optimal features to a linked amalgam.

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Adresa za korespondenciju:

Jovanka Gašić
Medicinski fakultet
Klinika za stomatologiju
Braće Tasković 52, Niš
018 226-216
018 245-689

Address for correspondence:

Assoc. Prof. Jovanka Gašić, D.D.S., M.S.D., Ph.D.
52 Blvd Zoran Djindjić
18 000 Niš
Serbia and Montenegro
Phone: +381 (0) 18 226 216
+381 (0) 18 245 689