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SYNTHESIS OF SILVER'DOPED FLUOROAPATITE AS PERSPECTIVE FILLER FOR FOOD PACKAGE

Vojislav Stanić^a, Ana S. Radosavljević-Mihajlović^a, Vukosava Živković-Radovanović^b,
Branislav Nastasijević^a, Jelena P. Marković^a, Marija Janković^a, Karolina Kalić^c,
Dragoljub Jovanović^d

^a*University of Belgrade, Vinča Institute of Nuclear Sciences, P.O. Box 522, 11001 Belgrade, Serbia*

^b*University of Belgrade, Faculty of Chemistry, P.O. Box 51, 11158 Belgrade, Serbia*

^c*University of Belgrade, Faculty of Physical Chemistry, P.O. Box 137, 11158 Belgrade, Serbia*

^d*University of Belgrade, Faculty of Veterinary Medicine, 11000 Belgrade, Serbia*

voyo@vinca.rs

Antimicrobial material based on fluorapatite with 0,5% silver, was done by wet method from CaO, NaF, AgNO₃ and H₃PO₄. Characterization studies from XRD, SEM and FTIR spectra showed that particles of the sample is of nano size and they do not contain any discernible crystalline impurity. Antimicrobial studies have demonstrated that the silver-doped fluorapatite sample exhibit excellent antimicrobial activity in vitro against the Staphylococcus aureus. The percentages of viable cells reduction was 98%.

Antimicrobial packaging is one of the innovative food packaging concepts that has been introduced as the need to maintain quality of the food for a long-time. Microbial contamination of food reduces the quality of food, limits the shelf life of the food, and increases the risk from illness of the consumer. The use of packaging materials containing antimicrobial agents must meet several conditions: efficiency and the migration of the antimicrobial agents from the packaging material to the surface of the product negligible.

Commercial polymeric composites containing silver-substituted zeolite (Zeomic®, AgIon®, Apacider®, and Bactekiller®) proved to be excellent material for food packaging.

The objective of this research is to synthesize new antimicrobial material based on silver-doped fluorapatite (AgFAP) as polymer composite filler. Antimicrobial activities of the synthesized material was tested for Staphylococcus aureus. That pathogen is very important, because it has the ability to make many different toxins that are frequently responsible for food poisoning [3]. Antimicrobial materials based on silver-doped apatite have shown excellent antimicrobial activity, long lasting and physico-chemical stability [4].

The starting materials were CaO, AgNO₃, H₃PO₄ and NaF of p.a. grade of purity. The Ca(OH)₂ suspension was prepared by stirring a required amount of CaO into 500 mL distilled water heated to 60 °C. A required amount of AgNO₃ and NaF was dissolved in 300 mL of 0.5 M H₃PO₄. The Ag fraction is represented at starting atomic ratios, Ag/(Ca + Ag); 0.005 with the atomic ratio (Ag + Ca)/P, fixed at 1.67. Then the solution was added dropwise to a suspension of Ca(OH)₂. After the titration, stirring in the suspension at 25 °C was continued for a further 10 h. The obtained precipitate was filtered in a Buchner funnel, washed by distilled water, dried at 105 °C and grained in a mortar.

Antimicrobial activities of the sample was investigated against *S. aureus* by liquid challenge method in buffer solution (pH 7.4) according to Stanić et al. [4].

Synthesized sample was analyzed by X-ray diffractometry, XRD (Philips PW 1050), using CuK1.2 Ni-filtered radiation.

The FTIR spectra of synthesized apatite sample, was recorded by a Nicolet 6700 FTIR spectrophotometer (Thermo Scientific) using the ATR technique.

The morphology of the obtained powder was studied by scanning electron microscopy (SEM) TESCAN Mira3 XMU at 20 kV. The XRD patterns of the sample is shown in Fig. 1.

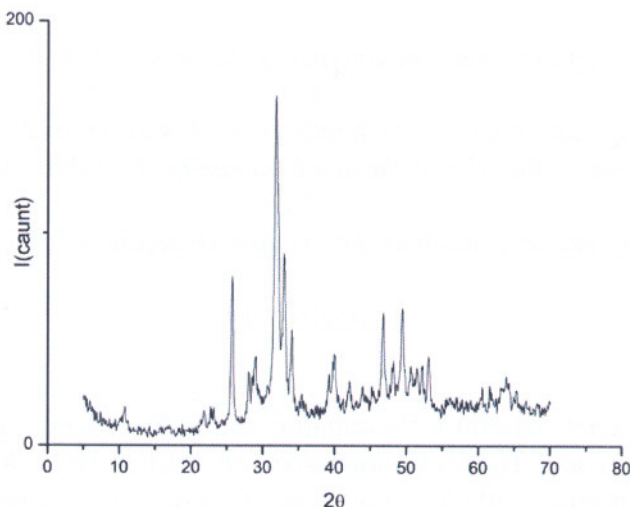


Fig. 1. XRD pattern of the AgFAP sample.

The XRD patterns of AgFAP sample is in accordance with ASTM data (Card 15-0876).

All of the diffraction peaks were sharp and well resolved, indicating the obtained phase pure, well crystallized silver-doped fluorapatite. Fig. 2 shows the FTIR spectra of the AgFAP sample.

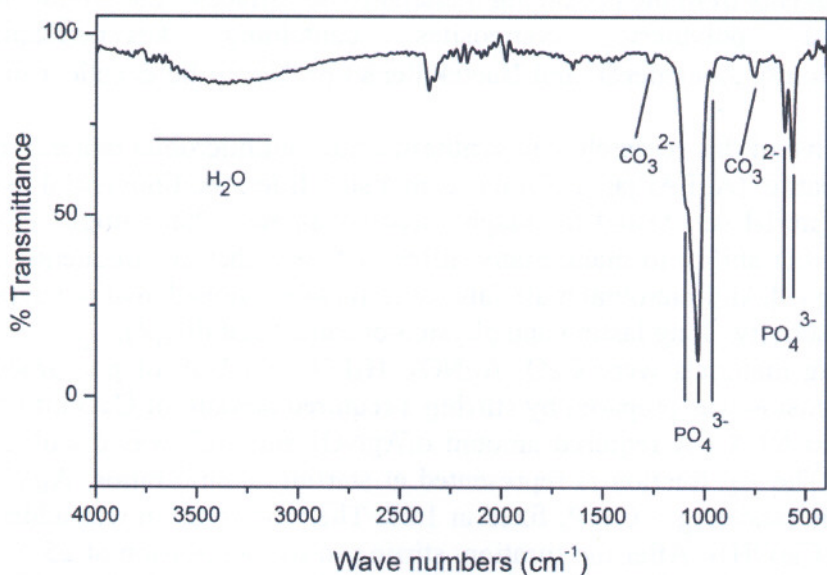


Fig. 2. FTIR spectra of the AgFAP sample

The spectra show the characteristic bands of absorbed water, carbonate and phosphate species. SEM micrograph of the synthesized sample is presented in Fig. 3.

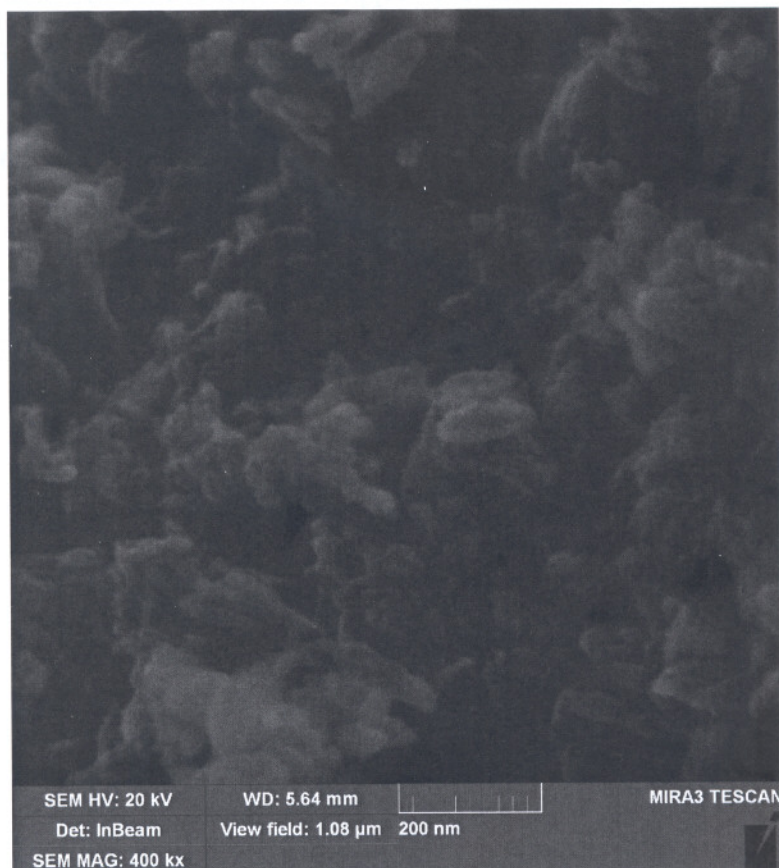


Fig. 3. SEM micrographs of the AgFAP sample

It can be seen that the particles are more or less uniformed in size and of irregular shape in all cases. The average length of particles in all samples is about 80 nm and about 15 - 25 nm in diameter.

The results of the quantitative antimicrobial tests in liquid medium demonstrate that the AgFAP sample show viable cell reduction of 98% of *S. aureus*.

In this study, the wet method has been successfully used for synthesis of monophasic silver-doped fluorapatite nanoparticles, which have antimicrobial properties. The analysis of XRD, FTIR and SEM showed that particles of silver-doped fluorapatite sample are of nano size and homogenous in composition. Antimicrobial study shows that synthesized sample has excellent activity against *S. aureus*. Thus, prepared silver-doped fluorapatite nanopowders can be applied as antimicrobial materials of various purpose such as for synthesis of antimicrobial food packaging, treatment of microbiologically polluted water, for medicine and for general use.

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- [1] P. Appendini, J. H. Hotchkiss, *Innovative Food Sci. Emerging Technol.* 2002, **3**, 113-126.

- [2] Y. Echevoyen, C. Nerín, *Food Chem. Toxicol.* 2013, **62**, 16–22.
- [3] Y. L. Loir, F. Baron, M. Gautier, *Genet. Mol. Res.* 2003, **2**, 63-76.
- [4] V. Stanić, D. Janačković, S. Dimitrijević, S. B. Tanasković, M. Mitrić, M. S. Pavlović, A. Krstić, D. Jovanović, S. Raičević, *Appl. Sur. Sci.* 2011, **257**, 4510–4518.