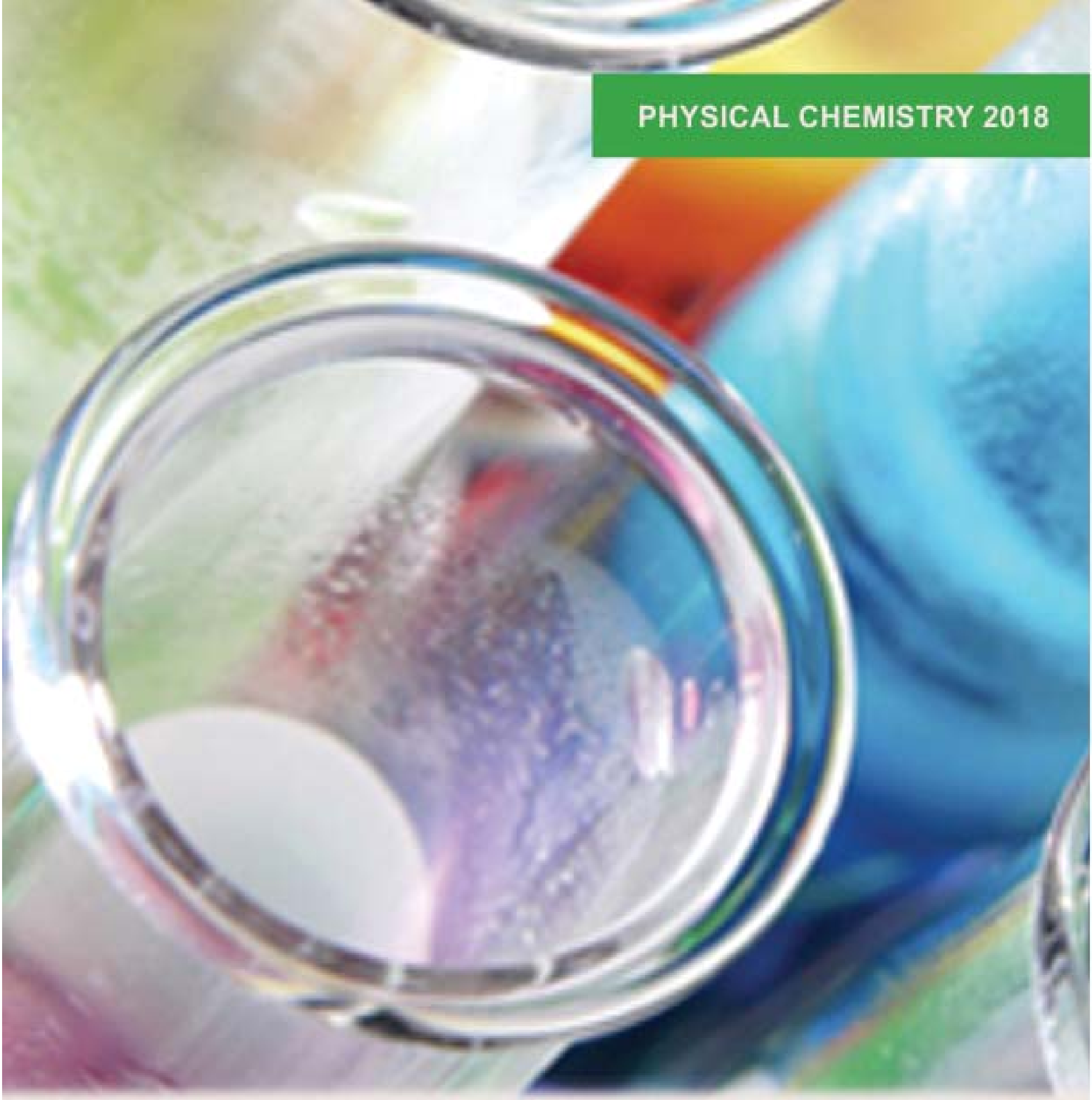
PHYSICAL CHEMISTRY 2018



6th Workshop

SPECIFIC METHODS FOR FOOD SAFETY AND QUALITY

September 27th 2018, Vinča Institute of Nuclear Sciences, Belgrade, Serbia

PROCEEDINGS

SPECIFIC METHODS FOR FOOD SAFETY AND QUALITY

6^{th} WORKSHOP: SPECIFIC METHODS FOR FOOD SAFETY AND QUALITY

September 27^{th}, 2018, Belgrade, Serbia

is a satellite event of

PHYSICAL CHEMISTRY 2018

14^{th} International Conference on Fundamental and Applied Aspects of Physical Chemistry

Organized by

VINČA INSTITUTE OF NUCLEAR SCIENCES
Vinča – Belgrade, Serbia

in co-operation with

THE SOCIETY OF PHYSICAL CHEMISTS OF SERBIA

and

FACULTY OF PHYSICAL CHEMISTRY
UNIVERSITY OF BELGRADE, SERBIA

Held under the auspices of the

MINISTRY OF EDUCATION, SCIENCE AND TECHNOLOGICAL DEVELOPMENT

Organizing Committee

Chairman
Branislav Nastasijević (Serbia)

Members
Mirjana Čolović (Serbia)
Milovan Stoiljković (Serbia)
Aleksandra Bondžić (Serbia)
Ana Vujačić Nikezić (Serbia)
Jasmina Dimitrić Marković (Serbia)

International Scientific Committee

Chairman
Vesna Vasić (Serbia)

Members
Giovanna Marazza (Italy)
Enisa Omanović-Mikličanin (Bosnia and Herzegovina)
Polonca Trebše (Slovenia)
Robert Săndulescu (Romania)
Claudia Nunes Pinho (Portugal)
Salette Reis (Portugal)
Raul D. Rodriguez (Russia)
Evgeniya Sheremet (Russia)
Tatjana Momić (Serbia)
Andreja Leskovac (Serbia)
Sandra Petrović (Serbia)
Tamara Lazarević-Pašti (Serbia)

PHYSICAL CHEMISTRY 2018

14th International Conference on Fundamental and Applied Aspects of Physical Chemistry

6th Workshop

SPECIFIC METHODS FOR FOOD SAFETY AND QUALITY

September 27th, 2018, Vinča Institute of Nuclear Sciences, Belgrade, Serbia

PROCEEDINGS

BELGRADE, SERBIA 2018

6^{th} WORKSHOP: SPECIFIC METHODS FOR FOOD SAFETY AND QUALITY

PROCEEDINGS

Publisher
VINČA INSTITUTE OF NUCLEAR SCIENCES
Vinča - Belgrade, Serbia

Editor
Dr Vesna Vasić

Reviewers
Dr Sandra Petrović
Dr Andreja Leskovac
Dr Tamara Lazarević-Pašti
Dr Tatjana Momić

Design
Milica Ševkušić

Printed by
Skripta Internacional, Beograd

Print run
70 copies

ISBN
978-86-7306-148-1

BELGRADE, SERBIA 2018

PREPARATION AND ANTIMICROBIAL ACTIVITY OF POLYETHYLENE COMPOSITE WITH SILVER-DOPED FLUORAPATITE

V. Stanić[1], K. Kalić[1], S. Dimović[1], D. Milojkov[1, 2], S. Tanasković[3] and V. Živković-Radovanović[4]

[1]University of Belgrade, Vinča Institute of Nuclear Sciences, P.O. Box 522, 11001 Belgrade, Serbia. *(voyo@vinca.rs)*
[2]University of Novi Sad, Faculty of Technology, Department of Materials Engineering, Bulevar Cara Lazara 1, 21000 Novi Sad, Serbia.
[3]University of Belgrade, Faculty of Pharmacy, 11000 Belgrade, Serbia.
[4]University of Belgrade, Faculty of Chemistry, P.O. Box 51, 11158 Belgrade, Serbia.

ABSTRACT

The high-density polyethylene/silver-doped fluorapatite composite (HDPE/AgFAP) with 1 wt% of AgFAP was produced using a twin screw extruder. Characterization studies of XRD and TEM results showed that AgFAP particles were drowned and partially dispersed in the polymer. Antimicrobial study has demonstrated that composite exhibits antimicrobial activity *in vitro* against the *S. aureus*.

INTRODUCTION

Antimicrobial packaging is one of the innovative food packaging concepts that has been introduced as the need to maintain the quality of 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 negligible migration of the antimicrobial agents from the packaging material to the surface of the product. Different materials and methods can be used in order to develop antimicrobial packaging systems. Antimicrobial packaging can be made by incorporation and immobilization of antimicrobial agents or by surface modification and surface coating [1-3].

Several antimicrobial compounds have been combined with different types of carriers (plastic and rubber articles, paper-based materials, textile fibrils and food-packaging materials) for commercial use (Table 1). Most systems consist of silver active agents. Silver ions in particular show oligodynamic effect with a minimal development of microorganism's resistance [4]. Low concentrations are not toxic to mammals, but high concentrations can cause cytotoxicity.

Table 1. Examples of commercial antimicrobial packaging products and manufacturers.

Concentrate	Antimicrobial agent	Trade Name	Manufacturer
	Ag-Zeolite; Cu-Zeolite	Agion® Antimicrobia l	AgION Technologies LLC (USA)
	Ag-zeolite and others Composites Metals (Ag, Cu or Zn) with hydroxyapatite	Apacider	Sangi Co. (Japan)
	Ag-zeolite	Zeomix	Shinanen New Ceramics Co. (Japan)
	M-oxide; M-silicate; (M= Ag, Cu or Zn)	MicroFree	DuPont (USA)
	Ag-oxide	Piatech	Daikoku Kasei Co.(Japan)
	Ag-oxide	Silvi Film	Nimiko Co. (Japan)
	Ag-halide	Surfacine	Surfacine Development Co. (USA)
Extract	allyl mustard oil	WasaOuro®	
	Bamboo extract	Take Guar	Takex Co.(Japan)

EXPERIMENTAL

The $CaCO_3$, Ag_2O and H_3PO_4 (Merck) were used for the synthesis of AgHAP powder. The CaO was obtained by calcinations of $CaCO_3$ for 20 h at 1000°C. The HDPE Hiplex HHM 5502, copolymer of ethylene and hexene, (ρ=0.955 g/cm3 , Mw=300 000) (HIP-Petrohemija a.d., Pancevo, Serbia), Irganox 1076 (Ciba Specialty Chemicals) and PEG 4000 (Merck) were used in the preparation procedure.

The synthesis of AgFAP was prepared by neutralization method in an air atmosphere [1].
The HDPE/AgFAP composite was manufactured via extrusion in a twin screw extruder Haake rheomix. The extrusion process was carried out at a screw speed of 11.6 rpm and temperature profile of 170–210 °C. The PEG 4000 was used for the surface modification of AgFAP particles to provide their uniform distribution in the HDPE phase. The mass fraction of AgFAP in the composite was 1%. The extruded strands were pelletized.
The phase composition and crystalline structure of synthesized samples was studied by X-ray diffractometry, XRD (Bruker D8 Advance Diffractometer), using Ni filtered Cu Kα1,2 radiation. Diffraction data were recorded in a 2θ range from 10° to 70° with a scanning step size of 0.05°.
The IR spectra of synthesized samples were run on a Nicolet 6700 FTIR spectrometer (ATR technique) in the range 400–4000 cm-1.
The morphology of the AgFAP particles and their dispersion in AgFAP/PE composite samples was analyzed using a Philips EM 208, Transmission Electron Microscope (TEM).
Antibacterial properties of the HDPE/AgFAP composite were investigated on ATCC strain *S. aureus* 6538 according to the International standard method ISO 22196:2007(E). The covered surface area of tested specimens was 400 mm2 with 100μL of bacterial inoculum containing 8 x 105 cells/mL in 1/500 nutrition broth during 24 h at 37 °C. Plastic copolymer without Ag+ doped fluoroapatite was taken as a control. Antibacterial activity (R) was calculated using equation: R =Ut-At, where Ut is the logarithm of the number of viable bacteria (in cells/cm2) recovered from the control test specimen after 24 h, and At is the logarithm of the number of viable bacteria recovered from the treated test specimens I or II after 24 h. The obtained R value for specimens I and II was 0.08 and 0.51, respectively.

RESULTS AND DISCUSSION

The high density polyethylene (HDPE) has a wide variety of applications in food packaging because of the many qualities such as a good heat and cold resistance, high rigidity, good resistance to stress cracking and easy fabrication and recycling [5]. Fluorapatite (FAP; $Ca_{10}(PO_4)_6F_2$) was recognized as a possible biomaterial for bone repair due to its biocompatibility, no toxicity and potential antibacterial activity [6]. The XRD patterns of AgFAP, HDPE and HDPE/AgFAP are presented in Fig. 1. The positions of X-ray diffraction peaks of AgFAP sample are in accordance with (Card 15-0876) data for pure fluorapatite phase. The diffraction peaks were sharp and well resolved, indicating the obtained wellcrystallized fluorapatite. The XRD pattern of HDPE and composite sample contains two characteristic crystalline peaks at 2θ of 21.7° and 24.15° assigned to 110 and 200 spacing, and an amorphous peak at 2θ of 19.55°. The pattern of the composite also contains two additional characteristic peaks from the AgFAP at 2θ of 31.8° and 32.6°.

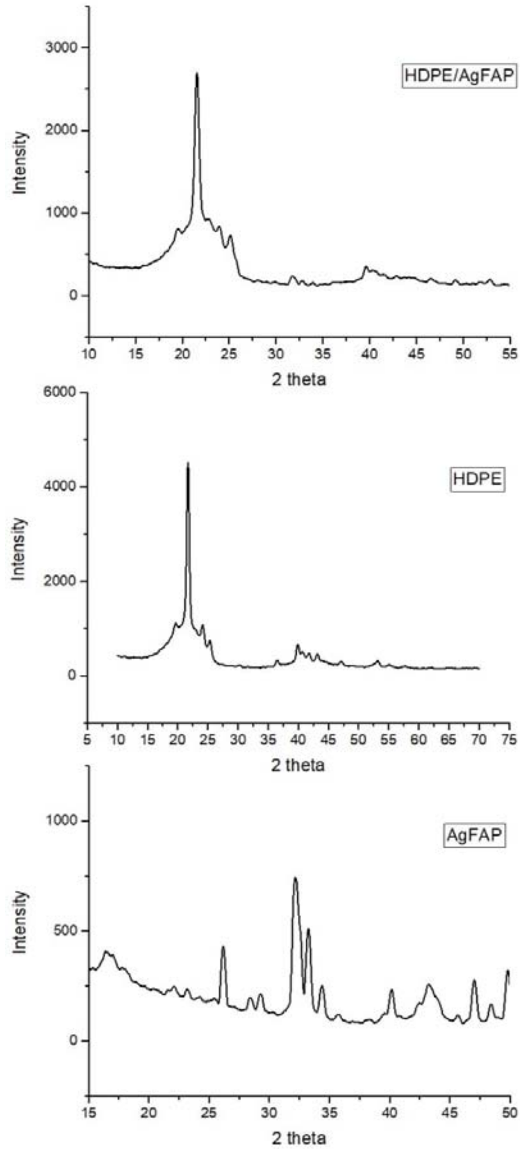


Figure 1. XRD patterns of the AgFAP, HDPE and HDPE/AgFAP composite.

The composite micrograph (Fig.2.) shows that AgFAP particles were not homogeneously distributed in the HDPE polymer. The large agglomerations, small groups, and free particles could be seen. The agglomerates were almost evenly distributed in the polymer matrix. The particles of AgFAP in agglomerates were generally separated one from another.

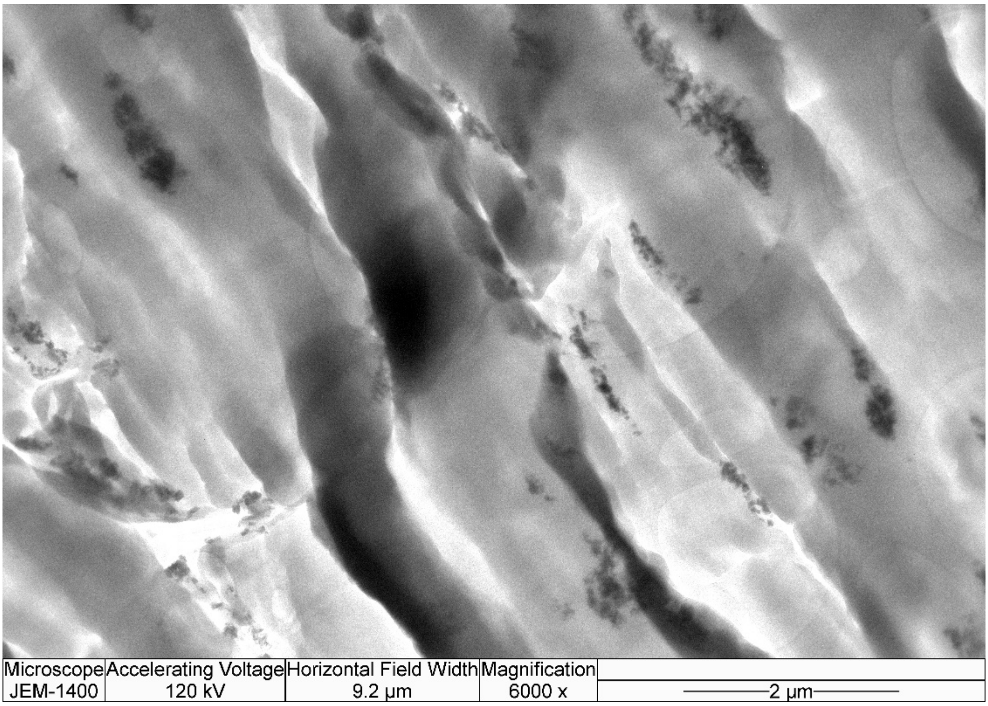


Figure 2. TEM micrograph of the HDPE/AgFAP composite.

Antimicrobial activity of HDPE/AgFAP composite was tested against *S. aureus* that can contaminate food. The obtained R value for HDPE and HDPE/AgFAP specimens was 0.08 and 0.51, respectively. The results showed that the HDPE/Ag/AFAP composite material exhibited an excellent antibacterial activity against *S. aureus*. Several *in vitro* studies reported that silver ions in the apatite materials play an important role in preventing bacterial infections [7, 8].

CONCLUSION

New active antimicrobial polymer composites are very promising in the development of packaging materials. In the present work, the HDPE/AgFAP composite was prepared by simple extrusion process. The presence of AgFAP nanoparticles in the HDPE polymer matrix was confirmed by XRD, FTIR and TEM analysis. Antimicrobial study has demonstrated that HDPE/AgFAP composite exhibits bactericidal effect against *S. aureus*. The HDPE/AgFAP composite is promising as active food packaging material.

Acknowledgement

This work was supported by the Ministry of Education, Science and Technological Development of the Republic of Serbia (Project No. III 43009).

REFERENCES

[1] P. Appendinia, J. H. Hotchkiss, Innovative Food Science & Emerging Technologies, 2002, **3**, 113–126.
[2] S. Quintavalla, L. Vicini, Meat Science, 2002, **62**, 373–380.
[3] P. Suppakul, J. Miltz, K. Sonneveld, S.W. Bigger, Journal of Food Science, 2003, **68**, 408-420.
[4] R. Shrestha,, D. R. Joshi, J. Gopali, S. Piya, Nepal Journal of Science and Technology, 2009, **10**, 189–193.
[5] C. Silvestre, D. Duraccio, S. Cimmino, Progress in Polymer Science, 2011, **36**, 1766–1782.
[6] V. Stanić, S. Dimitrijević, D. G. Antonović, B. M Jokić, S. P. Zec, S. T. Tanasković, S. Raičević, Applied Surface Science, 2014, **290,** 346–352.
[7] V. Stanić, A. S. Radosavljević-Mihajlović, V. Živković-Radovanović, B. Nastasijević, M. Marinović-Cincović, J. P. Marković, M. D. Budimir, Applied Surface Science, 2015, **337**, 72–80.
[8] V. Stanić, D. Janaćković, S. Dimitrijević, S. B. Tanasković, M. Mitrić, M. S. Pavlović, A. Krstić, D. Jovanović, S. Raičević, Applied Surface Science, 2011, **257**, 4510-4518.