CHARACTERIZATION OF RECYCLED RESINS WITH AMINOPHOSPHONIC GROUPS FOR A FUTURE ANTIMICROBIAL TEST

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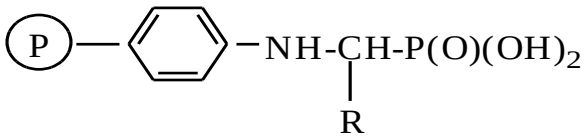
Abstract
This article describes analysis of resins with functional group (by type aminophosphonic groups) recuperated from a biological environment for the use in a new study of antimicrobial test. Their stability was confirmed by Fourier transform infrared spectroscopy, energy-dispersive X-ray analysis and scanning electron microscopy.

Introduction

Bacteria show a significant role in our life. They are dispersed in soil, air and water. They may cause numerous health related problems. For prevent the infection of bacteria some materials were employed as antimicrobial agents. One of the remarkable substances is aminophosphonic acid. It has an important role in biological system [1]. The compounds of aminophosphonic acid can be use as enzyme inhibitors, potential antibiotics and pharmacological agents [1].
In the past decade, the field of macromolecular systems with antimicrobial properties has intensive development including the chemical modification of known polymers with bioactive groups by polymer-analogous reactions [2-5].
This study concerns the characterization of recycled resins with aminophosphonic groups (see figure 1) [6] for using in a new antimicrobial test. The efficacy of the aminophosphonic pendant groups grafted on styrene-6.7%divinylbenzene copolymers was investigated by Fourier transform infrared spectroscopy, energy-dispersive X-ray analysis and scanning electron.



Where: R= $-C_6H_5$ (1Ba-Bz), $-C_2H_6$ (2Ba-Pr); P- styrene-6.7%divinylbenzene copolymers

Figure 1. Aminophosphonic pendant groups grafted on styrene-6.7%divinylbenzene copolymers

Experimental Part

Instruments

Fourier transform infrared spectroscopy (FTIR) using a JASCO-FT/IR-4200 spectrophotometer. Energy-dispersive X-ray analysis (EDAX) and scanning electron microscopy (SEM) were carried out using a Quanta FEG Microscope equipped with EDAX ZAF quantifier.

Method of work

All six samples (1Ba-Bz (1), 2Ba-Pr (1), (1Ba-Bz (2), 2Ba-Pr (2) and (1Ba-Bz (3), 2Ba-Pr (3) were recovered from antibacterial solutions where they were tested against one specie of Gram-positive bacteria (*Staphylococcus aureus*) (code: 1-*S. aureus*) and one specie of Gram-negative bacteria (*Pseudomonas aeruginosa*) (code: 2-*P. aeruginosa*) and a specie of yeast (*Candida albicans*) (code: 3-*C. albicans*). Then, the samples were filtrated, autoclaved at 1 atm pressure and temperature of 120 °C for 30 minutes. These recuperated samples were characterized by FTIR, SEM and EDAX for confirming the presence of active aminophosphonic groups' pendant.

Results and discussion

All the aminophosphonic acid groups grafted on styrene-6.7%divinylbenzene copolymer were analyzed after recovery by FTIR analysis. The absorption bands of samples (see table 1) are presented in the region 3300-3500, 1200-1250 and 1000-1100 cm^{-1} for –NH, P=O and P-OH respectively [6].

Table 1. FTIR spectra for the aminophosphonic acid groups grafted on styrene-6.7%divinylbenzene copolymer.

	1-*S. aureus*		2-*P. aeruginosa*		3-*C. albicans*	
	1Ba-Bz (1)	2Ba-Pr (1)	1Ba-Bz (2)	2Ba-Pr (2)	1Ba-Bz (3)	2Ba-Pr (3)
-NH	3413.39	3358.43	3406.64	3411.46	3415.31	3423.03
P=O	1213.97	1212.04	1213.97	1212.04	1213.97	1209.14
P-OH	1076.08	1072.23	1073.19	1072.19	1075.12	1171.54

In figure 2 is presented EDAX image of 2Ba-Pr (2) after recover from the test with *P. aeruginosa*. It confirm the presence of chemical elements as P, N, Cl, O and C in the of aminophosphonic groups grafted on copolymer

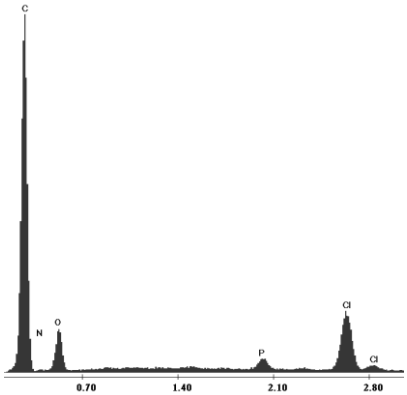


Figure 2. EDAX image of 2Ba-Pr (2) after recover from the test with *P. aeruginosa*

Elemental analysis of all six samples (1Ba-Bz (1), 2Ba-Pr (1), (1Ba-Bz (2), 2Ba-Pr (2) and (1Ba-Bz (3), 2Ba-Pr (3), are shown in Table 2. Composition of these chemical elements confirm the possibility of reuse of these polymers in a new antimicrobial test.

Table 2. Composition of aminophosphonic groups grafted on copolymer after recover them determined by elemental analysis

Elem. / Wt%	1-*S. aureus*		2-*P. aeruginosa*		3-*C. albicans*	
	1Ba-Bz (1)	2Ba-Pr (1)	1Ba-Bz (2)	2Ba-Pr (2)	1Ba-Bz (3)	2Ba-Pr (3)
C	80.02	75.80	80.39	75.57	75.03	69.07
N	4.26	7.03	5.10	2.01	7.17	9.37
O	15.13	16.86	13.82	21.15	17.60	21.38
P	0.22	0.09	0.25	0.29	0.10	0.05
Cl	0.36	0.22	0.43	0.97	0.10	0.14

Determination of the FTIR spectra and energy-dispersive X-ray analysis (EDAX) after the first cycle of antibacterial activity confirmed that the aminophosphonic acid groups can be used as the active groups in a new the antibacterial test.

The morphology of the resins grafted with aminophosphonic pendant groups can be directly visualized by SEM images (Figure 2 and 3)

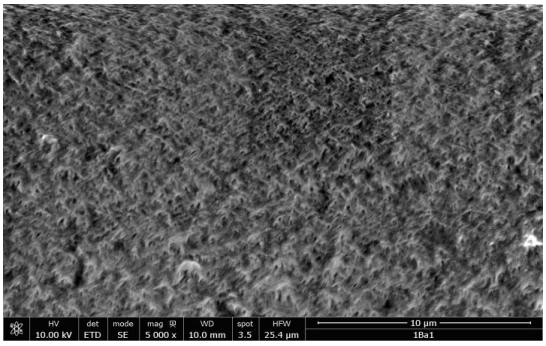


Figure 2. SEM image 1Ba-Bz (1) after recover from the test with *S. aureus*

The surface of the 1Ba-Bz (1) (see figure 2) is rough with interconnected micro-particles and many micro-pores.

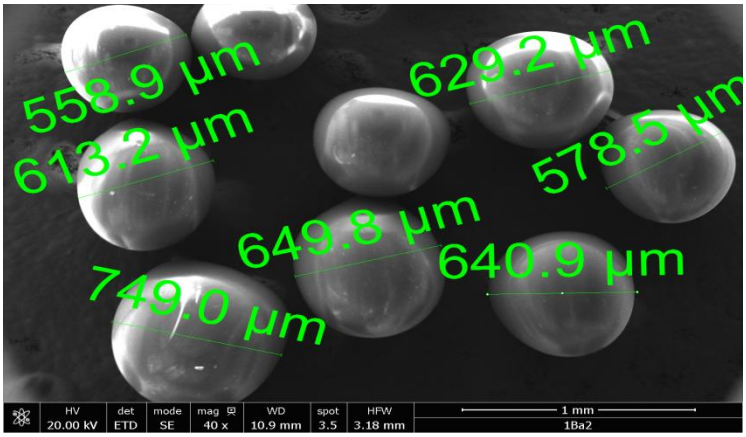


Figure 3. SEM image 1Ba-Bz (2) after recover from the test with *P. aeruginosa*

The beads (see figure 3) are approximately spherical with an average diameter that there is a range from 570 to ~ 750 µm.
The functional groups by type aminophosphonic acid on the bead surface are stable, and antibacterial properties are possible in a repeated application using the same beads.

Conclusion

The investigation of the functional aminophosphonic groups grafted on copolymer styrene-6.7% divinylbenzene recovered from a biological environment confirms the stability of their functional groups.
This is the reasons to recommend their application for a new antimicrobial test.

References

1. Ch. Mohan, B. Hari Babu, C. Naga Raju, R. Usha Nagalakshmi, E-Journal of Chemistry, 2008, 5(4), 679-687.
2. L. Timofeeva, N. Kleshcheva, Appl Microbiol Biotechnol, 2011, 89, 475–492.
3. I. Nichita, A. Popa, E.S. Dragan, S. Iliescu, G. Ilia, Journal of Biomaterials Science, Polymer Edition, 2015, 26(8), 483–496.
4. A. Popa, C.M. Davidescu, R. Trif, G. Ilia, S. Iliescu, G. Dehelean, Reactive and Functional Polymers, 2003, 55(2), 151-158.
5. I. Nichita , A. Popa , R.V. Gros, S. Iliescu, M. Stoia , M. Seres, A. Morar, Proceedings of 22nd International Symposium on Analytical and Environmental Problems, Szeged, Hungary, 10 October, 2016, 243-246, ISBN 978-963-306-507-5.
6. I. Nichita, L. Lupa, M. Stoia , E.S. Dragan, A. Popa, Polymer Bulletin, 2019, 76, 4539–4557, https://doi.org/10.1007/s00289-018-2613-6.

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