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**SARADNJA ISTRAŽIVAČA RAZLIČITIH  
STRUKA NA PODRUČJU KOROZIJE  
I ZAŠTITE MATERIJALA**

## **KNJIGA RADOVA**

**COOPERATION OF RESEARCHES  
OF DIFFERENT BRANCHES IN THE FIELD  
OF MATERIALS PROTECTION**

## **PROCEEDINGS**

*Pod pokroviteljstvom*  
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# ION-ACTIVATED WATER SOLUTION CONTAINING FLAME RETARDANT FOR PLASMA AIDED TECHNOLOGY OF FIRE PROTECTION AND SAFETY

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## ABSTRACT

The aim of this paper relates to a method for plasma chemical surface modifying of materials and items, and specifically for plasma aided impregnation with ion-activated water solutions containing fire retardants to make porous materials. The subject matter is solved by a new method of plasma chemical surface modification of porous materials and workpieces to provide fire spread and ignition resistance consisting in atmospheric pressure impregnation and further drying at room temperature, a characteristic of which is that before the impregnation their surfaces are submitted to a treatment under cold non-equilibrium plasma of glow (dielectric barrier) electrical discharge at atmospheric pressure that burns under high voltage and industrial frequency.

## INTRODUCTION

Relating the fire protection and safety to wooden materials and construction structures brings up the issue of protecting wood from fire in such a way that it will acquire the properties of non-inflammability and non-combustibility, and will not propagate burning not only during the initial period of the fire, but also during its development and under the conditions of spreading fire. Impregnation is a contemporary technology for fire protection of wood, wooden materials and structures – wooden floors, wooden construction structures, wooden boards, boards of wood particles and the pressed cardboard, corrugated cardboard, paper, which leads to impeding their combustibility and inflammability. Impregnating is conducted mainly by means of solutions containing flame retardants, [1].





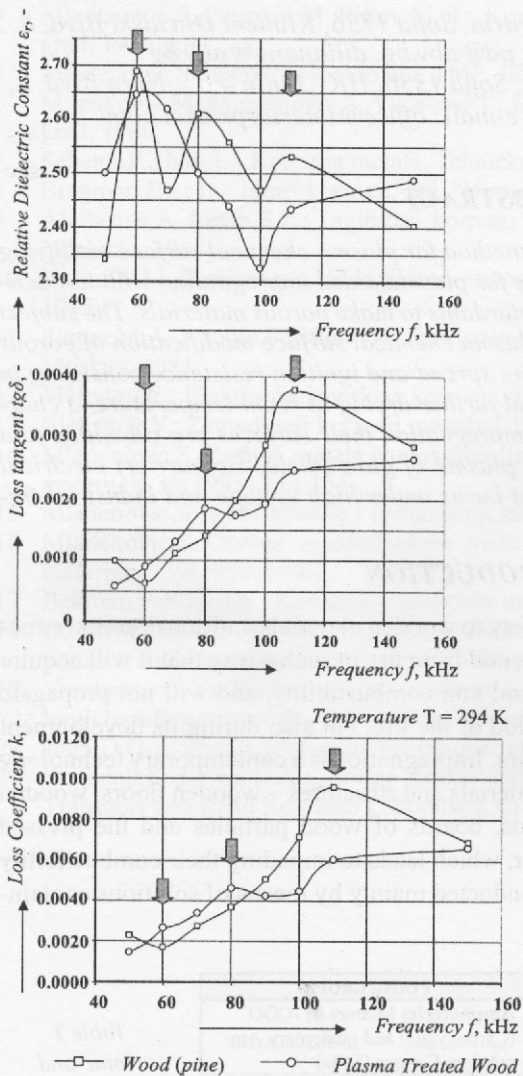
SURFACTANTS		POLAR GROUPS
	Amphoteric	<b>Amphoteric:</b> Mixture of $\text{-COO}^-$ (Carboxylate) and quaternary Ammonium Cations (Salts)
	Anionic	<b>Anionic:</b> $\text{-COO}^-$ (Carboxylate), $\text{-SO}_3^-$ (Sulphonate) and $\text{-SO}_4^{2-}$ (Sulphate)
	Cationic	<b>Cationic:</b> $\text{-NR}_4^+$ - Quaternary Ammonium Cations (Quats); R – the same or different alkyl groups
	Non-Ionic	<b>Non-ionic:</b> $\text{-OH}$ (Alcohol) and $\text{-O-}$ (Ether)
Hydrophilic	Hydrophobic	

Table 1  
Ionic and non-ionic surfactants

In its practice, *Interiorprotect, Ltd.* (Sofia, Bulgaria) makes use of patent-protected water solutions of phosphorus-containing flame retardants with the trade name of HSI-96, which act by being carbonized on the ignited material and by suppressing the pyrolysis and emission of combustible gases, necessary for maintaining the flame burning. Nitrogen-containing additives increase the effectiveness of the phosphorus-containing flame retardants, [1].



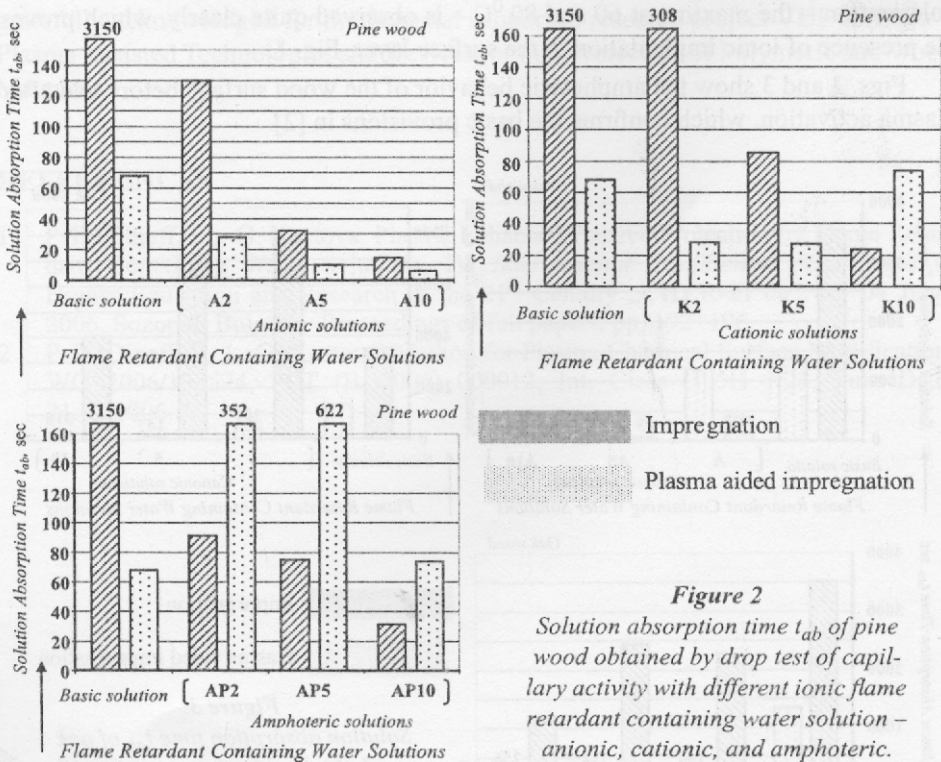
**Figure 1**  
Variation of dielectric properties of pine wood after plasma polarization in dielectric barrier discharge: frequencies of wood response – 60, 80, and 110 kHz. Atmospheric plasma dielectric barrier air discharge activation for 60 sec at 10 kV and industrial frequency.

Plasma aided impregnation technology with ionic (anionic, cationic, and amphoteric) water solutions of phosphorus- and nitrogen-containing flame retardants, [2], which is intended for fire protection of wood and wooden products. This technology has been permitted for implementation in construction after successful tests conducted by an accredited state-owned laboratory in accordance with DIN 4102/1998 (which is similar to EN ISO 11925-2) and BSS 16359-86, [1, 2].

The TASK of the present investigation consists in studying the impact of the acquired (and increased) activity, (chemical and ionic), of the wood surface, exerted directly upon its capillary activity and thence on its susceptibility to impregnation with solutions of flame retardants, which contain ionic surfactants, Fig. 1.

The technology of fire protection through plasma aided capillary impregnation of wood at atmospheric pressure and room temperature involves mandatory preliminary activation of the protected surface in the plasma of a dielectric barrier air discharge, DBD, whereupon impregnation is carried out by applying the solution with the help of a brush, [1].

Long-term investigations of ours, conducted with respect to the plasma activation of various low-energy surfaces (plastics, wood, cotton, wool), have demonstrated that the wood surface is activated by changing its ionic activity as well. The wood surface (cellulose, hemicellulose) is characterized by amphoteric ionic activity. After the plasma-chemical activation, the surface ionic activity remains amphoteric and grows up, but is shifted to the cationic behavior – this is a fact, which we had to take into consideration during the creation of this new technology.



**Figure 2**  
 Solution absorption time  $t_{ab}$  of pine wood obtained by drop test of capillary activity with different ionic flame retardant containing water solution – anionic, cationic, and amphoteric.

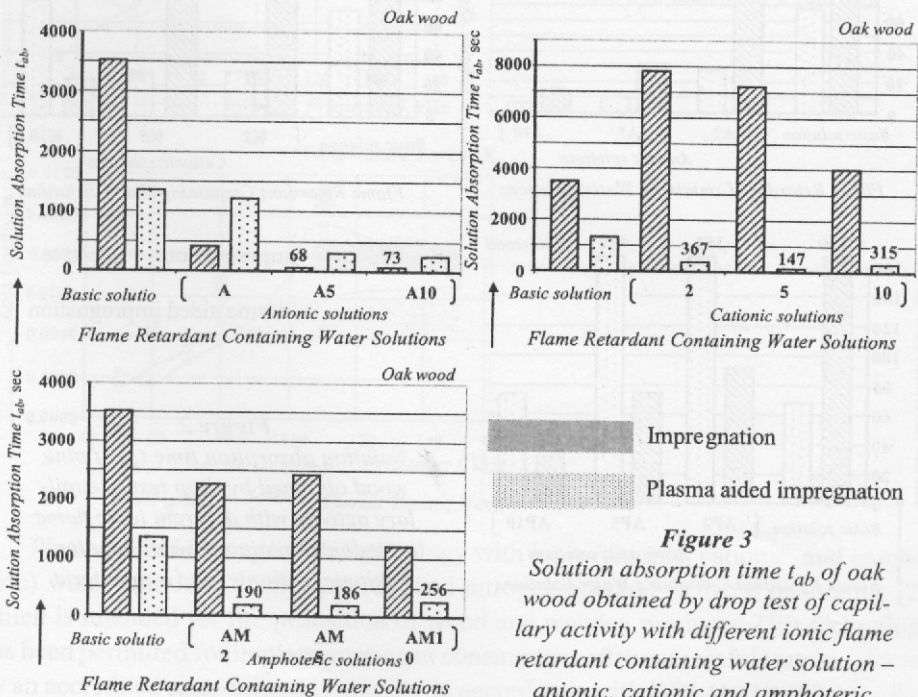
## EXPERIMENTAL RESULTS AND DISCUSSION

Revealing the surface ionic activity is achieved by means of dyeing 0.8-mass-% test solutions: a cationic solution of methylene blue -  $C_{16}H_{18}N_3ClS$  or 3,7-bis(Dimethylamino)-phenazathionium chloride tetramethylthionine chloride, and an anionic solution of methyl orange -  $C_{14}H_{15}N_3O_3S$  or *p*-dimethylamino-azobenzenesulfonic acid, [2].

Three ionic correcting solutions with patent-protected compositions, added in a proportion of 2, 5 and 10 vol. % to the basic solutions (HSI-96), are used in this investigation, as well as for revealing the ionic activity of the wood surface before and after plasma-chemical activation.

The increased surface ionic activity is due to: *first*, the chemical changes and the occurrence of polar groups [1], and *second*, the injection of molecular and atomic ions into the surface layer, which leads to the so-called interstructural polarization and the occurrence of a surface electrical charge. Proofs for the changes occurring in the electrical polarization of wood (pine wood) after plasma activation are recorded by means of the frequency relationships between the tangent and the angle of dielectric losses, relative dielectric permeability, and factor of losses. The increase in the low-frequency polarization – the maxima at 60 and 80 °C – is observed quite clearly, which proves the presence of ionic implantation in the surface layer, Fig. 1.

Figs. 2 and 3 show the amphoteric behavior of the wood surface before and after plasma activation, which confirms the basic provisions in [2].



**Figure 3**  
 Solution absorption time  $t_{ab}$  of oak wood obtained by drop test of capillary activity with different ionic flame retardant containing water solution – anionic, cationic and amphoteric.



## CONCLUSION

Through the ionic solutions used it has been demonstrated experimentally that the wood surface is of amphoteric ionic character, which is not only amplified under plasma chemical activation in DBD-discharge, but also shifted to anionic or cationic behavior depending on the type of wood.

The new technology created, which combines the plasma aided impregnation with using ion-activated solutions, turns out to be a good possibility for matching the acquired ionic activity with the ionic activity of the fire-protecting solution. There is a wood material (pine wood), for which the plasma activation ensures a good effect for the practice, but there is also a wood material (oak wood), for which the new technology is mandatory. That is why the so created approach to the plasma aided impregnation is perceived as a universal means for managing the impregnation susceptibility of wood and wooden materials.

## ACKNOWLEDGEMENT

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- 2 P. D. Dineff, P., L. G. Kostova. Method for Plasma Chemical Surface Modification. WO 2006/133524, PCT (BG2006) 000012, Int. Class H05H 1/24, Pub. Date: 21.12.2006.