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ПЛАЗМЕНО-ПОДПОМОГНАТА КАПИЛЯРНА ИМПРЕГНАЦИЯ ЗА ОГНЕЗАЩИТА НА ДЪРВО ЧРЕЗ ЗАБАВИТЕЛИ НА ГОРЕНЕТО I. ПОДПОМОГНАТА С ПОВЪРХНОСТНО АКТИВНИ ВЕЩЕСТВА КАПИЛЯРНА ИМПРЕГНАЦИЯ

Петър Динев, Ивайло Иванов, Диляна Господинова

Резюме: Плазмено-подпомогнатата огнезащита на дърво и дървени изделия чрез забавители на горенето е осъществена като резултат от появата и развитието на плазмено-подпомогнатата капилярна импрегнация. Предшестващото капилярната импрегнация плазмено химично активизиране на повърхността променя съществено електрическата, химичната и капилярната активност на порестата повърхност, което от своя страна е причина за подобряване на основни характеристики на импрегнационния процес. Използван е капков метод, за да се разкрие влиянието на водни анионни ПАВ върху плазмено-подпомогнатата капилярна импрегнация на три вида тропическа дървесина - мексикански бял кедър (*Cupressus Lusitanica*), махагон (*Swietenia macrophylla*) и тцалам (*Lysiloma Bahamensis*).

Ключови думи: диелектричен бариерен разряд (ДБР) във въздух, плазмено подпомогнатата капилярна импрегнация (ППКИ), фосфор и азот-съдържащи забавители на горенето, водни йонни и нейонни ПАВ, тропическа дървесина.

PLASMA-AIDED CAPILLARY IMPREGNATION FOR FLAME RETARDANCY OF WOOD I. SURFACTANTS-ASSISTED CAPILLARY IMPREGNATION

Peter Dineff, Ivaylo Ivanov, Dilyana Gospodinova

Abstract: The plasma aided flame retardation of wood and wooden products has been conceived and developed as a result of plasma aided process of capillary impregnation. The plasma-chemical surface pre-treatment in air at atmospheric pressure substantially alters its electrical, chemical and capillary activity, thus improving some basic characteristics of the impregnation process, such as penetration depth, solution spreading and adsorption speed, adsorbed solution capacity. Sessile drop technique has been used to reveal the impact of an anionic aqueous surfactant on plasma-aided capillary impregnation of three rain-forest woods - Mexican white cedar (*Cupressus Lusitanica*), Mahogany Caoba (*Swietenia macrophylla*), and Tzalam (*Lysiloma Bahamensis*).

Keywords: dielectric barrier air discharge (DBD), plasma-aided capillary impregnation (PACI), phosphor and nitrogen containing flame retardants, aqueous ionic and non-ionic surfactants, rain-forest woods.

1. INTRODUCTION

Wood is a preferred engineering material. It is low in processing energy, economical, renewable, strong, healthful, and aesthetically pleasing. It has however several disadvantageous properties such as impregnability, ignitability, combustibility, biodegradability, and large scale dimension changing with varying moisture content. These properties of wood are all the result of chemical reaction involving processing and degradative environmental agents, [6].

Wood is a three-dimensional biopolymer composite composed mostly of cellulose, hemicellulose, and lignin. These polymers make up the cell wall and are responsible for most of the physical and chemical properties of wood and wooden products. For example, wood burns primarily because the cellulose and hemicellulose polymers undergo pyrolytic and oxidative reactions with increasing temperature, and giving off flammable gases. The lignin, being more thermally stable, contributes more to char formation than does cellulose and hemicellulose. Increased char formation reduces flammable gas formation and helps insulate wood in depth from further thermal degradation. The high molecular weight of cellulose is primarily responsible for wood strength, which decreases as cellulose degrades thermally by pyrolytic mechanisms, [2, 6].

It is well known that heat treatment (drying for example) and machining reduces the chemical activity and wood wettability (spreading, penetration) by modifying its water-reactive matrix in different ways. Flame retardant chemicals have been added to wood to improve the fire performance of wood through reducing the amount of flammable gases and increasing the amount of char. These water soluble chemicals have been traditionally impregnated inorganic salts, [2, 3, and 4].

2. PLASMA-AIDED CAPILLARY IMPREGNATION AND FLAME RETARDANCY

Earlier, it was found that the cold plasma pre-treatment of hard woods like cherry and oak improves such technological characteristics as impregnation solution spreading and flame retardant absorption (penetration) speed and quantity. The plasma-aided flame retardation of wood has been developed as a result of a plasma-aided process of capillary impregnation that comprises the surface plasma pre-treatment for alteration of chemical activity of wood surface as well as its electrical (ionic) and capillary activities, and in general to improve the capillary impregnation process. The plasma-chemical surface pre-treatment has modified significantly the ionic and chemical activity of wood surface as well as its capillary activity. As a result of that the capillary impregnation process was also improved, [2, 3, and 4].

The cold plasma pre-treatment of wood, improves water solution spreading and penetration (or wicking) speed, as well as a specific amount of the adsorbed or penetrated flame retardant. In this way, the plasma pre-treatment of wood and wooden products improves its flame retardation, [1, 2, 3, and 4].

The plasma-aided flame retardation of wood has been developed as a result of a new plasma-aided process of capillary impregnation that comprises [1]:

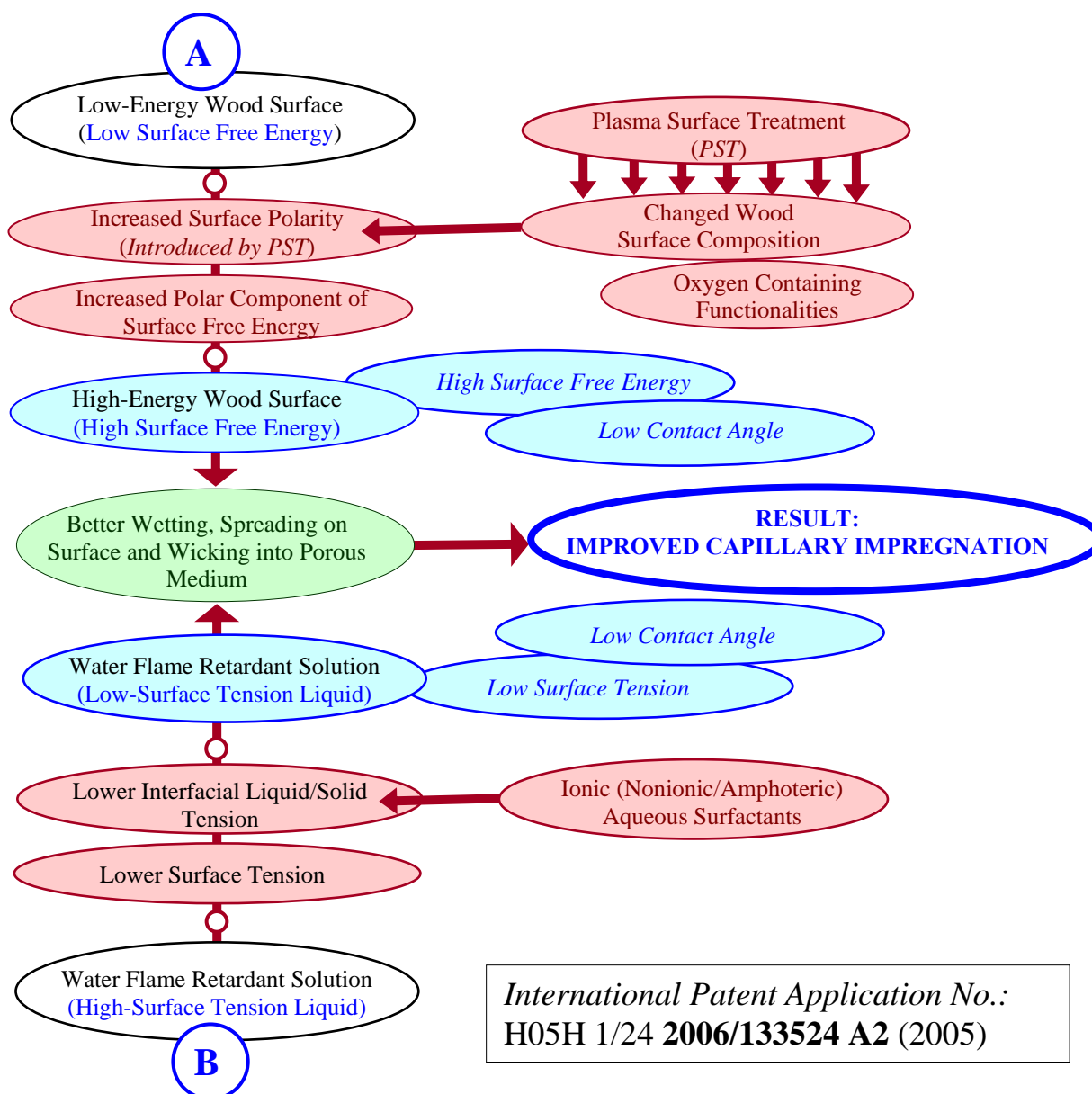


Fig.1. Two ways (**A**, **B**) for improvement of capillary impregnation: **A** - plasma-chemical surface pre-treatment (*DBD* in air): the response of wood surface on plasma pre-treatment is complex but it appears to be controlled by its surface composition, especially by the introduced oxygen containing functionalities and increased surface polarity; **B** - using impregnating (flame-retardant) water solution modified by aqueous (or micelle-forming) surfactants; and the third or integral way **A+B** - plasma-aided and surfactants assisted capillary impregnation (*Dineff*, 2005).

- i* - surface plasma pre-treatment for alteration of chemical, electrical (ionic) and capillary activities of wood surface as well as its surface energy;
- ii* - general change of ionic activity and surface tension of flame retardant (*FR*) containing water solution by ionic and non-ionic aqueous surface-active agents (surfactants), and in general to improve some characteristics of the capillary impregnation process such as solution spreading and wicking speed, as well as specific amount of the penetrated (sorbed) flame retardant. In this way, the plasma pre-treatment of wood improves wooden flame retardation, [1, 2, 3, and 4].

3. CAPILLARY IMPREGNATION AND WETTING PHENOMENA

The wetting theory, expressed in terms of thermodynamic wetting parameters, such as the contact angle, the surface tension, and the surface free energy, is the most widely

used approach in impregnation and adhesion science at present, and this work considers only this type of capillary impregnation phenomena, also referred here as *wetting phenomena*.

Surface energy analysis helps define and illustrate the impact of the plasma-chemical surface activation on plasma-aided capillary impregnation. This activation significantly decreases the contact angle within the range of 10÷15 deg and increases considerably the polar component of surface free energy, [1].

The change of contact angles in time describes the processes at the liquid/solid and vapour/liquid boundaries of the drop during the time after its setting on the wood surface i.e. during the spreading and wicking processes, Fig.2, [6].

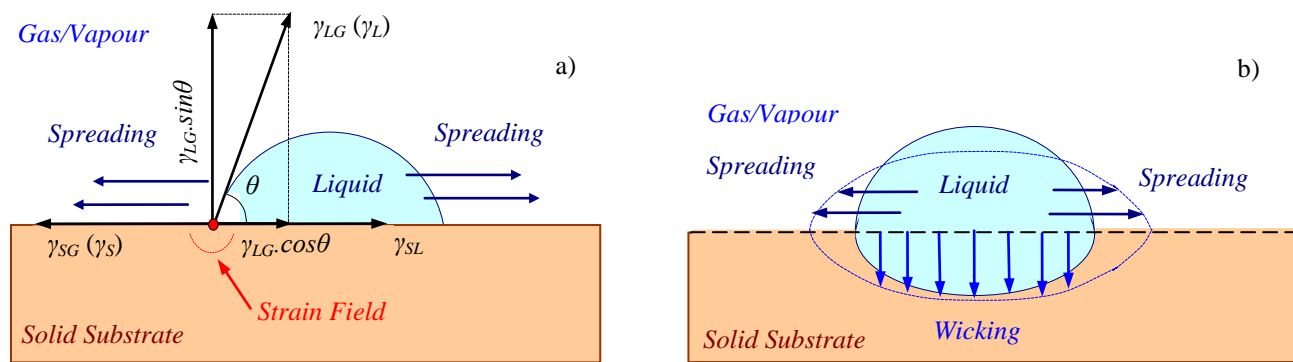


Fig. 2. Schematic illustration of *Young-Bikerman-Good* dynamic model of wetting phenomena on: **a** - smooth, non-porous and rigid surface; **b** - rough, porous, heterogeneous, or hygroscopic surface. Wetting does not include evaporation of liquid, dissolution or swelling of the solid by the liquid or any kind of chemical reaction between the liquid and solid substrate that changes the system's composition.

A liquid drop was placed on a smooth, non-porous, and rigid solid substrate, both exposed to a gas/vapour, and if this system is not in equilibrium and the liquid "wets out" the solid substrate then the liquid exhibits a contact angle of zero against the solid i.e. so if $\gamma_{SG} > \gamma_{SL} + \gamma_{LG}$, then $\cos\theta = 1$ and $\sin\theta = 0$ ($\theta = 0$) and $\gamma_{LG} \sin\theta = 0$ (Good, 1993).

The wetting phenomena on a real surface can be involved by: *i* - spreading of the liquid over a solid surface; *ii* - wicking (or capillary rising) of the liquid into a porous solid (such as wood) (Berg, 1993).

A boundary is not formed instantaneously but requires some time before a dynamic equilibrium is established. This is why a drop volume which is too high should not be selected for measuring retreating angles, as otherwise the contact angle will be measured at a boundary which has not been completely formed. However, it should also not be too slow as the time effects mentioned above will then again play a role. In practice a drop volume between 5 and 15 μl (our choice is 14÷15 μl ; 1 $\mu\text{l} = 1 \text{ mm}^3$) can be recommended; higher flow rates should only be used for the simulation of dynamic processes.

Since wood surfaces are porous, rough and not perfectly smooth, the sessile drop method requires some type of video capture in order to measure the contact angle which changes as the droplet is absorbed.

The way in which different surfactants interact with solid substrates has recently been clarified with the help of atomic force microscopy, [5], and these results provide basis for an explanation of the different spreading properties of the aqueous surfactants

(Venzmer and Wilkovski, 1998). When micelle-forming surfactants adsorb on a hydrophobic substrate, hemi-micelles are formed. This arrangement forces hydrophilic head-groups into contact with the hydrophobic substrate, an orientation less than ideal for lowering interfacial tension, Fig.3.

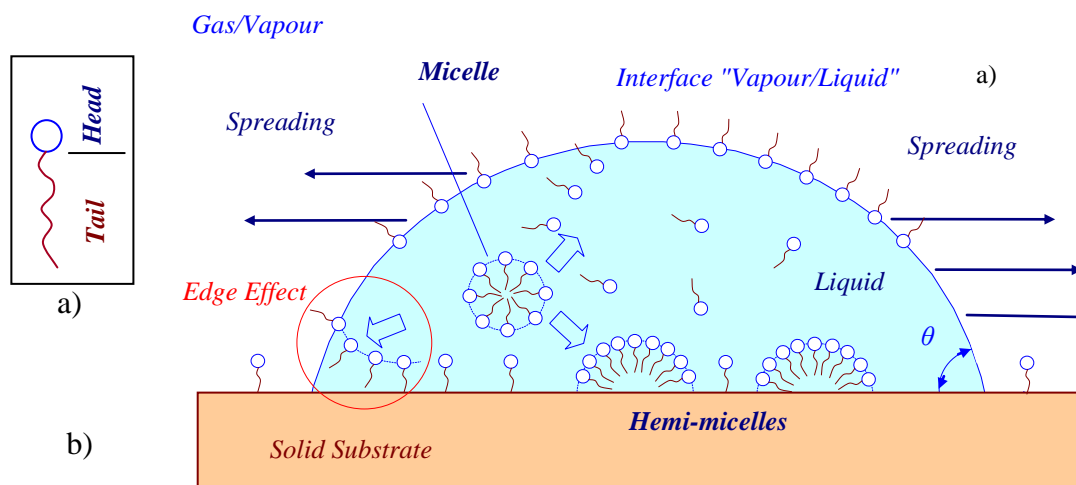


Fig.3. Schematic illustration (b) of *micelle-forming aqueous surfactant* interaction on liquid/solid and vapour/liquid interfaces (see Manne and Gaub, 1995).

The "head" of the surfactant molecule (a) is the hydrophilic or "water loving" part, also called lipophobic or "oil hating". The "tail" of the molecule (a) is the hydrophobic or "water hating" part, also called lipophilic or "oil loving".

Micelle-forming surfactants (b) provide a system with some area with local high concentration of surfactant molecules (hemi- micelles) on the spreading interface and lower interfacial tension (see Venzmer and Wilkowski, 1998).

The objective of this paper was to study the effect of plasma chemical surface pre-treatment of dielectric barrier discharge (DBD) in air (oxidative atmosphere) at atmospheric pressure and room temperature, industrial frequency (50 Hz) and 18 kV (RMS) voltage, on the wood surface wettability monitored by one of the basic thermodynamic wetting parameters - the dynamic (mean) contact angle and its evolution. Therefore, we will focus mainly on the evolution of plasma-aided wetting phenomena in time (1 hour and 24 hours) after plasma-chemical pre-treatment.

4. EXPERIMENTAL INVESTIGATION

A new flame retardant product based on ortho-phosphorous acid, urea and ammonia has been produced and studied. This *phosphorus and nitrogen containing flame retardant* has been used in this experimental study as 30 wt. % water solution. The impregnating flame retardant water solution (*PhFRIS*) was based on it: dry substance of 30 wt.%; phosphorus content of about 13 wt. %, pH = 7÷8 and density of 1.15÷1.14 g/cm³.

Some experimental results on time-depending change of retreating (mean) contact angle θ of three probe liquids - basic water impregnating solution of phosphor and nitrogen containing flame retardant (*PhFRIS*); *PhFRIS* with 5 vol. % anionic aqueous phosphate surfactant (-AS5), are presented here: *i* - plasma-aided capillary impregnation for wood flame retardancy improvement; *ii* - new phosphorous and nitrogen containing flame retardant impregnation solution for plasma-aided retardation; *iii* - condi-

tioning of the applied impregnating solution with anionic surfactant. Our screening experimental studies directed us to use an anionic surfactant - “Aniticrystallin A”, Chimatech, Ltd., Bulgaria, in quantity of 5 vol. %.

Three species of Mexican rain-forest heartwood were investigated: *i* - Tzalam (*Lysioloma bahamensis*); *ii* - Caoba Mahogany (*Swietenia macrophylla*); and *iii* - Mexican White Cedar (*Cupressus Lusitanica*).

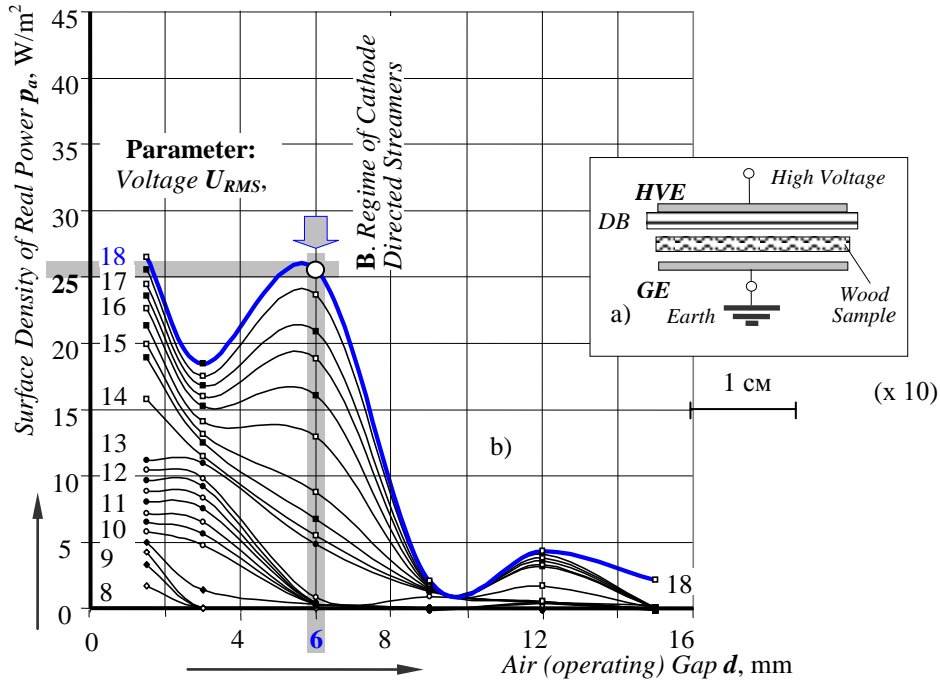


Fig.4. Dielectric barrier air discharge (DBD) in asymmetric coplanar electrode system with one (alkali glass) dielectric barrier (a), technological discharge characteristic " $p_a - U_{RMS}$ ", pick, and choose regime (b) of plasma pre-treatment at industrial frequency (50 Hz) and 25.4 kV (peak value).

On the basis of prior art, as well as on our own former experience in plasma-aided impregnation an oxidative (nitrogen oxides, NO_x) surface plasma pre-treatment has been applied on the test samples for 60 sec in a non-equilibrium cold plasma of dielectric barrier air discharge (DBD) at atmospheric pressure and 18 kV (RMS) or 25.4 kV (PV), [2, 3, and 4].

The DBD-plasma system consisted of coplanar shaped rectangular electrodes with one glass barrier (3 mm thick) closely arranged to the high voltage electrode (HVE), with 6 mm operating gap (OG) between HVE and dielectric barrier (DB), Fig.4a. The DBD was provided by a low frequency (50 Hz) voltage generator. The wood samples were disposed in operating volume and were treated under chosen operational regime (25.4 kV; OG: 6 mm), Fig.5b.

5. EXPERIMENTAL RESULTS AND DISCUSSION

The way in which different surfactants interact with solid substrates has recently been clarified with the help of atomic force microscopy, and these results provide basis for an explanation of their spreading properties (Manne and Gaub, 1995).

When micelle-forming surfactants adsorb on a hydrophobic substrate, hemi-micelles are formed. This arrangement forces hydrophilic head-groups into contact with the hydrophobic substrate, an orientation less than ideal for lowering interfacial tension.

Therefore, the interfacial tension at this interface can be expected to be lower than that without surfactant. In such system containing micelles, these thermo-dynamically stable aggregates are in equilibrium with single surfactant molecules in solution and surfactant monolayer at the vapour/water interface, Fig.3, [5].

The studied *plasma-aided capillary impregnation* was based on both: *i* - plasma pre-treatment of wood sample surface, Fig.4a; *ii* - anionic surfactant assisted impregnation by flame retardant aqueous solution, expecting that an increased capillary activity and impregnating solution penetration speed and capacity would allow good enough flame retardancy, [1].

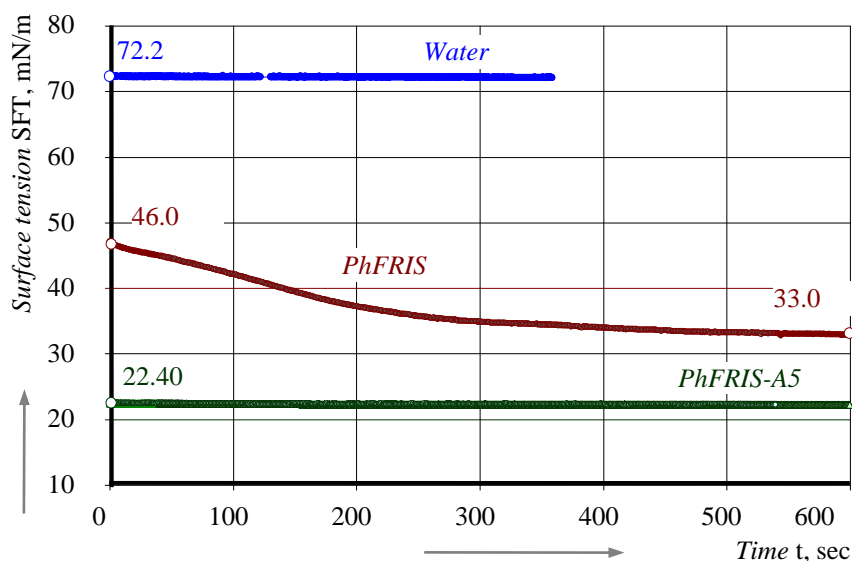


Fig.5. Surface tension time-depending change of a flame retardant (FR) water solution: *PhFRIS* - aqueous impregnating solution of phosphor and nitrogen containing flame retardant; *PhFRIS-A5* - impregnating solution with 5 vol. % anionic aqueous (micelle-forming) surfactant.

6. CONCLUSION

Contact angle analysis helps to define and illustrate the impact of the plasma-chemical surface activation and surfactant assistance on plasma-aided capillary impregnation:

1. The flame retardant aqueous solution (*PhFRIS*) shows a very interesting behavior during the contact angle measurement. There was a transition period during which its surface tension amended from 46.4 to 33.0 mN/m for a time of about 12 minutes. The introduction of anionic aqueous surfactant (*PhFRIS-A5*) leads to both disappearance (less than 10 sec) of the transitional period and significant reduction of surface tension (less than 23 mN/m) - that means a good wetting, spreading, wicking, and chemical affinity, Fig.5.

2. The change of contact angle in a drop test with water after plasma chemical surface treatment is determined by the so-called chemical reorganization of the wood surface in its interaction with the oxygen in air. It is well known that the effect of plasma-chemical functionalization decreases considerably within one day.

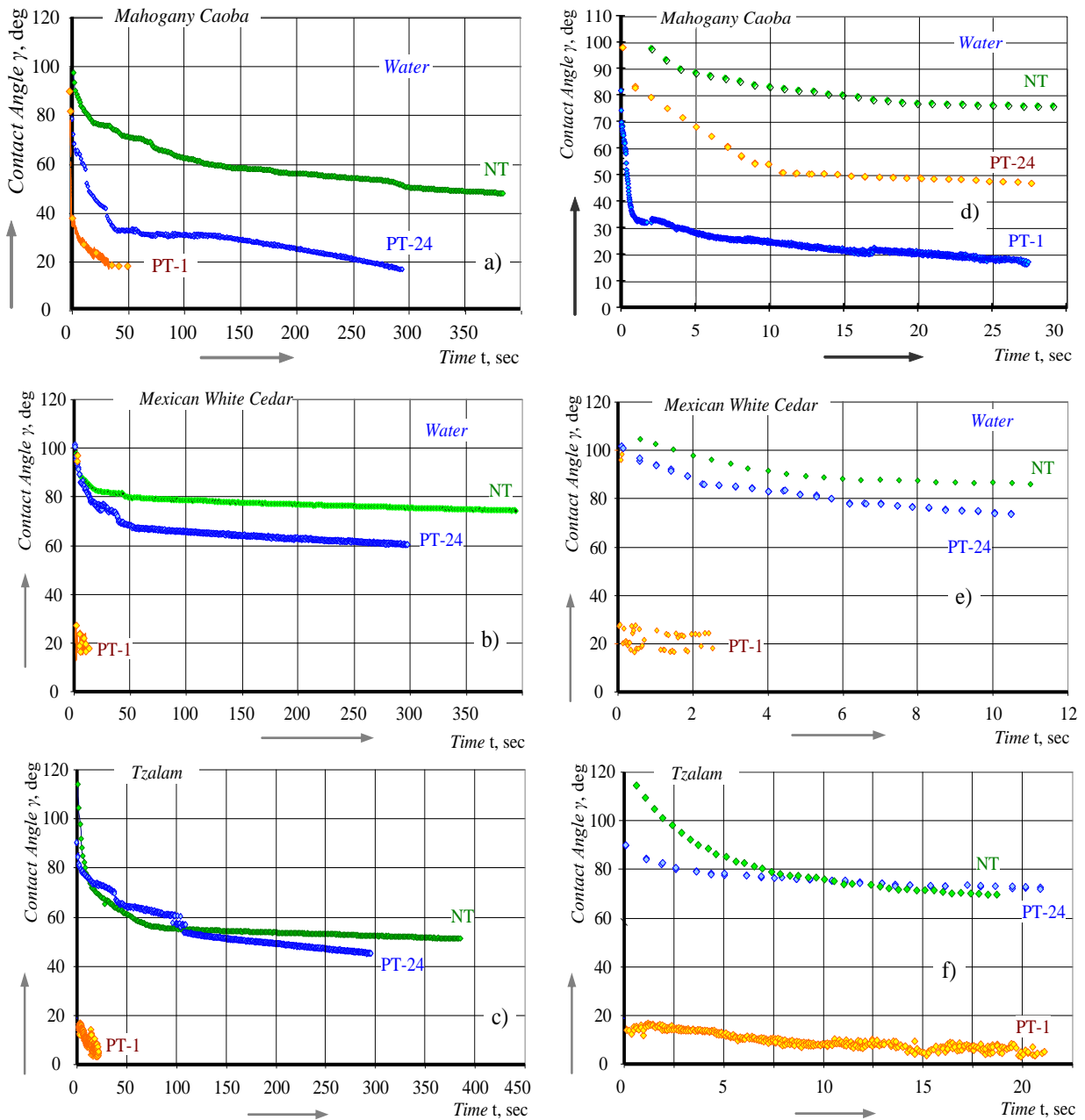


Fig.6. Time-depending change of mean contact angle θ of a water droplet (sessile drop technique), as it advances over a non-ideal surface of three rain-forest heart wood samples (*Mexico, Yucatán*) - plasma surface treated (PT-) or non-treated (NT) in DBD, - after 1 (PT-1) and 24 (PT-24) hours after plasma-chemical treatment: a, b, c - long time control; d, e, f - short time control.

This fact was confirmed by our studies on the plasma surface activation of the three kinds of wood. The effect of plasma-chemical activation disappears about 24 hours for *Tzalam* wood. We can arrange the three rain-forest heartwood samples extent of preserving for 24 hours the effects of the plasma pre-treatment in the following order: i - *Caoba mahogany*; ii - *Mexican white cedar*; and iii - *Tzalam*, Fig.6.

3. The change of mean contact angle determined in a drop test with *PhFRIS* demonstrates, however, quite different reality: about 2 to 5 seconds, the contact angle decreases above 50 deg and it is maintained after 1 hour and 24 hours from the moment of the plasma-chemical surface pre-treatment. Moreover, it is effective in all three quite different wood samples equally. Regardless of the *open time* between plasma pre-treatment and capillary impregnation - 1 or 24 hours, the application of

surfactants provides good wetting and wicking, and good capillary impregnation, Fig.7 and Fig.8.

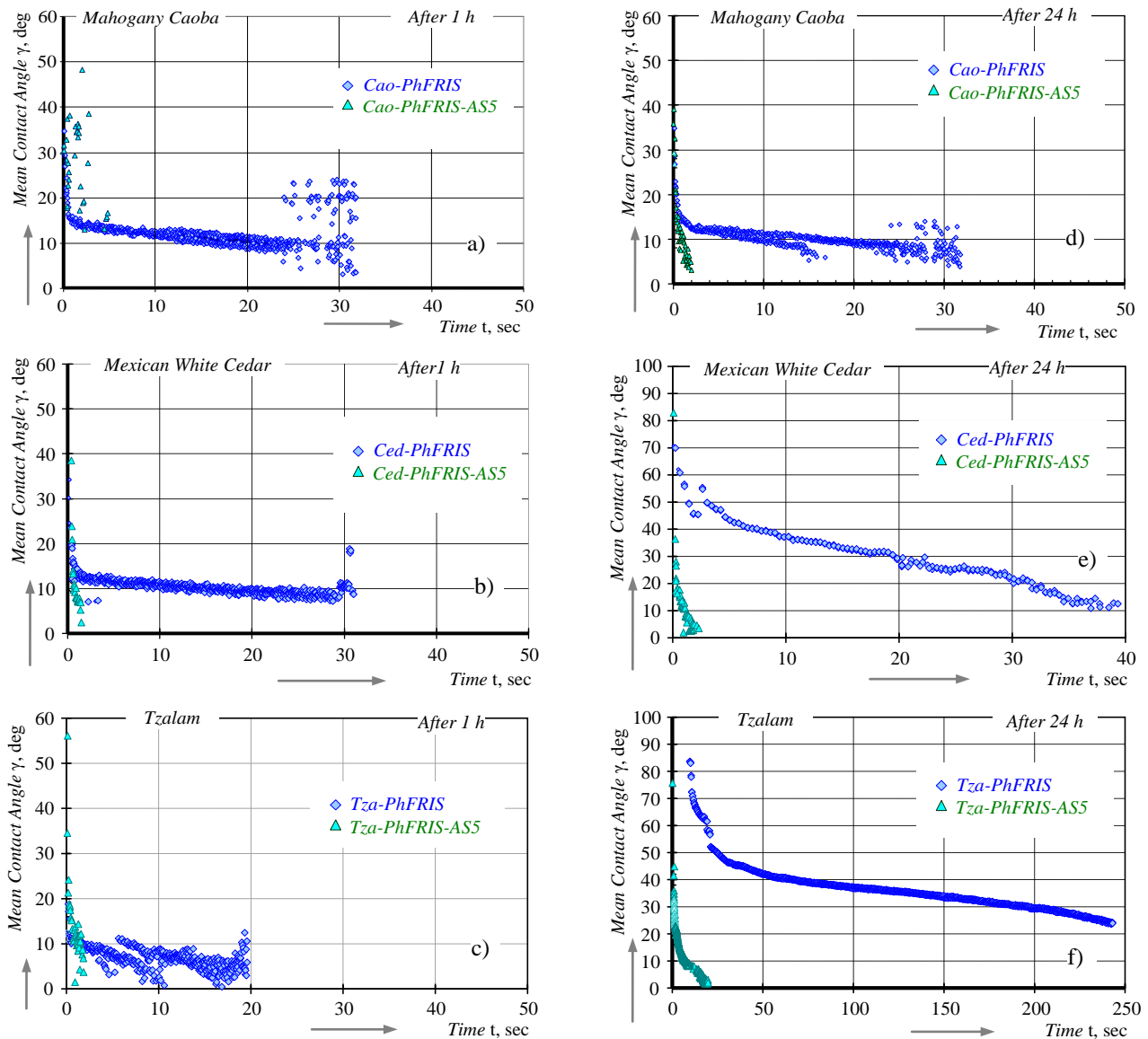


Fig.7. Time-depending change of mean contact angle θ of PhFRIS as it droplet advances slowly over a non-ideal surface of three rain-forest heart wood samples (*Mexico, Yucatán*) 1 hour (a, b, and c) and 24 hours (d, e, and f) after DBD surface treatment in air. The PhFRIS's are: -PhFRIS - basic impregnating FR aqueous solution; -PhFRIS-AS5 - impregnating solution with 5 vol. % anionic aqueous surfactant.

For us, this reality is not surprising as this fact has led to combine back in 2005 plasma-chemical surface pre-treatment of the wood surface using surfactants for lasting change in the surface tension of the impregnation phosphorus containing flame retardants. It is the basis of the resulting patent for plasma-surface activation of wood surfaces - *International Patent Application H05H 1/24 2006/133524 A2 (2005)* [1].

4. Simultaneous use of plasma or DBD surface pre-treatment and micelle-forming surfactants, and in this case an anionic aqueous surfactant, shows one more chance to reduce the surface tension of the impregnation solution and a sustainable use of the positive effect of increased surface energy on wood within 24 hours after plasma surface pre-treatment. The use of aqueous anionic surfactants is admissible only in concentration from 5 to 10 vol. % - it was 5 vol. % for the used surfactant.

Plasma or *DBD*-aided capillary impregnation means both decrease in the surface tension of impregnation solution by using surfactants and increase in the surface energy of the wood surface through plasma surface pre-treatment, Fig.1, [1].

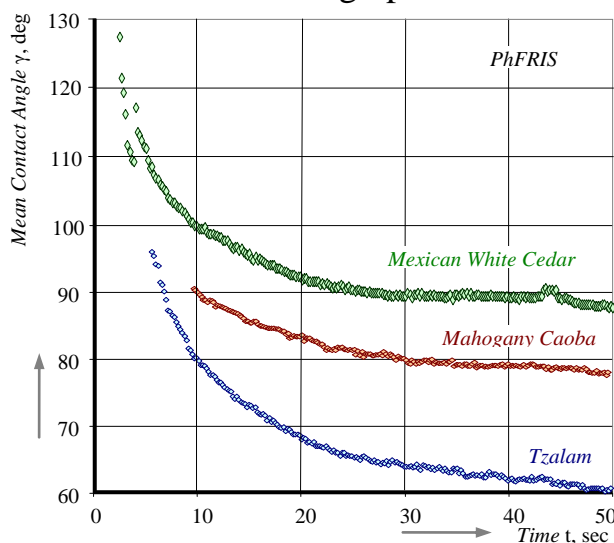


Fig.8. Time-depending change of mean contact angle θ of a phosphor and nitrogen containing flame retardant impregnating solutions (*PhFRIS*) as its droplet advances over a non-ideal surface of three different rain-forest heart wood samples (*Mexico, Yucatán*):
- Mexican white cedar (*Cupressus Lusitanica*)
- Mahogany caoba (*Swietenia macrophylla*);
- Tzalam (*Lysiloma bahamensis*).

5. Contact angle analysis reveals both processes on liquid/solid and vapour/liquid boundaries of the droplet during the time after its setting on the wood surface i.e. during the concurrent spreading and wicking processes determining the result of the capillary impregnation.

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