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Резюме: *Появата и развитието на плазмено-подпомогнатата капилярна импрегнация имат съществен принос към реализирането на плазмено-подпомогнатата огнезащита на дърво и дървени изделия. Използването на плазмено химично активиране преди реализирането на капилярна импрегнация на дадена повърхност променя съществено нейните свойства и характеристики, водейки до подобрение на импрегнационния процес. Целенасоченото намаляване на повърхностното напрежение на водните импрегнационни разтвори чрез внасяне на класически (йонни) в комбинация със силициево-органични повърхностно активни вещества (ПАВ) представлява съществена част от технологията на плазмено-подпомогната капилярна импрегнация. Настоящето изследване е част от задълбочено проучване на плазмено-активираната (функционализирана) повърхност на дървесина и третирането й със забавители на горенето.*
Ключови думи: *диелектричен бариерен разряд (ДБР), плазмено подпомогната капилярна импрегнация (ППКИ), фосфор и азот-съдържащи забавители на горенето, тропическа дървесина, трисилоксан (силикон, силиконов полиетер) ПАВ*

PLASMA-AIDED CAPILLARY IMPREGNATION FOR FLAME RETARDANCY OF WOOD

II. SILICONE SURFACE-ACTIVE AGENT EFFECT

Peter Dineff, Ivaylo Ivanov, Dilyana Gospodinova

Abstract: *The advent and development of plasma-aided capillary impregnation contribute substantially towards the realization of plasma aided flame retardation of wood and wooden products. Using the plasma chemical activation before applying capillary impregnation on a surface substantially alters its properties and characteristics thus improving the impregnation process. The purposeful reduction of the surface tension of aqueous impregnation solutions by introducing classic (ion) in combination with silicon-organic surface-active agents (surfactants) is an essential part of the technology of plasma-aided capillary impregnation. This study has been developed as part of a large investigation on plasma-activated (functionalized) wood surface and flame retardant treated wood.*
Keywords: *dielectric barrier air discharge (DBD), plasma-aided capillary impregnation (PACI), phosphor and nitrogen containing flame retardant, rain-forest wood, trisiloxane (silicone, silicone polyether) surfactant.*

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1. INTRODUCTION

Surfactants play an important role in enhancing an aqueous solution's ability to wet, penetrate and spread on solid surfaces. Surfactants are used also as emulsifiers and dispersants in different formulation to assist with the delivery of chemicals to a spray mixture. The phenomena of wetting, wicking and spreading of a water droplet on porous solid surfaces is frequently encountered in many industrial applications, such as coating, printing and painting [5, 4].

The spontaneous spreading (called *superspreading*, i.e. the rapid coverage of hydrophobic surfaces; *Zhu et al.*, 1994) of aqueous solution of trisiloxane-ethoxylate surfactants (*superspreaders*) has impressive surface properties that promote extensive and rapid spreading over hydrophobic substrates and efficiently reduce the surface tension at the air/solution interface to 21÷22 mN/m. Superspreaders have a variety of commercial and industrial applications, and can be used as adjutants, surface modifiers for fabrics, cleaners and much more [2, 3, 5, and 6].

Trisiloxane-ethoxylate surfactants are characterized by a *critical wetting concentration* (*CWC*). *CWC* is a concentration above which a transition from partial wetting to complete wetting occurs at spreading over moderately hydrophobic surfaces, hence, the *CWC* is associated with the beginning of the superspreading. The knowledge of the *CWCs* is important for many wetting/spreading applications [6].

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 and wood flame retardancy were also improved. The plasma-aided flame retardation of wood has been developed lately as a result of a new plasma-aided process of capillary impregnation assisted by an aqueous anionic (normal, ordinary) surfactant supported in turn by trisiloxane-ethoxylate super-surfactant, [1].

The plasma-aided process of capillary impregnation comprises: *i* - surface plasma pre-treatment for alteration of chemical, electrical (ionic) and capillary activities of wood (porous) surface as well as its polarity and surface energy; *ii* - general change of ionic activity and surface tension of flame retardant (*FR*-) containing water solution by ionic aqueous and trisiloxane-ethoxylate 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 the wooden flame retardation, Fig. 1.

The objective of this paper was to study the effect of a trisiloxane-ethoxylate surfactant (*TES*) on plasma-aided process of capillary impregnation and more precisely on the wood surface wettability monitored by one of the basic thermodynamic wetting parameters - the advancing contact angle and its amendment during the capillary impregnation.

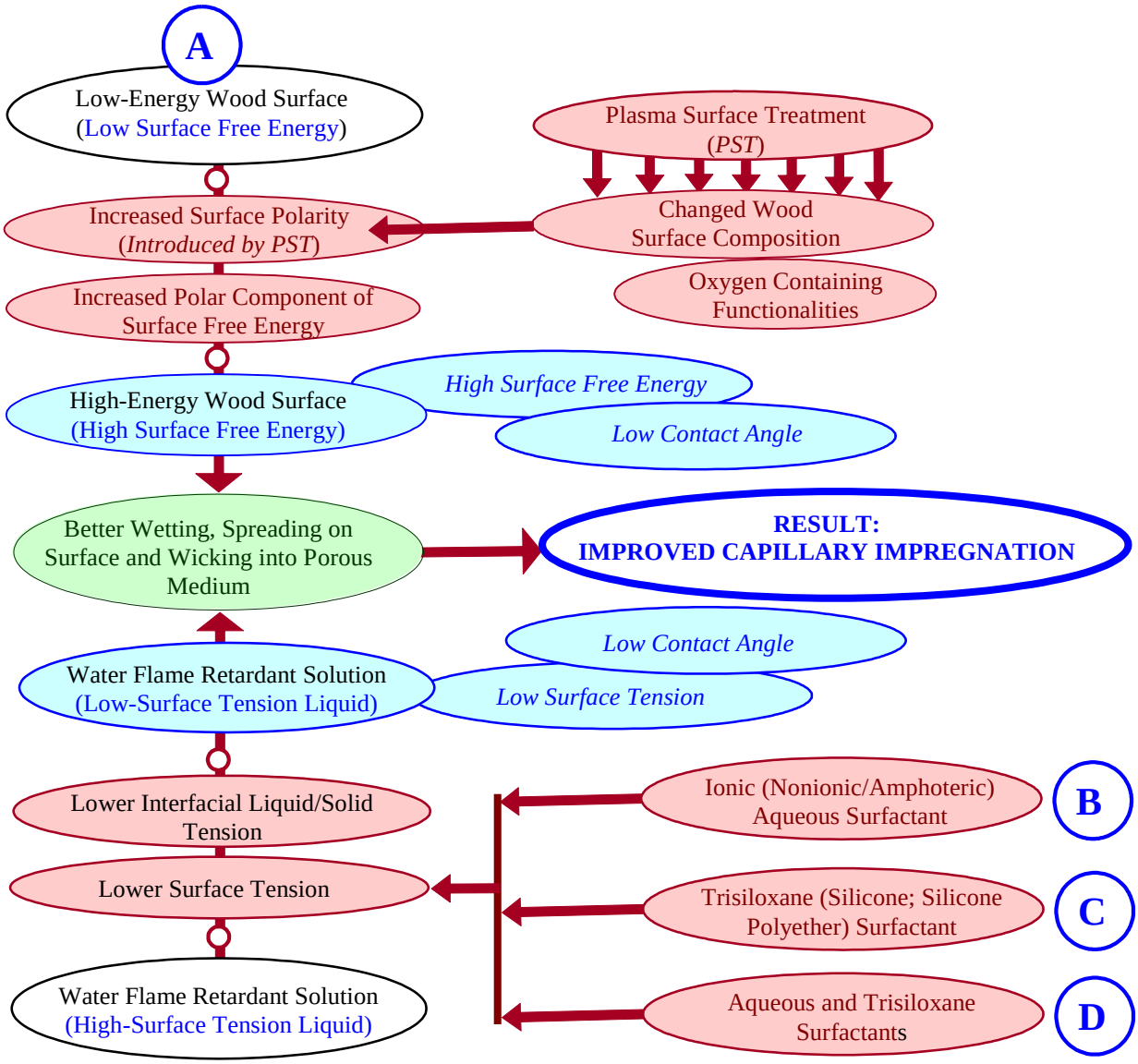

Fig.1. Four ways (***A***, ***A+B, A+C,*** *and* ***A+D***) for improvement of capillary impregnation for flame retardancy of wood: ***A*** - plasma-chemical surface pre-treatment (*DBD* in air): the response of wood surface on plasma pre-treatment is complex but it appears to be controlled by its surface composition, especially by the introduced oxygen containing functionalities and increased surface polarity; ***B*** - plasma-chemical surface pre-treatment followed by the use of an impregnating (flame-retardant) water solution modified by aqueous (or micelle-forming) surfactants (*Dineff*, 2005); ***C*** - plasma-chemical surface pre-treatment and after that applying a siloxane-ethoxylate surfactant; and ***D*** - plasma-chemical surface pre-treatment and after that applying a blend of ionic aqueous (normal) and siloxane-ethoxylate surfactants (*Dineff*, 2010).

Therefore, we will focus our investigation mainly on the plasma-aided wetting phenomena at different "*open time*" (1 hour and 24 hours) elapsed since plasma-chemical surface pre-treatment by a dielectric barrier discharge (*DBD*) in air (oxidative atmosphere) at atmospheric pressure and room temperature, industrial frequency (50 Hz), and 25.4 kV voltage [1, 7].

2. THEORETICAL BASIS OF THE STUDY

The Young's equation (*Young*, 1805) describes the interaction of a liquid droplet on a smooth, non-porous and rigid substrate, Fig. 1:

$$\gamma_S = \gamma_{Sl} + \gamma_l \cos\theta = \gamma_l \left(\cos\theta - 1\right), \qquad (1)$$

where: γ_l is the surface tension of the liquid; γ_S - the surface tension of the solid; γ_{Sl} - the interfacial tension between the liquid and the solid; θ - contact angle of the droplet of liquid on the solid surface.

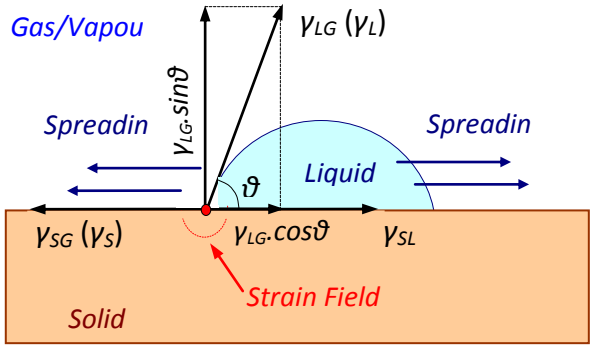

Fig.2. Schematic illustration of *Young-Bikerman-Good* dynamic model of wetting phenomena on smooth, non-porous and rigid surface.

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 composition.

This relation can be expressed in terms of a *spreading coefficient S* (*Ross* and *Becher*, 1992), Fig.2:

$$S = \gamma_S - (\gamma_l + \gamma_{Sl}), \tag{2}$$

where the requirement for complete wetting is a positive spreading coefficient ($S > 0$). Coefficient S measures the difference between the surface free energy γ_S and its value in the case of complete wetting. Notice that $S \leq 0$ and $S = 0$ only in the case of complete wetting, [2].

Therefore, the critical spreading parameter which determines sign and magnitude of the spreading coefficient must be the interfacial tension between the surfactant solution and the substrate γ_{Sl}. This value should be calculated from surface tension γ_S and contact angle θ, and it can not be measured directly. The contact angles in such liquid systems are all very low and do not provide means to differentiate between trisiloxanes. Therefore, this classical approach can not explain the differences in spreading behavior directly, [2].

While the phenomenon of superspreading is easily observed, explanations for this behavior have been more elusive. Over the early years, a number of mechanistic theories on superspreading have been put forth. Early concepts attributed the molecular shape of trisiloxanes, Fig.3, to be critical to superspreading (*Anathapadmanabhan* et al., 1990). The *T-shaped configuration* of the trisiloxane surfactant was described to roll or "*zipper*" at the spreading front of the droplet, Fig.4, [2, 5].

These theories, however, do not explain why only some trisiloxane surfactants exhibit superspreading behavior. Why minor variations in the hydrophilic head group can lead to dramatically reduced spreading.

The ratio of the size of the hydrophobic and the hydrophilic part of the surfactant molecules, Fig.3, can be expressed in terms of a critical packing parameter P (*Israelachvilli* et al., 1976), [2]:

$$P = V/(a_0\, l_C) \tag{3}$$

where V is hydrophilic chain volume; a_0 - interfacial area hydrocarbon-water; l_C - length of hydrophilic chain.

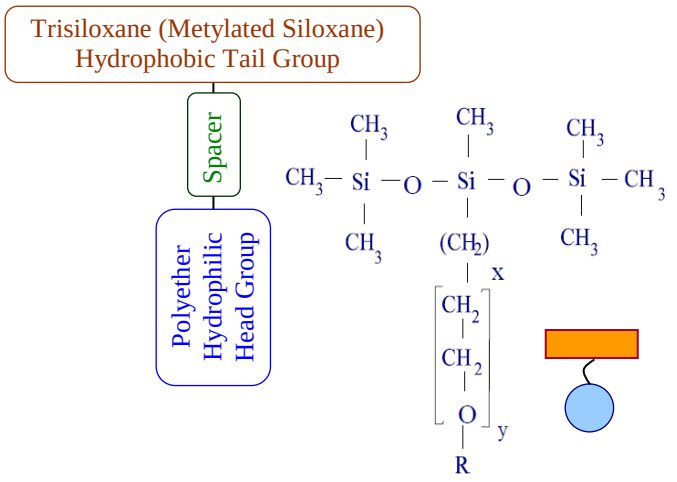

Fig.3. *T-shaped* molecular structure of a non-ionic trisiloxane (silicone polyether, silicone) surfactant (*Knoche*, 1994; *Venzmer* and *Wilkowski*, 1998).

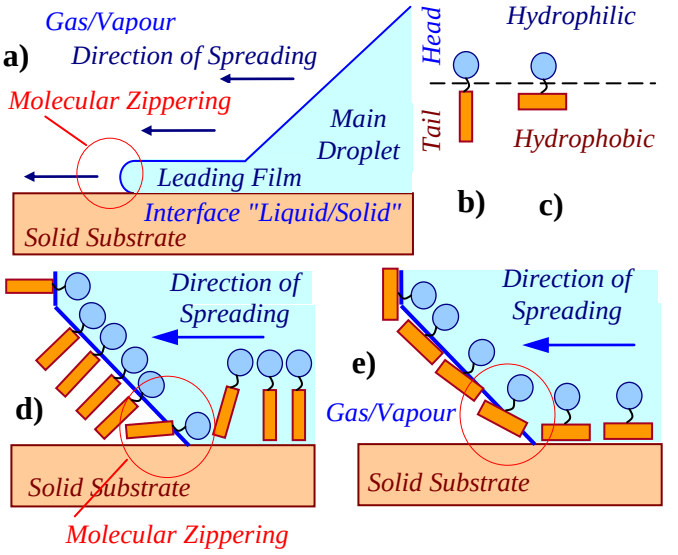

Fig.4. "*Molecular zippering*", zipper impedance effect (*a*, *d*) or the effect of the surfactant large "tail" (*Ananthapadmanabhan*, 1990) upon spreading (*d*). The "head" of the surfactant molecule (*b*, *c*) is the hydrophilic or "water loving" part, also called lipophobic or "oil hating". The "tail" of the molecule (*b*, *c*) is the hydrophobic or "water hating" part, also called lipophilic or "oil loving".

The hydrophobic tail (red) of the micelle-forming surfactants (*b*, *d*) would offer higher resistance to the movement in the direction of spreading. On the contrary, the hydrophobic tail of the bilayer-forming trisiloxane surfactants would offer minimum resistance (*e*) to the movement (*Venzmer* and *Wilkowski*, 1998).

According to this concept, surfactant molecules with a hydrophilic head group larger than the hydrophobic part ($P < 1$) form curved aggregates such as spherical or cylindrical micelles in a solution. When the hydrophobic and hydrophilic parts are of equal size ($P \approx 1$) the spontaneous mean curvature is zero, resulting in the formation of bilayer aggregates - flat lamellae, spherically closed bilayer - vesicle, liposome, or sponge (*Mitchell* et al 1983, *Strey* et al. 1990). When micelle-forming surfactants adsorb on a hydrophobic substrate, hemi-micelles are formed. This arrangement forces hydrophilic head group into contact with the hydrophobic substrate, less than ideal for lowering interfacial tension, Fig.5.

In contrast, when bilayer-forming surfactants with a critical packing parameter of $P \approx 1$ interact with hydrophobic substrate, such curved aggregates can not develop. In this case, only hydrophobic parts of the surfactants are in contact with the surface. The interfacial tension at this interface can be lower than with micelle-forming surfactants. Considering Young's equation, this is a reason for a favorable spreading coefficient in the case of bilayer surfactants, Fig.6.

There is a fundamental difference between the behavior of micelle- and of bilayer-forming surfactants at the air-water interface. Micelles are thermodynamically stable aggregates that are in equilibrium with single surfactant molecules in solution and a surfactant monolayer at air-water interface, Fig.5.

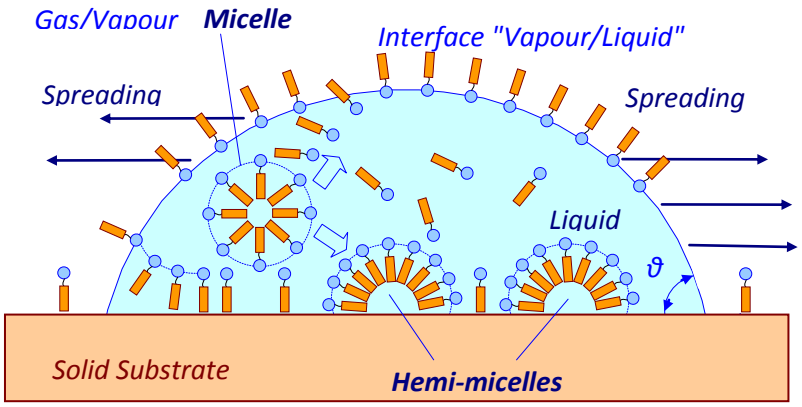

Fig.5. Schematic illustration of ***micelle-forming aqueous surfactant*** interaction on liquid/solid and vapour/liquid interfaces. Micelle-forming surfactants provide a system with some area with local high concentration of surfactant molecules (hemi- micelles) on the spreading interface and lower interfacial tension (*Venzmer* and *Wilkowski*, 1998).

When a vesicle comes in contact with the surface, it spontaneously bursts, deposing large amounts of surfactant at the surface. In this case, bilayer-forming surfactants provide a system with high concentration of surfactant molecules at the spreading interfaces, Fig.6.

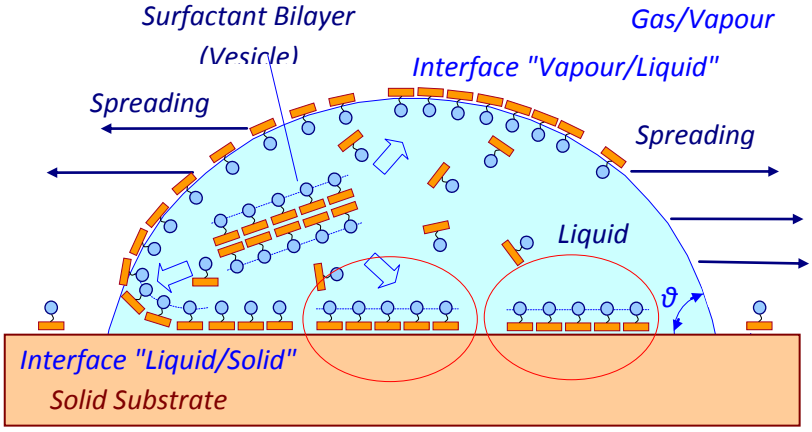

Fig.6. Schematic illustration (*a*) of bilayer-forming (vesicle) trisiloxane surfactants interaction on liquid/solid substrate and vapour/liquid interface. Bilayer-forming surfactants (*a*) provide a system with high concentration of surfactant molecules on the spreading interfaces (*Venzmer* and *Wilkowski*, 1998).

The hydrophobic and hydrophilic parts of the surfactant should be of equal size ($P \approx 1$) enabling the formation of bilayer aggregates and this leads to lower interfacial tension and super-spreading (super-wetting).

Another model (*Nikolov* et al. 2002) of superspreading, Fig. 7, suggests that the spreading of trisiloxane ethoxylate is controlled by a surface tension gradient, which forms when a droplet of surfactant solution is placed on the solid surface. The spreading front stretches, the surfactant concentration becomes lower and the surface tension increases at the front relative to the top of the droplet. A dynamic surface tension gradient is established. The driving force for spreading is due to the *Marangoni effect*. The superspreading behavior of trisiloxane ethoxylates is a consequence of the molecular configuration at the air-liquid surface. The trisiloxane ethoxylate molecule must have small and compact hydrophobic part [3]. In our opinion, there are some main requirements for superspreading effect: *i* - a small and compact trisiloxane hy-

drophobic part is necessary for low surface tension (*Venzmer* et al. 1998; *Nikolov* et al. 2002); *ii* - the hydrophobic and hydrophilic parts of the trisiloxane surfactant should be of equal size ($P \approx 1$) enabling the formation of bilayer aggregates and leading to lower interfacial tension and faster spreading (*Venzmer* et al. 1998); *iii* - trisiloxane surfactants are characterized by a critical wetting concentration (CWC): the CWC is associated with the beginning of the superspreading or the transition from partial wetting to complete wetting occurs at spreading over hydrophobic surfaces, [6]; *iv* - when a trisiloxane surfactant is combined with a conventional adjuvant (co-surfactant) it forms a blend and the "*molecular zippering*" model suggests that the conventional (micelle-forming) surfactant may compete with the trisiloxane surfactant molecules in the liquid-solid interface, (*Murphy* et al. 1991), [5].

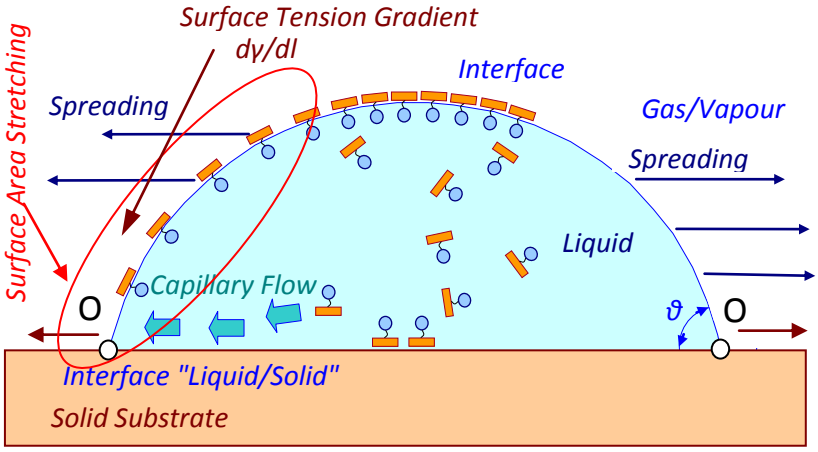

Fig.7. Schematic illustration of the mechanism of spreading driven by surface tension gradient (*Marangony flow* effect) including the drop's surface area stretching at the three-phase contact region (*O*) that causes a decreased local surface concentration of the surfactant and a higher surface tension at the leading edge than in the middle of the drop - the centre part of the drop surface then moves outwards and it drags the liquid layer, thereby increasing the overall spread area (*Nikolov* et al., 2002).

Since wood surfaces are not moderately hydrophobic and ideal - they are porous, rough and not perfectly smooth, the superspreading can not be observed as usual. The surface tension lowering does not always enhance spreading (*Murphy* et al. 1991). Our screening investigations have also shown that the use of trisiloxane surfactants (superspreaders) does not contribute significantly to improve the spreading and penetration of the flame retardant solutions. The effect of superspreaders remains lower than the effect of the used anionic conventional surfactants.

The impact of the plasma-chemical surface activation (functionalization, dielectric polarization) increases considerably the polar component of wood surface free energy. The plasma surface pre-treatment modifies significantly the ionic and chemical activity of wood surface as well as its capillary activity. As a result the capillary impregnation process is improved. The anionic aqueous surfactants play an important role in enhancing the aqueous solution's ability to wet, penetrate, and spread on the wood surface, Fig.1.

Our screening experiments show very good results when a trisiloxane surfactant in CWC (0.1 vol. %) was combined with a conventional anionic surfactant (2, 5, and 10 vol. %), or co-surfactant, to capillary impregnate wood samples with flame retardant aqueous solution, [1].

4. EXPERIMENTAL RESULTS AND DISCUSSION

The studied *plasma-aided capillary impregnation* was based on both:

i - plasma-chemical surface pre-treatment of all wood samples, [7];

ii - surfactants-assisted impregnation by flame retardant aqueous solution, expecting that an increased capillary activity and impregnating solution penetration and capacity would allow good enough flame retardancy, [7].

The non-equilibrium air (oxidative) plasma pre-treatment was combined with anionic aqueous (conventional) surfactant (*AS*, "Anticrystalin A", *Chimatech*, Ltd., Bulgaria) and trisiloxane-ethoxylate surfactant (*S*, Y-17113, *Momentive Performance Materials* GmbH & Co. KG, Germany) assisted capillary impregnation of well known research methods, [7].

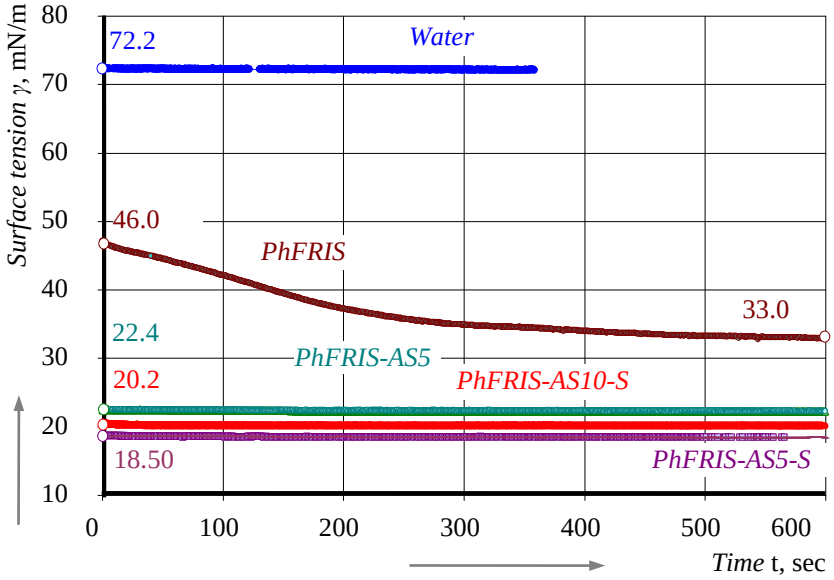

Fig.8. Surface tension γ time-depending change of a flame retardant (*FR*) water solution: *PhFRIS* - aqueous impregnating solution of phosphor and nitrogen containing flame retardant; *PhFRIS-AS5* - impregnating solution with 5 vol. % anionic (micelle-forming) surfactant; *PhFRIS-AS5-S* - water solution with a combination of 5 vol. % anionic surfactant and 0.1 vol. % non-ionic trisiloxane surfactant; *PhFRIS-AS10-S* - water solution with a combination of surfactants containing 10 vol. % anionic surfactant and 0.1 vol. % non-ionic trisiloxane surfactant.

Contact angle and surface tension are two important concepts related to both surfactants. The surface tension is determined by the physicochemical properties of the surfactants. The flame retardant aqueous solution (*PhFRIS*) shows very interesting behavior during the surface tension measurement that includes a transition period of about 12 minutes. The anionic surfactant assistance (*PhFIRS-AS5*) leads to both disappearance of the transitional period and significant reduction of surface tension - less than 23 mN/m. The combination of surfactants (*PhFRIS-AS5-S*) ensures even greater reduction in surface tension - less than 19 mN/m, Fig.8. That means good surfactants assisted wetting, spreading, wicking, and capillary activity, [1].

The contact angle is a result of interaction between liquid droplet and target surface. Some authors reported that a formulation with contact angle from 50 degrees or less is considered as having good wetting capability, while complete wetting is possible if the contact angle is 29 degrees or less (*Zabkiewicz* et al., 1985).

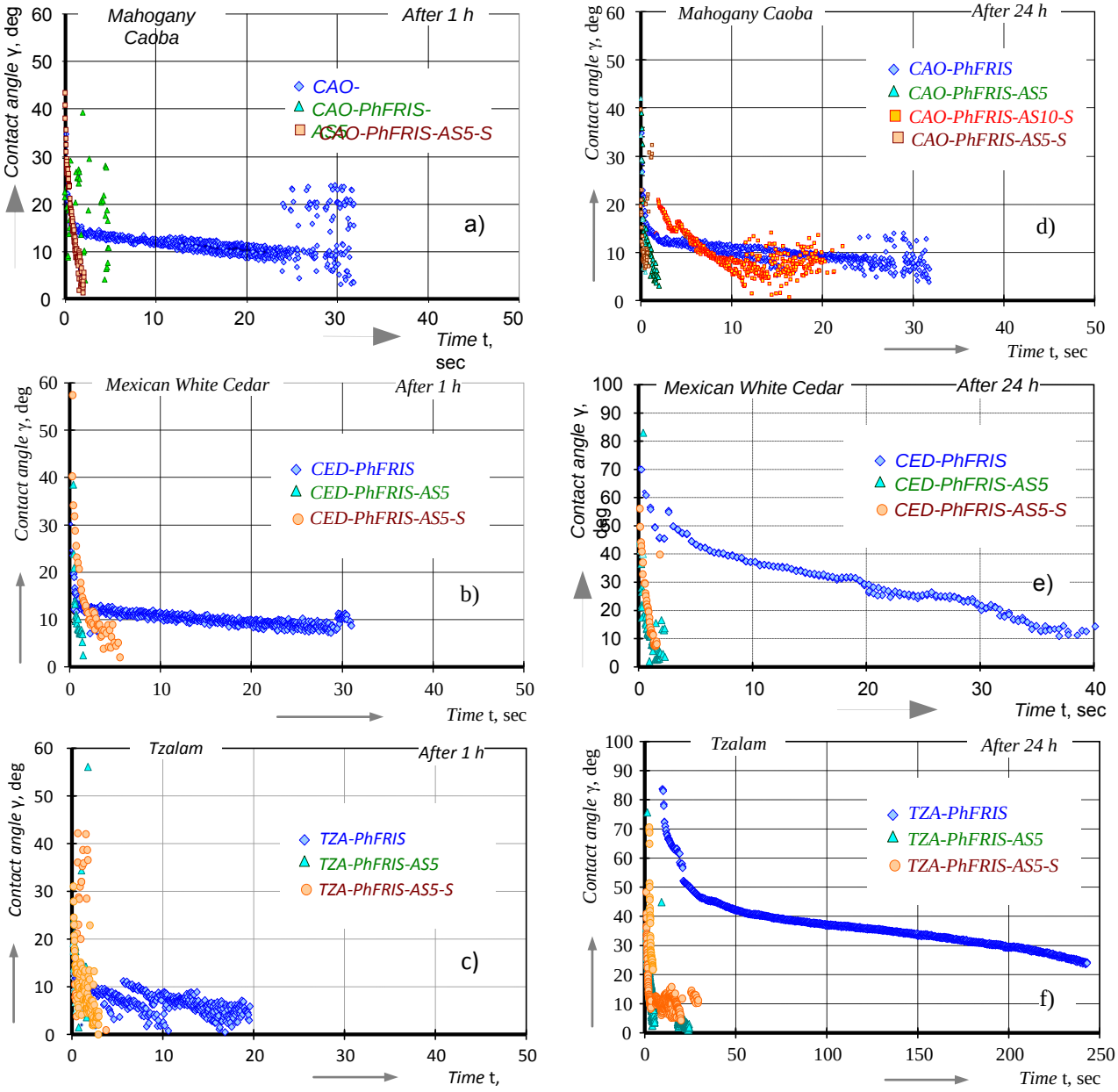

Fig.9. Time-depending change of contact angle θ of a phosphor and nitrogen containing flame retardant impregnating solutions (*PhFRIS*) as its advance slowly over a non-ideal surface of three rain-forest (*Mexico, Yucatán*) heart wood samples in the frame of 1 hour (a, b, and c) and 24 hours (d, e, and f) after *atmospheric dielectric barrier discharge* surface treatment in air.

The three rain-forest woods are: Caoba mahogany *(Swietenia macrophylla)*, Mexican white cedar *(Cupressus Lusitanica)*, Tzalam *(Lysiloma bahamensis)*. The *PhFRIS*'s are: -*PhFRIS* - basic impregnating aqueous solution; -Ph*FRIS-AS5* - impregnating solution with 5 vol. % anionic aqueous surfactant; -Ph*FRIS-AS5-S* - impregnating solution with 5 vol. % anionic aqueous surfactant and 0.1 % non-ionic trisiloxane surfactant; -Ph*FRIS-AS10-S* - impregnating solution with 10 vol. % anionic aqueous surfactant and 0.1 % non-ionic trisiloxane surfactant.

The contact angle change after plasma pre-treatment is determined by both chemical reorganization (by the oxygen from ambient air) and dielectric depolarization of wood. It is well known that the effect of plasma-chemical activation (functionalization) decreases considerably within one day. This fact was confirmed by our studies on the plasma surface activation of the three kinds of wood. The effect of plasma-chemical activation decreases significantly for about 24 hours at *Tzalam* and *Mexican*

white cedar woods. We can arrange the three rain-forest heartwood samples extent of preserving the effects of the plasma pre-treatment in the following order: *i - Caoba mahogany*; *ii - Mexican white cedar*; and *iii - Tzalam*, Fig. 9.

CONCLUSION

Contact angle analysis helps to define and illustrate the impact of the plasma-chemical surface activation and surfactants assistance on plasma-aided capillary impregnation: *i* - combined use of micelle-forming and bilayer-forming surfactants, and in particular an anionic aqueous surfactant in combination with a non-ionic trisiloxane surfactant, shows one more possibility to reduce the surface tension of the impregnation solution and the sustainable use of the positive effect within 24 hours after the cold plasma (*DBD*) surface pre-treatment. The use of trisiloxane surfactants is admissible only in CWC. *ii* - The changed surface composition and increased polarization after plasma or *DBD* pre-treatment constitute the main reason for changing the surface condition of the studied wood specimens. At the same time, they are the reason for the observed change in the contact angle upon aging of the plasma-treated wood surface in air.

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