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Processing Efficiency of Plasma-Aided Porous Media Finishing

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***Abstract*— The efficiency of a plasma-aided finishing process e.g., surface impregnation can be predicted only by setting two basic parameters of the real wood porous surface and the actual impregnating solution – surface free energy and surface tension. Successful finishing can be expected if the process efficiency parameter take a positive value after a specified aging time of the plasma-chemical surface activation – 2 or 24 hours.**

Keywords—processing efficiency, plasma-aided finishing; porous media, surface free energy, surface tension

I. INTRODUCTION

Plasma-aided technique can be used to facilitated the surface finishing rather the surface impregnation of porous media such as wood, wooden and cellulosic materials [1, 2, 3 and 4].

Porous materials with low surface free energy (*SFE*, σ_S) were subjected to plasma-chemical surface activation (functionalization) for increasing its *SFE*. So, this plasma-aided process cannot be applied out of the production line because *SFE* decreases quickly after an aging in ambient air [1].

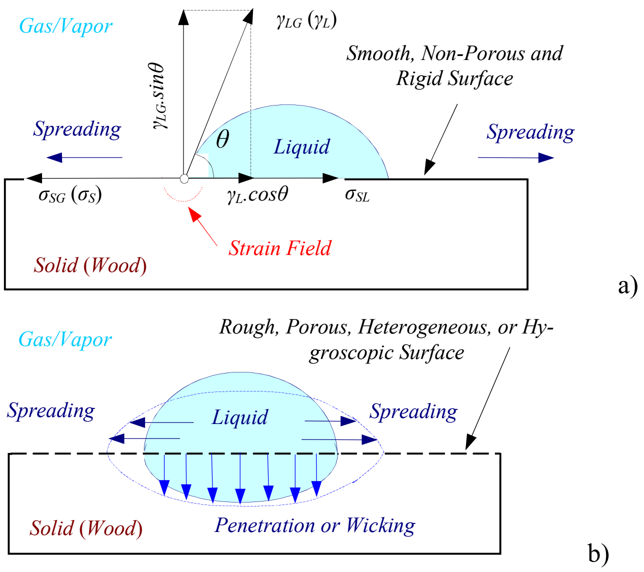

Fig. 1. Schematic illustration of two model of wetting phenomena: ***a*** – the wetting phenomena on an ideal surface when a liquid drop is placed on a smooth, non-porous and rigid solid, both exposed to a gas/vapor, the system will be not in equilibrium and the liquid "wets out" the solid then the liquid exhibits a contact angle of zero against the solid i.e. so if $\sigma_S > \sigma_{SL} + \gamma_L \cos\theta$, then $\cos\theta = 1$ and $\sin\theta = 0$ ($\theta = 0^\circ$), and $\gamma_L \sin\theta = 0$ (*Good,* 1993); ***b*** - the wetting phenomena on a real surface can be involved by: *i* - the spreading of liquid over a solid surface; *ii* - the penetration or wicking of an impregnating liquid into a porous solid (*Berg*, 1993).

II. PARAMETER OF PLASMA ACTIVATION PROCESS EFFICIENCY

Both *SFE* and liquid(-solid) surface tension (*LST*, γ_L) are essential in porous media finishing phenomena [1]. Plasma-chemical surface functionalization technique can be used to enhance surface finishing: materials with low *SFE* were subjected to oxidative plasma pre-activation for increasing it, fig. 2.

Surfactants are compounds that can be used to lower the interfacial tension (*IFE*, σ_{SL}) between a liquid and a solid, fig. 1.

The higher the *SFE* of the solid (wood) substrate, the better the finishing including surface impregnation. The lower the *LST*, the better the result of finishing. In sum the difference between *SFE* and *LST* should be as large and positive: $\sigma_S - \gamma_L > 0$, (or $\sigma_S > \gamma_L$), the effective shall be the surface impregnation [1, 2 and 3].

In general, the following processing efficiency parameter (*PSP*) was found as [1]:

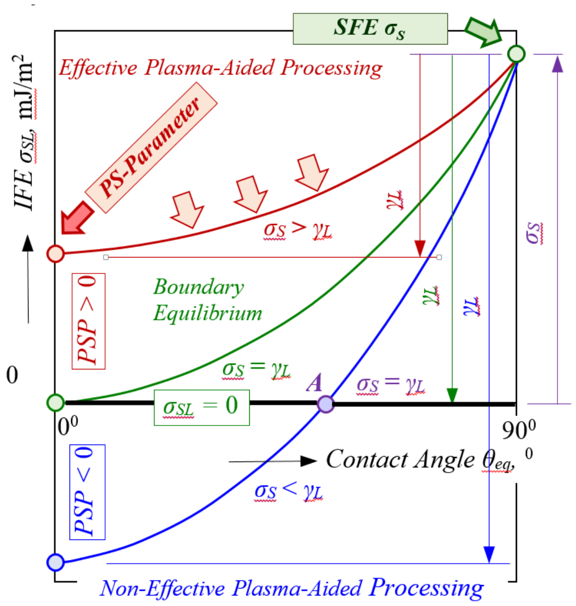

Fig. 2. Penetration-spreading parameter (*PSP* = σ_S - γ_L) of an effective plasma-aided surface impregnation processing at liquid surface free tension γ_L: *PSP* = σ_S - γ_L > 0 ($\sigma_S > \gamma_L$). A non-effective plasma-aided surface impregnation has *PSP* < 0.
Relationship between the "*solid-liquid*" interfacial energy (*IFE*) σ_{SL} and the static contact angle θ_{eq} measured by *Sessile Drop Technique*.
SFE (σ_S) – surface free energy, mJ/m²; *LST* (γ_L) – liquid surface tension, mN/m; *Line of boundary equilibrium* (in green) – a boundary line at *PSP* = σ_S - γ_L = 0 or $\sigma_S = \gamma_L$.

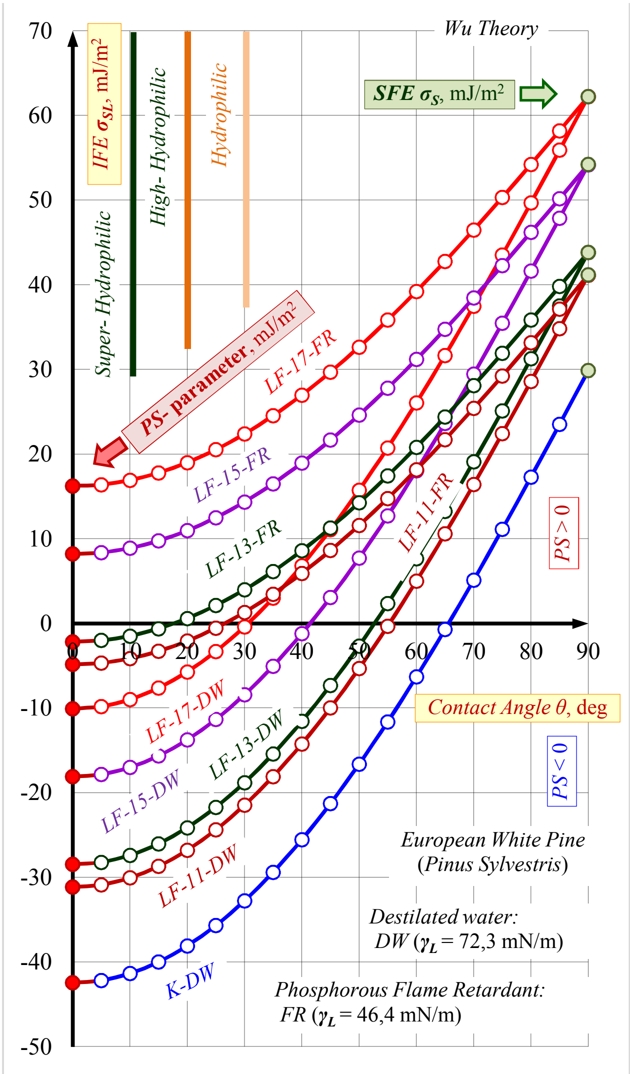

Fig. 3. The use of distilled water (*DW*) as a test liquid does not carry adequate information on the effectiveness of the process of surface impregnation of *European white pine* (*Pinus Sylvestris*, Bulgaria) wood with an aqueous solution of a phosphorus flame retardant (*FR*). The effective mode (*LF-XX-*: *APP*-activation at 11, 13, 15, and 17 kV rms; 50 Hz) selection of plasma-aided wood impregnation can be determined by testing only with the used impregnating solution or the relationship between the "*solid-liquid*" interfacial free energy (*IFE*) σ_{SL} and static contact angle θ_{eq} measured by *Sessile Drop Technique*: $\sigma_{SL} = PSP$ ($\theta_{eq} = 0$). The all *SFE* values were determined for aging 2 hours in the *theory of Wu*.

FR- basic water impregnation liquid containing phosphor- and nitrogen flame retardant *PhFR*; *SFE* (σ_S) – surface free energy, mJ/m²; *SFT* (γ_L) – liquid surface free tension, mN/m; *IFE* (σ_{SL}) – interface "solid-liquid" free energy, mJ/m². *PSP* – penetration-spreading parameter, mJ/m².

"Plasma-aided wood finishing of a porous material will be more successful and this material will be more susceptible as the difference between its SFE σ_S and the LST γ_L or the so-called penetration-spreading parameter is positive: $PSP = (\sigma_S - \gamma_L) > 0$. If not, wetting and finishing (impregnating or penetration, draining and spreading) problems will be occur."

Plasma-aided surface impregnation process will be more successful and the material will be more susceptible as the $PSP > 0$ and better – $PSP >> 0$. This is the essence of the rule for obtaining effective plasma-enhanced impregnation process.

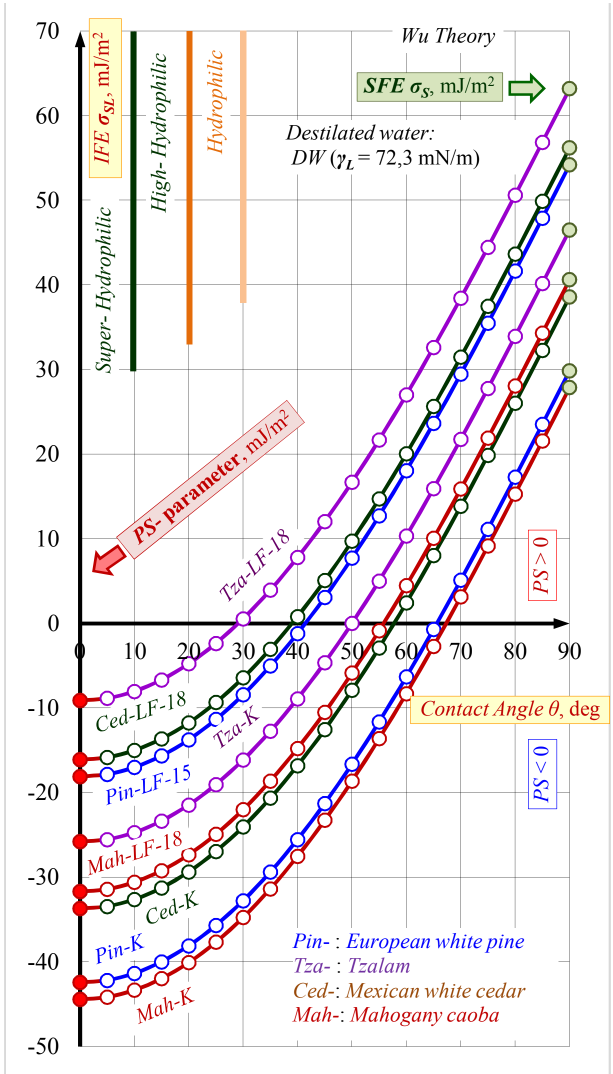

Fig. 4. The use of distilled water (*DW*) as a test liquid does not carry adequate information on the effectiveness of the process of surface impregnation of investigated bare (*-K*) and plasma-chemical, activated (*-LF-XX*) woods by an aqueous solution of a phosphorus flame retardant (*PhFR*). The surface impregnation and the plasma-aided surface impregnation are not effective: min σ_{SL} ($\theta_{eq} = 0^{\circ}$) = *PSP* and $PSP < 0$.

The maximum value of the *IFE*, at $\theta_{eq} = 90°$, is equal to the value of wood *SFE*, $\sigma_{SL} = \sigma_S$, fig. 2 and 3. The minimum value of the "solid-liquid" interfacial energy (*IFE*, σ_{SL}) at $\theta_{eq} = 0^0$ is equal to the value of *PSP*: $\sigma_{SL} = PSP$:

$$PSP = \sigma_S - \gamma_L = \sigma_{SL} + \gamma_L (\cos\theta - 1) > 0 \qquad (1)$$

Therefore, the requirement for effective finishing process

$$\sigma_S > \sigma_{SL} > PSP > 0 \qquad (2)$$

can be transformed into a new requirement for effectiveness, which already refers directly to *IFE*: "it is necessary for the entire range of variation of the static contact angle ($0^0 \le \theta_{eq} \le 90^0$) the value of the *IFE* be greater than zero: $\sigma_{SL} > 0$" [1].

This means that the *PSP* accepts a value of IFE at $\theta_{eq} = 0^0$ or in this way, the *PSP* has the physical nature of the *IFE*.

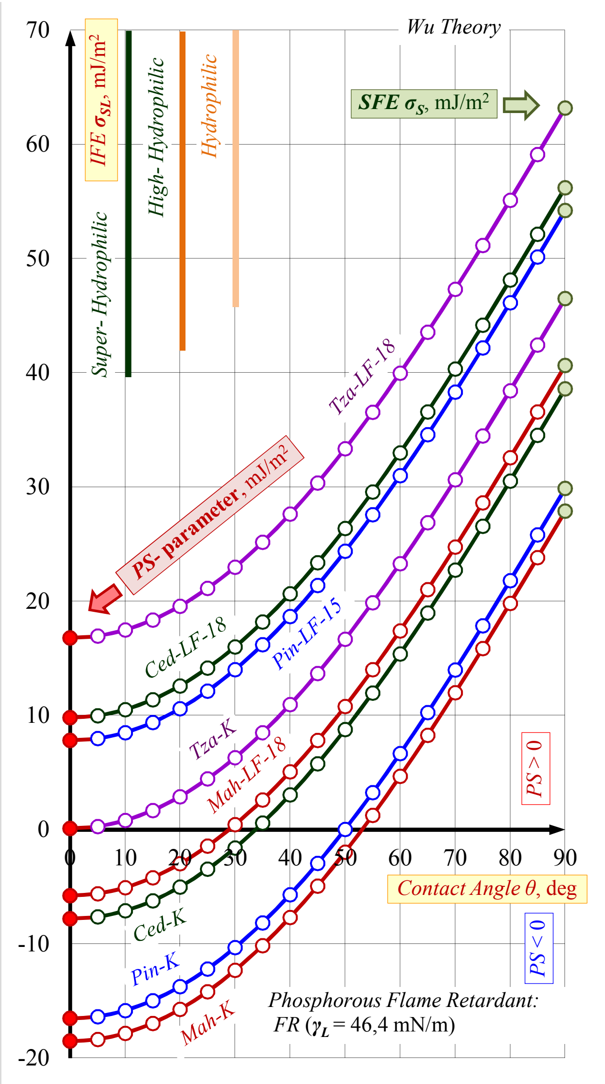

Fig. 5. Effective mode selection of *APP*- activation for an effective plasma-aided impregnation processing was found only for European white pine (*Pin-LF-15*), Mexican white cedar (*Ced-LF-18*), and Tzalam (*Tza-LF-18*): *PSP* > 0. No operative *APP*-activation mode for Mahogany caoba (*Mah-LF-18*) was found.

THE OBJECTIVE of this paper was to study the effect of *APP*- activation at industrial frequency (50 Hz) and critical voltage of 15 (or 18) kV rms, after 2 and 24 hours of aging, on the wood surface impregnability monitored by *PSP* and the processing efficiency rule: *PSP* > 0.

The plasma-chemical surface functionalization ages rapidly on ambient air after *APP*-activation. For this reason, plasma-aided surface impregnation can not be used effectively “out of production line” e.g. after 2 or 24 hours of aging.

III. EXPERIMENTAL REZULTS AND DISCUSION

The development of an effective process of plasma-enhanced surface impregnation passes through three stages:

- *The first stage* or selection of a plasma-chemical mode of surface activation (functionalization) that provide the necessary high *SFE* after an aging of 2 hours, fig. 2;
- *The second stage* or selection of an anionic micelle-forming water surfactant concentration to ensure the required minimum of *LST* (30 mJ/m² > γ_L > 20 mJ/m²), fig 4.
- *Third stage* or selection of an effective *APP*- activation that provide the necessary high *SFE* after an aging of 24 hours and an adequate surfactant concentration, fig. 5;

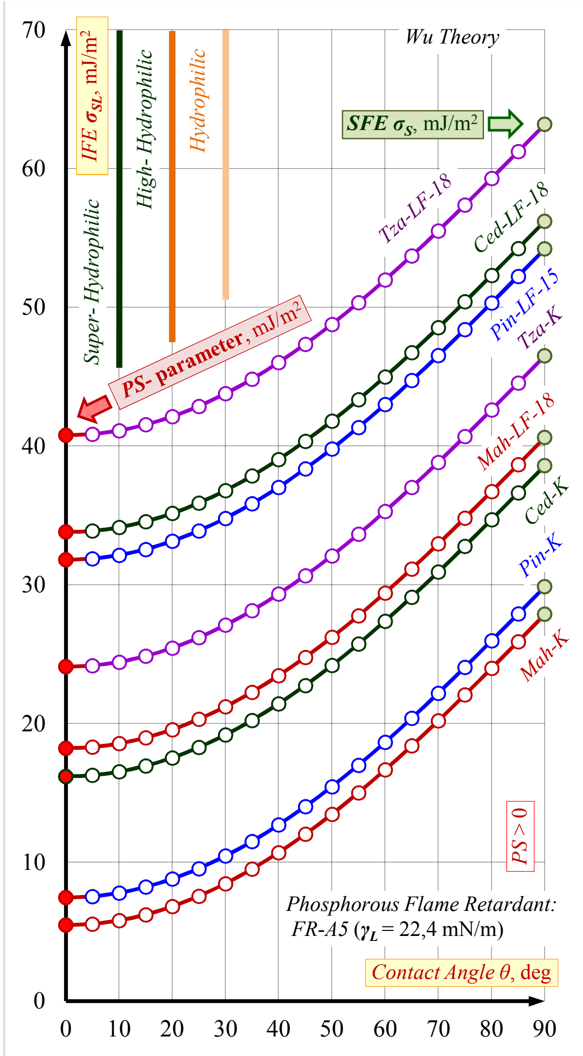

Fig. 6. Effective mode selection of water impregnation solution of phosphoric flame retardant modified with *anionic micelle-forming* or *water surfactant* at concentration of 5 vol. % (*FR-A5*). The rule of processing efficiency was met for all applications of plasma-aided impregnation.

This experimental study was carried out on four kind of wood samples according the well known method, [1, 2, 3, and 4]; the plasma- chemical surface activation was carried out with atmospheric-pressure dielectric barrier discharge in a working gap of 6 mm and time of 60 sec.

On the first stage the *PSP*- testing (*PSP* = 8.2 mJ/m² > 0) indicates that the voltage of the *APPT* of European white pine samples should be higher than 15 kV rms (σ_S = 54.2 kJ/m^2) for an effective impregnation processing, *PSP* > 0, fig. 5.

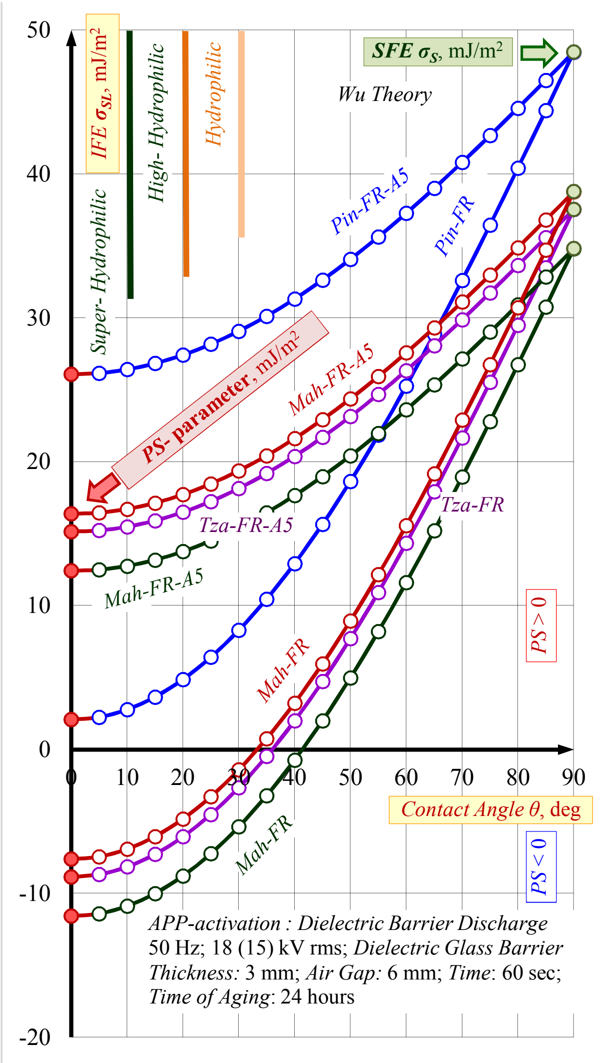

Fig. 7. Effective mode of plasma-aided surface impregnation for all investigated wood samples – *APP*- activation at critical voltage of 18 (15) kV rms and surface impregnation by modified *FR*-impregnation solution (γ_L = 22.4 mJ/m^2) with anionic micelle-forming surfactant at concentration of 5 vol. % (*-FR-A5*). The rule of processing efficiency was met only for all applications of plasma-aided impregnation with the modified *FR*-impregnation solution: *PSP* > 0.

The critical tension which fulfill the requirement *PSP* > 0 for other wood samples – Tzalam, Mexican white cedar and Mahogany caoba, was determined using the same procedure and it is 18 kV rms.

On the second stage the *PSP*-testing of *European white pine* indicates that the basic *FR*-impregnation solution (γ_L = 46.4 mJ/m^2) and the modified *FR*-solution with less *LST* (γ_L = 22.4 mJ/m^2) perform the rule of processing efficiency after 2 and 24 hours of aging, *PSP* > 0, fig. 6 and 7.

But this is not so for the other studied wood samples. The processing efficiency rule was performed for all application of plasma-aided surface impregnation, after 2 hours of aging, and the use of modified impregnating *FR*-solution, γ_L = 22.4 mJ/m^2: *PSP* > 0, fig. 6.

On the third stage the PSP-testing of all studied wood samples indicates that the APP- activation at critical voltage and surface impregnation with the modified impregnating FR-solution (γ_L = 22.4 mJ/m^2) satisfy the rule of processing effectiveness: *PSP* > 0, fig. 6.

This study proves our claim that the plasma-aided surface impregnation can be applied “out of the process line” after aging of one day or 24 hours between *APP*-activation and surface impregnation.

This study meets another issue which can be formulated so: “is it possible a simple drop test with distilled water to adequately replace the entire study conducted with the real impregnating FR-water solution”. The answer can be found in the information submitted to the two figures – fig. 1 and 2.

Distilled water has too high surface tension: γ_L = 72.3 >> 46.4 > 22.4 mJ/m^2. Atmospheric pressure plasma activation cannot compensate it by increasing surface free energy σ_S, so be enforced rule process efficiency: $\sigma_S < \gamma_L$; *PSP* < 0, fig. 4.

The study of the *APP*-activation by distilled water carries quite different information about the outcome of plasma-aided surface impregnation with modified (γ_L = 22.4 mJ/m^2) or not modified (γ_L = 46.4 mJ/m^2) *FR*-solution. Normally it fails to predict effective plasma-aided impregnation, fig. 1 and 2.

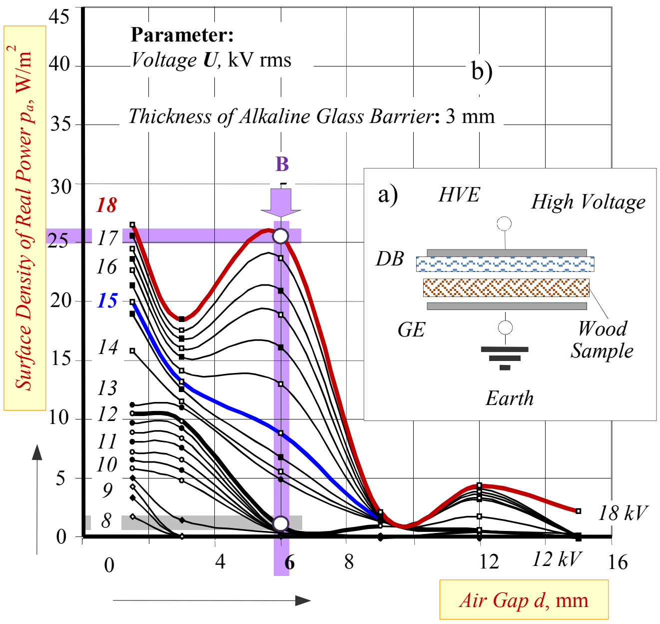

Fig. 8. Atmospheric pressure plasma activation of wood samples by non-equilibrium DBD in asymmetric coplanar electrode system with one glass barrier (*a*), technological discharge characteristic “*specific surface active power p_a – voltage U*”, and cathode-directed streamer mode (A) of plasma-chemical surface activation at industrial frequency (*b*).
HVE - high-voltage electrode; *GE* – grounded electrode; *DB* – dielectric alkaline barrier.

All experimental studies relate to plasma-chemical surface activating the wood specimens in a specific burning mode of the dielectric barrier air discharge (DBD) at a maximum of the surface density of real power $p_a = 26$ W/m^2 (18 kV rms) and 8 W/m^2 (15 kV rms) in asymmetric coplanar electrode system with 6 mm air gap. The effective operating modes of *DBD* at voltages above 13 kV rms (18.3 kV peak value) determine a surface density of real power above 4 W/m^2, fig.8.

In a well known impregnation FR-solution with measured surface tension and depleted possibilities for its reduction, the only possibility for effective surface impregnation remains to be sought to increase the surface free energy by selecting a suitable plasma-chemical surface activation technology, fig. 9.

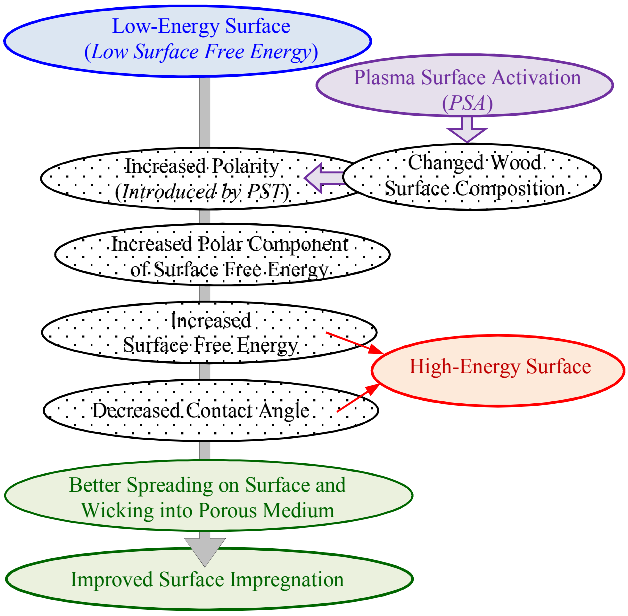

Fig. 9. The response of wood surface on plasma-chemical surface activation was complex but appears to be governed by its changed surface composition, especially by the introduced oxygen containing functionalities that increased the surface free energy and polarity.

The created new plasma-aided surface impregnation processes can find its wide application only if it can be predicted and managed so as to be executed the process efficiency rule: *PSP* > 0. The condition for this is the possibility of surface free energy measurement "in situ" out of the lab.

We believe that this approach can be successful, as the market now has the technical device for measuring the surface free energy "in situ". There is an innovative "mobile surface analyzer" for measuring surface free energy with two liquids using new dispenser with two parallel drops with "one click" for direct analysis of the contact angle and the derived results of the surface free energy [1].

IV. Conclusion

The efficiency of a plasma-aided process of impregnation can be predicted only by setting the two basic parameters of the real porous wood surface and the actual FR-impregnating solution – surface free energy and liquid surface tension. Successful finishing process, including effective surface impregnation, can be expected if the process efficiency parameter take a positive value (*PSP* > 0) after a prescribed aging time – 2 or 24 hours.

The evaluation of the finishing process effectiveness by instrumentally determining *first*, the reduced by surfactants impregnation liquid surface tension, and *second,* the increased surface free energy after plasma-chemical surface activation, is able to predict the result of a plasma enhanced finishing process.

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