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CONTENTS

MECHANICAL SCHEMES AND SUSTAINABILITY OF PLASTIC FLOW METAL
Sosenushkin E.H., Yanovskaya E.A., Sosenushkin A.E. 3

THE STUDY OF THE PROCESS OF COMPLEX DIFFUSION SATURATION WITH BORON AND VANADIUM ON THE CARBON STEELS
Lygdenov B., A. Guriev, A. Sitnikov, Mei Shunqi,Y. Khraraev, V. Butukhanov, B. Tsyretorov 7

SIMULTANEOUS THERMAL ANALYSIS INVESTIGATION ON PLASMA-AIDED FLAME RETARDANCY OF WOOD
Ivanov I., D. Gospodinova, P. Dineff, L. Veleva (Muleshkova) 9

APPLICATION OF NUMERICAL METHODS IN CALCULATION OF ELECTROMAGNETIC FIELDS IN ELECTRICAL MACHINES
Sarac V., G.Galvincev 13

DETERMINING THE CATEGORY OF WELDED JOINTS FOR THE NON-REGULATED AREA OF MACHINE BUILDING
Zhelev A., T.Osikovski 17

GEOMETRICAL SYNTHESIS OF FINE-MODULE RATCHET TOOTHING
Sharkov O. 20

IMPROVING THE UNIFORMITY OF PROPERTY DISTRIBUTION ALONG THE SURFACE OF FILTER MATERIALS OBTAINED USING POROGENS
Ilyushchenko A., R. Kusin, I. Charniak, A. Kusin, D. Zhehzdryn 24

DIFFUSION BONDING MACHINERY FOR MANUFACTURING AEROSPACE PARTS
Lee Ho-Sung, Yoon, Jong-Hoon, Yoo, Joon-Tae 28

AN AGENT BASED PROCESS PLANNING SYSTEM FOR PRISMATIC PARTS
Andreadis G. 31

EXPERIMENTAL INVESTIGATION ON THE EFFECT OF COOLING AND LUBRICATION ON SURFACE ROUGHNESS IN HIGH SPEED MILLING
Leppert T. 35

STRUCTURE AND CHARACTERISTICS COMPLEX DIFFUSION LAYERS AFTER SATURATION BORON AND COPPER ON STEEL
Chernega S., I. Poliakov, M. Rrasovsky, K. Grynenko 39

ALUMINIUM BIMETAL STRUCTURE PRODUCTION BY LOST FOAM CASTING WITH LIQUID-LIQUID PROCESS
Kisasoz A., K. A. Guler, A. Karaaslan 43

FABRICATION OF AL/STEEL COMPOSITES BY VACUUM ASSISTED BLOCK MOULD INVESTMENT CASTING TECHNIQUE
Guler K.A. A. Kisasoz, A. Karaaslan 47

WOOD SURFACE ENERGY DETERMINED BY SESSILE DROP TECHNIQUE AS QUALITY PARAMETER OF PLASMA-CHEMICAL MODIFYED WOOD SURFACES
Ivanov I., D. Gospodinova, P. Dineff, L. Veleva 50

PLATE HEAT EXCHANGER WITH POROUS STRUCTURE FOR POTENTIAL USE IN ORC SYSTEM
Wajs J., D. Mikielewicz, E. Fornalik-Wajs 54

WOOD SURFACE ENERGY DETERMINED BY SESSILE DROP TECHNIQUE AS QUALITY PARAMETER OF PLASMA-CHEMICAL MODIFYED WOOD SURFACES

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Abstract: *The analysis of pre-treated wood surfaces, which have been plasma modified is also very informative for wood quality. Our measuring instruments determine the wettability based on the contact angle. The optical shape analysis of drops which are dispensed onto the surface is a reliable method for carrying out this measurement. The aim of this study was to verify possibility of determining the contact angle values of the plasma activated wood and calculate the surface free energy and its components of wood from the obtained contact angle values using Zisman, Equation of state (EOS), Fowkes and Wu theory and calculation method. Based on the contact angle data, the surface energy was obtained from the polar-dispersive(non-polar) approach. This study has been created as part of a large investigation on plasma-chemically activated wood surface and flame retardant treated wood.*

Keywords: ATMOSPHERIC DIELECTRIC BARRIER DISCHARGE, CONTACT ANGLE MEASUREMENT, FLAME RETARDANT, PLASMA-AIDED CAPILLARY IMPREGNATION, SESSILE DROP TECHNIQUE, SURFACE ENERGY DETERMINING.

1. Introduction

The plasma-aided flame retardation of wood, and wooden products has been developed as a result of a new plasma-aided process of capillary impregnation that comprises a surface plasma pre-treatment for alteration of chemical activity of wood surface as well as its electrical (ionic) and capillary activities, and in general for improvement of the capillary impregnation process. A technological system of plasma device and applicators has been created to produce cold technological plasma through dielectric barrier discharge (*DBD*) at atmospheric pressure and room temperature. The cold plasma pre-treatment of wood, improves water solution spreading and absorption speed, as well as a specific amount of the adsorbed flame retardant. In this way, the plasma pre-treatment of wood and wooden products improves its flame retardation [1, 2 and 6].

The objective of this paper was to study the effect of plasma pre-treatment on wood surface as well as the effect of wood surface polarity on the wetting phenomena, both aiming to improve the capillary impregnation process. This study has been developed as part of a large research on plasma-chemically activated wood surface and flame retarded rain-forest wood.

2. Experimental Investigation

Wetting phenomena of wood may be characterized by using thermodynamic wetting parameters, for example contact angles, surface free energy, and work of adhesion, work of spreading or work of wetting. It is important to know that such parameters are by definition bulk measurements, and they do not directly describe the interaction at a molecular level [3].

Some basic relations for the study of wetting phenomena

If a liquid droplet is placed on a smooth, non-porous and rigid solid both exposed to a gas/vapor, Fig. 1, and if the whole system is in equilibrium state, the *contact angle* θ is then defined as the angle between the tangent to the liquid surface and the liquid/solid surface at the point of liquid/solid/gas contact. *Young's equation* expresses the relation between the contact angle θ for a droplet of liquid deposited on a flat horizontal ideal - *smooth, non-porous and rigid* surface and *the work of adhesion*, W_a, defined (*Dupré*, 1869) as the work required to separate unit area of the solid-liquid interface, i.e.:

$$W_a = \gamma_L (1 + cos\theta) \qquad (1)$$

where γ_L is the surface free energy of the liquid (L) surfaces in vacuum assuming that $\gamma_L \approx \gamma_{LG}$ (γ_{LG} - the surface free energy of the liquid exposed to a gas/vapor) [3].

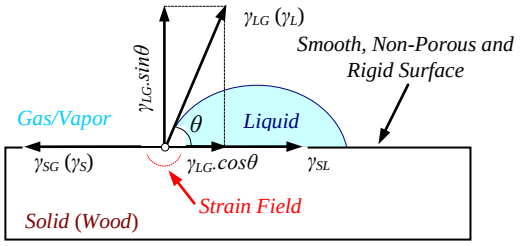

Fig. 1. *Schematic illustration of Young-Bikerman-Good model of wetting phenomena - if a liquid drop is placed on a smooth, non-porous and rigid solid, both exposed to a gas/vapor: if the system is not in equilibrium and the liquid 'wets out' the solid then the liquid exhibits a contact angle of zero against the solid, i.e. so if $\gamma_{SG} > \gamma_{SL} + \gamma_{LG}$, then $cos\theta = 1$ and $sin\theta = 0$ ($\theta = 0$) and $\gamma_{LG}\, sin\theta = 0$ (Good, 1993).*

Neumann and *Good* (1979) reviewed the classical techniques for measuring contact angles. Using well defined liquid, if the contact angle can be measured on a solid surface, the work of adhesion can be determined and the solid surface can be revealed. The most widely used technique, also regarding wood, involves digital image analysis of the profile dimensions of a droplet deposited on a horizontal surface from which the contact angle can be calculated - referred to here as the sessile drop method.

A low contact angle indicates a high solid surface energy, and a high or sometimes complete degree of wetting. For example, a contact angle of zero degrees will occur when the droplet has turned into a flat puddle; this is called complete wetting Fig. 1.

The wetting phenomena on a real (non-ideal) - *rough, porous, heterogeneous, or hygroscopic* wood surface, Fig.2, can be involved by: *i* - the spreading of liquid over a solid surface; *ii* - the wicking of a liquid into a porous solid (as wood).

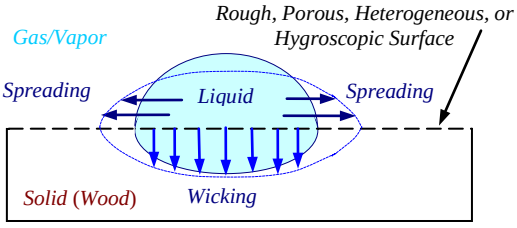

Fig. 2. *Young-Bikerman-Good model of wetting phenomena - the wetting phenomena on a real surface can be involved by: i - the spreading of liquid over a solid surface; ii - the wicking of a liquid into a porous solid (as wood). Wetting does not include dissolution or swelling of the solid by the liquid or any kind of chemical reaction between the materials that changes the system composition (Berg, 1993).*

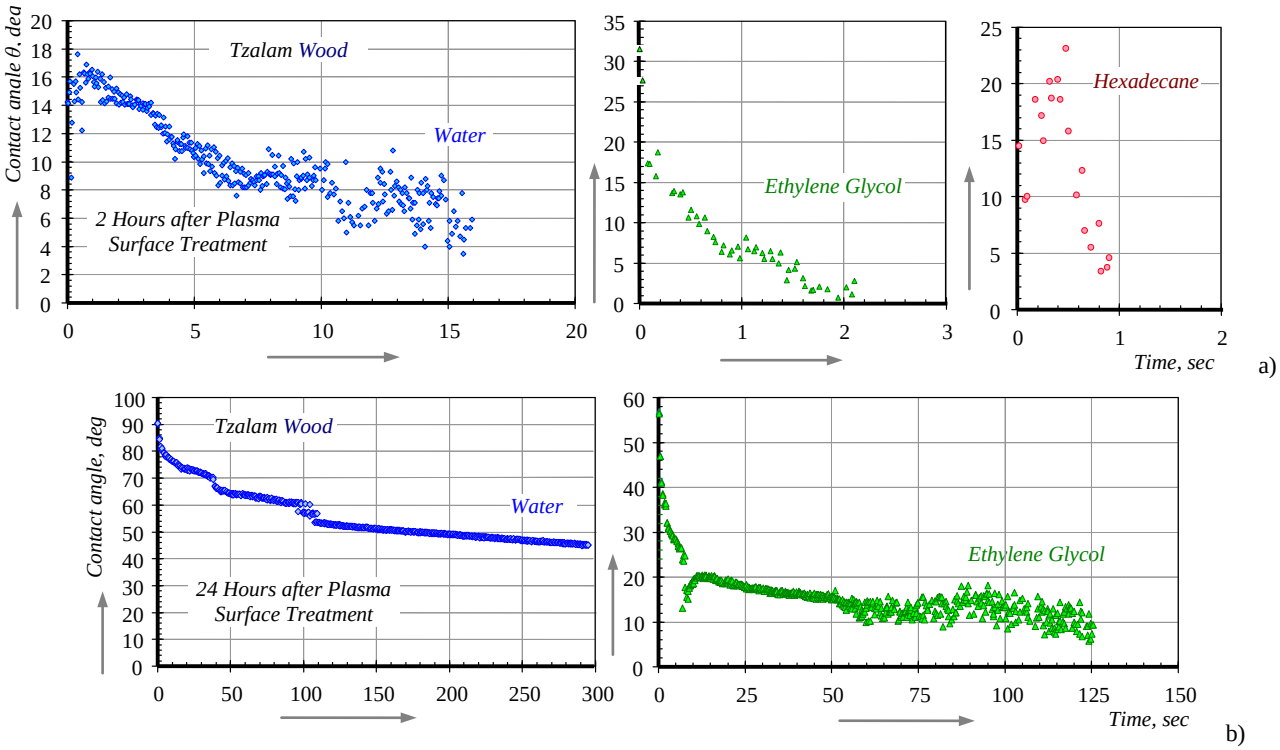

Fig. 3 *Time-depending change of contact angle θ of a liquid as it advances slowly over a non-ideal Tzalam (Lysiloma bahamensis) wood surface (e.g., not chemically homogeneous, rough or not perfectly smooth, porous and hygroscopic as in the case of most practical wood surfaces) - contact angle measurements 2 (a) and 24 (b) hours after atmospheric dielectric barrier discharge (DBD) surface treatment in air with specified test liquids: Water (bifunctional; θ = 70.2 ± 0.1 deg, total surface energy - 72.8 mN/m; dispersive component - 21.8 mN/m; polar component - 51 mN/m; acid component - 25.5 mN/m, and base component - 25.5 mN/m); Ethylene glycol (acidic, θ = 41 ± 0.1 deg, total surface energy - 47.5 mN/m, dispersive component - 29.3 mN/m; polar component - 18.2 mN/m; and n-Hexadecane (neutral, θ = 10.3 ± 0.1 deg, total surface energy - 27.6 mN/m, dispersive component - 27.6 mN/m; polar component - 0 mN/m).*

Wetting does not include dissolution or swelling of the solid by the liquid or any kind of chemical reaction between the materials that changes the system composition. It must be emphasized that the contact angle of a liquid as it advances slowly over a non-ideal surface (e.g. not chemically homogeneous, porous and not perfectly smooth, as in the case of wood surfaces) changes (decreases) synchronously to droplet change and movement. The droplet was deposited by a syringe pointed vertically down onto the wood surface, and a high resolution camera captures the image, which can then be analyzed by using image analysis software. By taking pictures incrementally as the droplet advances over the surface, the user can acquire a set of data to get a good time-depending change of the contact angle Fig. 3 and 4.

Experimental investigation

The apparatus used for this study was a KRÜSS Drop Shape Analyzer DA100. Measurement of the contact angle with three liquids ensures maximum accuracy when determining the surface free energy of wood. Precisely controlled tempering and humidity chambers help to provide a realistic modeling of the process conditions. Measuring range (referred to image analysis): contact angle - 1÷180 deg; surface free energy - 0.01÷1000 mN/m. Measurement resolution: contact angle - 0.1 deg; surface free energy - 0.01 mN/m. The aim of this study was to verify possibility of determining the contact angle values of the plasma pretreated wood surface and calculate the surface free energy and its components from the obtained contact angle values using *Zisman's*, *Equation of state* (EOS), *Fowkes* and *Wu's* theory and calculation method. All methods described there are integrated in the KRÜSS Drop Shape Analysis programs DSA1 and DSA2.

On the basis of prior art, as well as on our own experience in plasma-aided capillary impregnation of wood and wooden materials, [1, 2], 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 atmospheric

Table 1. *Calculated Total Free Surface Energy of Wood Samples at about 22 ^{0}C*

Wood Sample	Sessile Drop Test					Components of Total Surface Free Energy					
	Total Surface Free Energy					Fowkes Theory			Wu Theory		
	Zisman Theory	Equation of state (EOS) Theory		Fowkes Theory	Wu Theory	Polar Component	Dispersive Component	Polarity/ Non-Polarity	Polar Component	Dispersive Component	Polarity/ Non-Polarity
	mN/m	mN/m		mN/m	mN/m	mN/m	mN/m	-	mN/m	mN/m	-
Tzalam (*Lysiloma bahamensis*)											
2 h old	NA	47.08	± 21.17	57.27	63.18	32.09	25.18	0.56/0.44	38.88	24.30	0.62/0.38
24 h old	30.33	32.68	± 4.96	34.77	37.54	6.50	28.27	0.19/0.81	9.78	27.76	0.26/0.74
Caoba Mahagony (*Swietenia macrophylla*)											
2 h old	27.90	35.41	± 7.44	37.96	40.62	10.99	26.96	0.29/0.71	14.35	26.27	0.35/0.65
24 h old	29.77	33.89	± 5.48	36.07	38.77	8.19	27.88	0.23/0.77	11.46	27.31	0.30/0.70
Mexican White Cedar (*Cupressus Lusitanica*)											
2 h old	NA	43.47	± 21.75	49.25	56.20	24.41	24.84	0.50/0.50	33.25	22.95	0.59/0.41
24 h old	31.44	29.17	± 7.65	32.08	34.81	2.66	29.42	0.08/0.92	5.95	28.87	0.17/0.83

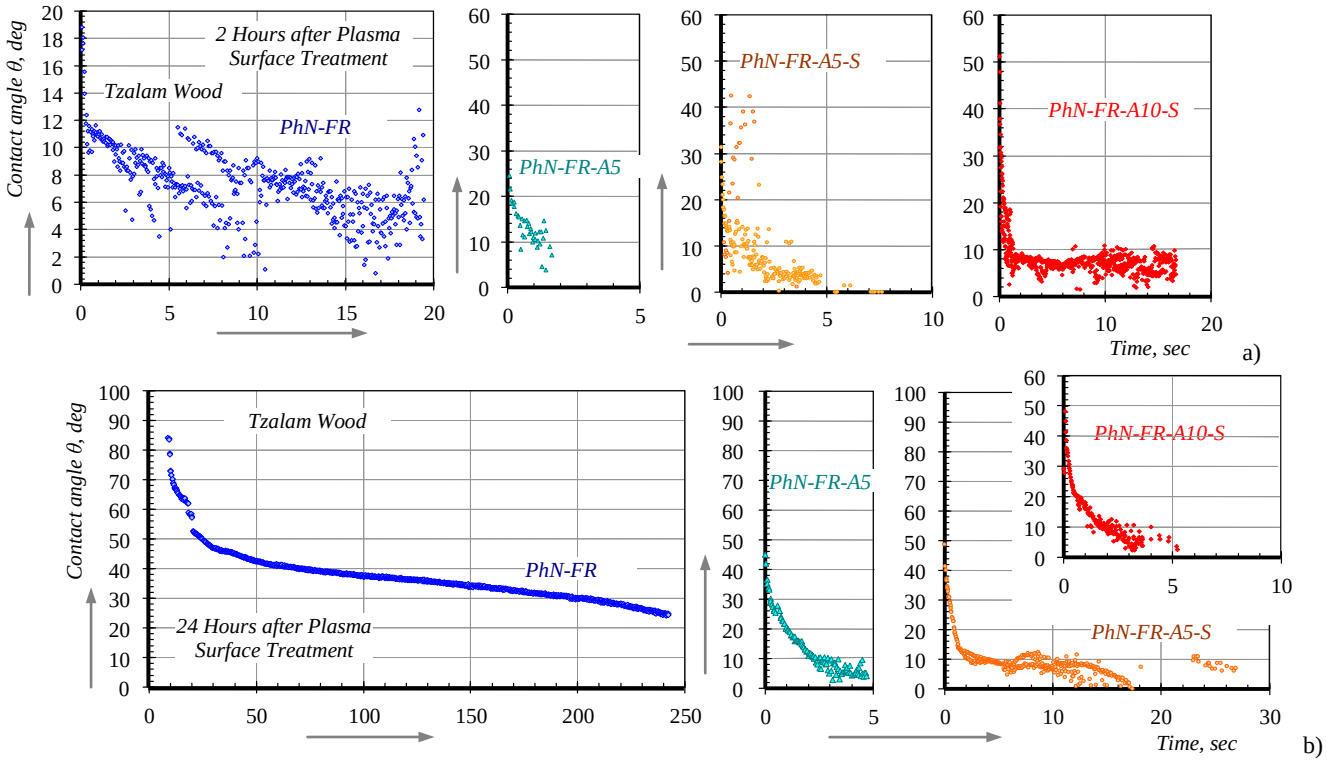

Fig. 4. *Time-depending change of contact angle θ of a flame retardant water solution as it advances slowly over a non-ideal (wood) surface (e.g., not chemically homogeneous, rough or not perfectly smooth, and porous as in the case of most practical wood surfaces): PhN-FR - 30 mass % water impregnation solution of phosphor and nitrogen containing flame retardant; PhN-FR-A5 - water solution with 5 vol. % anionic surfactant; PhN-FR-A5-S - water solution with 5 vol. % anionic surfactant and 0.1 vol. % spreader; PhN-FR-A10-S - water solution with 10 vol. % anionic surfactant and 0.1 vol. % spreader - 2 (a) and 24 (b) hours old surfaces - after atmospheric dielectric barrier discharge (DBD) surface treatment in air.*

dielectric barrier air discharge (*DBD*) in industrial frequency (50 Hz) and 18 kV (*RMS*) or 25 kV (*PV*) voltage.

Results and discussion

Wood surface energy as quality parameter of plasma treated wood surface

Since wood surfaces are porous, rough and not perfectly smooth, sessile drop method requires some type of video capture in order to measure the contact angle which changes as the droplet is absorbed. Time-depending change of contact angle θ of three probe liquids - *water*, *ethylene glycol* and *n-hexadecane*, as its advance slowly over the non-ideal wood surface are presented in Fig. 4.

Based on the contact angle data of plasma activated wood surface, the total surface free energy and its components were obtained using *Zisman's Equation of state*, *Fowkes* and *Wu's* theory and calculation methods. Total surface free energy and its polar and dispersive (non-polar) components are presented in Fig. 5 and Table 1.

The dispersive and polar surface free energies of the three rain-forest wood species were obtained using Wu and Fowkes theories and calculation methods. In general, plasma surface activated woods show a high polar surface free energy component (*PEC*) and polarity *p*. The fresh (2-hours-old) plasma activated surfaces show considerably greater polarity than surfaces kept for a long time (24-hours-old) after plasma pre-treatment: *Tzalam* - 0.56 against 0.19; *Mexican White Cedar* - 0.50 against 0.08; *Caoba Mahogany* - 0.29 against 0.23 (*Fowkes method*); *Tzalam* - 0.62 against 0.26; *Mexican White Cedar* - 0.35 against 0.30; *Caoba Mahogany* - 0.59 against 0.17 (*Wu method*). Plasma activated wood surfaces show a very high dispersive surface energy.

The 24-hours-old plasma treated surfaces have large non-polarity (1 - *p*): *Tzalam* - 0.81; *Mexican White Cedar* - 0.77 and *Caoba Mahogany* - 0.92 (*Fowkes method*);

Table 2. *Wood surface energy, especially the polar component (or the polarity in general) is related to the surface composition - especially to the oxygen/carbon ratio (XPS analysis)*

Wood Sample	Wood Surface Analysis of Plasma Surface Treated Samples (*DBD*, 18 kV RMS, 50 Hz)								
	XPS Analysis	Sessile Drop Test - Fowkes Theory				Sessile Drop Test - Wu Theory			
	Oxygen/ Carbon Ratio (*nO/nC*)	Total Surface Free Energy	Polar Component	Dispersive or Non-Polar Component	**Polarity**	Total Surface Free Energy	Polar Component	Dispersive or Non-Polar Component	**Polarity**
	-	J/m	J/m	J/m	-	J/m	J/m	J/m	-
Tzalam (*Lysiloma bahamensis*)									
After 2 h	**0.6131**	57.27	32.09	25.18	**0.560**	63.18	38.88	24.30	**0.615**
Mexican White Cedar (*Swietenia macrophylla*)									
After 2 h	**0.4477**	49.25	24.41	24.84	**0.495**	56.20	33.25	22.95	**0.592**
Caoba Mahogany (*Cupressus Lusitanica*)									
After 2 h	**0.3970**	37.96	10.99	26.96	**0.290**	40.62	14.35	26.27	**0.353**
In General									
Cellulose	**0.8300**								
Lignin	**0.3300**								

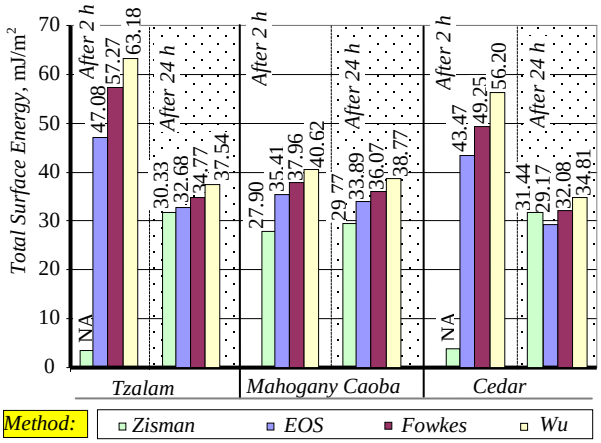

Fig. 5. *Total surface energy of three rain-forest (Mexico, Yucatán) heart wood samples - Mexican white cedar (Cupressus Lusitanica), Caoba mahogany (Swietenia macrophylla), and Tzalam (Lysiloma bahamensis), 2 and 24 hours after DBD - plasma treatment in air.*

Tzalam - 0.74; *Mexican White Cedar* - 0.70 and *Caoba Mahogany* - 0.83 (*Wu method*). Approximately 0.70 to 0.92 of the overall wood surface energy is attributed to dispersion forces, Table 1.

The 2-hours-old plasma treated surfaces have lower non-polarity (1/*p*): *Tzalam* - 0.44; *Mexican White Cedar* - 0.50 and *Caoba Mahogany* - 0.71 (*Fowkes method*); *Tzalam* - 0.38; *Mexican White Cedar* - 0.41 and *Caoba Mahogany* - 0.65 (*Wu method*) - approximately 0.38 to 0.71 of the overall wood surface energy is attributed to dispersion forces Table 1.

Zisman's theory and *EOS* theories are not suitable for high energy surfaces such as 2-hours-old plasma activated wood surfaces. They are mostly used for low energy surfaces Table 1.

Fowkes and *Wu's* theories are more suited for higher energy wood surfaces, and since they are rooted in theories about capillary impregnation, they are more suitable for the characterization of interactions where the solids and liquids have a high affinity for one another, Table 1.

Surface energy and chemical composition

By means of *XPS/ESCA*-analysis it is possible to analyze the chemistry and the surface reorganization after plasma-chemical pre-treatment to a depth of 5 to 10 nm.

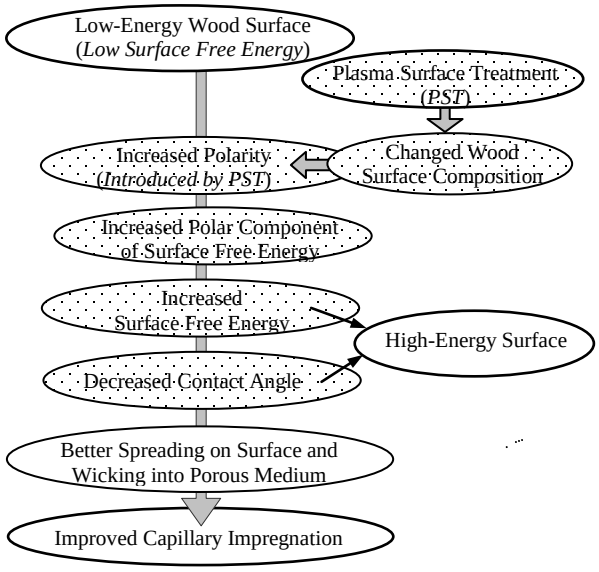

Fig. 6. *The response of wood surface on plasma-chemical surface pre-treatment is complex but it appears to be controlled by its surface composition, especially by the introduced oxygen containing functionalities and increased surface polarity.*

Obviously the polar component of surface free energy (for plasma treated woods) is well correlated with the *Oxygen*/*Carbon* (*nO/nC*) ratio (*XPS*/*ESCA*-analysis), Table 2.

Among the main factors determining the chemistry of the wood surface, the adsorption of gases and vapors (water), chemical composition, aging, thermal processing and machining, extractives migration to the surface and surface inactivation, plasma-chemical pre-treatment (activation) may be perceived as management tool for purposeful changing the surface chemistry and the total surface free energy. Wood surface free energy, especially its polar component is related to the distribution of oxygen containing groups. The response of wood surface to plasma-oxidative (*DVD*) treatment is complex but appears to be controlled by its oxygen containing functionalities [6, 7].

The observed increase in surface free energy has to be combined to significant reduction of surface tension of the flame retardant containing water solution by the addition of surfactants and spreaders Fig. 4.

The main objective of plasma surface oxidizing pre-treatment of wood is the conversion of the low-energy in high-energy wood surface, Fig. 6.

Conclusion

The wetting theory, expressed in terms of thermodynamic wetting parameters, such as the contact angle and the surface free energy, is the most widely used approach in adhesion science at present, and this work considers only this type of capillary impregnation phenomena, also referred to 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.

Acknowledgments

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