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SIMULTANEOUS THERMAL ANALYSIS INVESTIGATION ON PLASMA-AIDED FLAME RETARDANCY OF WOOD

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Abstract: *Simultaneous Thermal Analysis (STA) unifies the simultaneous application of thermogravimetry and differential scanning calorimetry to one and the same wood sample in a single instrument, under perfectly identical conditions - same atmosphere, gas flow rate, pressure, heating rate, thermal contact, etc. A new thermal analysis approach to distinguish between the flaming and glowing combustion of wood was discussed. The results obtained by STA were used in a new way, to reveal the influence of plasma-aided capillary impregnation on thermal decomposition and glowing of wood controlled by oxygen and nitrogen containing flame retardant. New integral criteria of thermal behavior and decomposition such as specific enthalpy change, and specific heat flux or heat release rate, have been developed by investigating three species rain-forest wood (Mérida, Yucatán) - Mexican white cedar (Cupressus Lusitanica); Caoba mahogany (Swietenia macrophylla); and Tzalam (Lysiloma bahamensis).*

Keywords: DIELECTRIC BARRIER DISCHARGE, FLAME RETARDANT, PLASMA-AIDED CAPILLARY IMPREGNATION, SIMULTANEOUS (TGA÷DSC) THERMAL ANALYSIS, CAOBA MAHOGANY, MEXICAN WHITE CEDAR, TZALAM WOOD.

1. Introduction

The plasma-aided flame retardation of wood, cellulosic and wooden products has been developed as a result of a new *plasma-aided process of capillary impregnation* that comprises: *i* - surface plasma pre-treatment for alteration of chemical, electrical (ionic) and capillary activities of wood surface as well as its surface energy; *ii* - modification of ionic activity and surface tension of flame retardant (*FR*) containing water solution by non-organic and siloxane surfactants (*surface-active agents*), and in general improvement of the technological characteristics of the capillary impregnation process such as solution spreading and wicking speed, as well as specific amount of the adsorbed flame retardant. In this way, the plasma pre-treatment of wood improves wooden flame retardation, Fig. 1 [1].

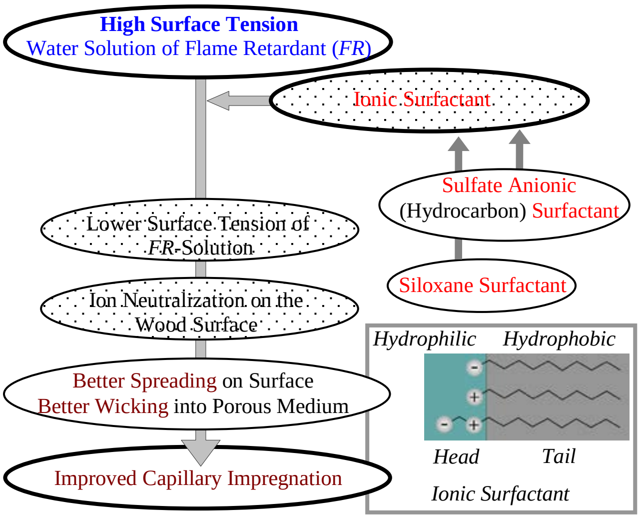

Fig. 1. *The main objectives of plasma-aided capillary impregnation technology (PACI) are both increasing of wood surface energy by plasma-chemical surface pre-treatment and decreasing of surface tension of impregnating FR-solution by ionic non-organic and siloxane surfactants (surface-active agents).*

In order to enhance the utilization of wood and its inherent properties, a long range Research and Development program, called *Non-equilibrium Air Plasma Surface Activation of Wood and Cellulosic Products*, has been formulated (*P. Dineff*, 2004). This concept was focused on achieving a basic understanding of wood and those surface properties that are not fully exploited in conventional wood manufacturing systems. The strategy was to activate these inherent properties and thus add economic value to completed wood products. Studies of *wetting phenomena on wood*, i.e. interactions of *FR*-water solutions with wood surface, may add valuable information about the gluing, coating, and impregnation (technological) properties of wood. Both non-polar and polar liquids can be absorbed into the porous cell structure of wood, but only polar liquids can penetrate (wicking) into the non-porous bulk material with resulting swelling [5].

In order to achieve this, a better knowledge of the fundamental behavior of wood surface was required, together with new applied plasma-aided processing technology and the development of necessary plasma-manufacturing systems [2 ÷ 4].

The objective of this paper was to study the effect of plasma pre-treatment on the wood surface energy as well as the effect of different surfactants on the surface tension of the *FR*-impregnation solution, both aiming to improve the wood flame retardation.

2. Experimental Investigation

A technological system including plasma device and applicators has been created to produce cold non-equilibrium air plasma through *dielectric barrier discharge* (*DBD*) at atmospheric pressure and room temperature.

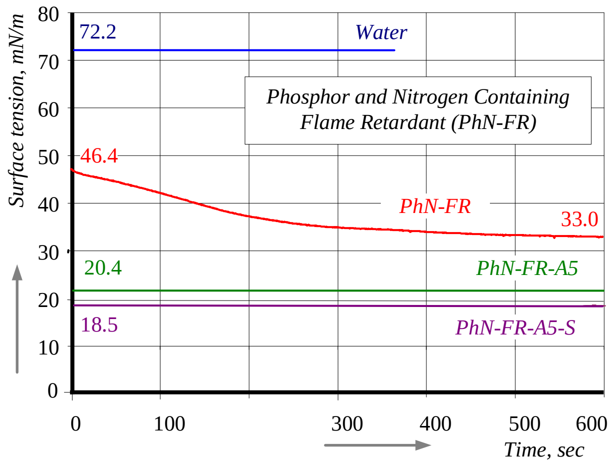

Fig. 2. *Surface tension of: PhN-FR - 30 mass % water impregnation solution of phosphor and nitrogen containing flame retardant; PhN-FR-A5 - water impregnation solution PhN-FR with 5 vol. % anionic phosphate surfactant; PhN-FR-A5S - water impregnation solution PhN-FR with 5 vol. % anionic phosphate s and 0.1 vol. % siloxane surfactant.*

Anionic phosphate surfactant ("*Aniticrystallin A*", Chimatech, Ltd., Bulgaria) in quantity of 5 vol. %, and siloxane surfactant (super spreader) (Y-17113, *Momentive Performance Materials* GmbH & Co. KG, Germany) in quantity of 0.1 vol. % have been

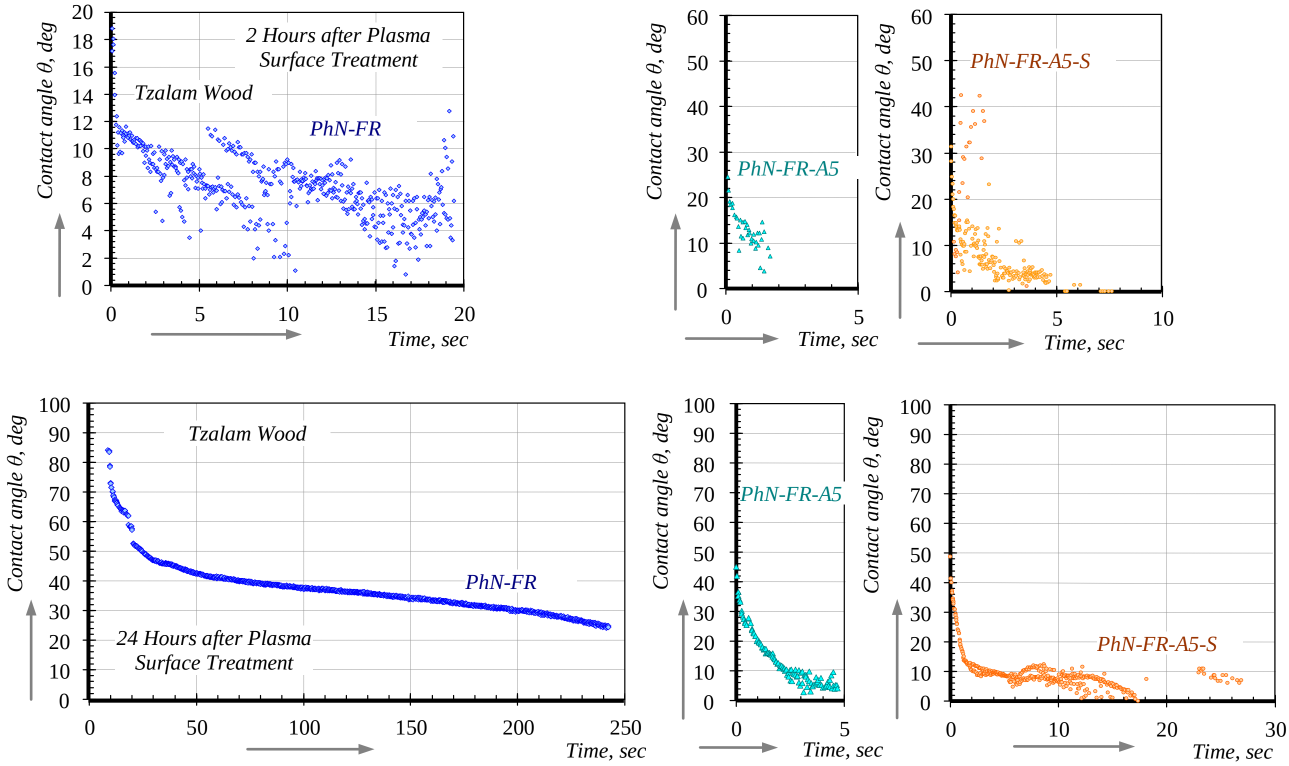

Fig. 3. *Time-depending change of contact angle θ of a FR-water solution as it advances slowly over a non-ideal (wood) surface (e.g., not chemically homogeneous, rough or not perfectly smooth, and porous as in the case of most practical wood surfaces): PhN-FR - 30 mass % water impregnation solution of 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.*

used in combination to control the ion activity of the *FR*-impregnation solution and its surface tension. The surfactants (*A5-S*) lower the surface tension of impregnating solution and thus allowing it to wet and penetrate solids. Sessile drop technique (CRÜSS Drop shape analyzer DA100) was used for these measurements.

The flame retardant (*FR*) water solution shows an interesting behavior during the measurement. There was a transition period during which its surface tension amended from 46.4 to 33.0 mN/m in a time of about 12 minutes. Introduction of surfactants (*PhN-FR-A5* and *PhN-FR-A5-S*) in this solution leads to both disappearance (less then 10 seconds) of the transitional period and a significant reduction of surface tension (less then 10 mN/m) - good wetting and chemical affinity. Regardless of the *open time* between plasma pre-treatment and capillary impregnation - two or twenty-four hours, the surfactants provide good wetting and wicking, and good chemical affinity Fig. 3.

3. Results and Discussion

The studied flame retardancy of wood was based on both: plasma-chemical pre-treatment of the wood surface to increase its surface energy and *PhN-FR*-solution modification by an ionic surfactant and combination of surfactants. It was expected that the increased wood capillary activity, *FR*-solution sorption speed and capacity, would allow good enough flame retardancy of porous wood surface [2, 3, and 4].

Based on our own experience in plasma-aided capillary impregnation of wood and wooden materials 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 dielectric barrier air discharge (*DBD*) at industrial frequency (50 Hz) and 18 kV (*RMS*) or 25 kV (*PV*) voltage [2 ÷ 4].

Preliminary results from a study of plasma-aided capillary impregnation allow us to formulate two new criteria of thermal behavior (pyrolysis and combustion), [4], - *specific heat flux* (*q*) and *specific enthalpy change* (*-Δh*) which are presented here. The criteria were formulated as a result of both non-equilibrium air plasma pre-treatment at atmospheric pressure and room temperature and capillary impregnation monitored by simultaneous (synchronous) thermal analysis (*STA, TGA and DSC*) of commonly used *thermogravimetric analysis* (*TGA*) and *differential scanning calorimetry* (*DSC*) Fig. 4 ÷7.

There are two possible ways to detect the influence of a surfactant (*A5*) or a combination of surfactants (*A5-S*) on wood flame retardancy by comparing the flaming resistivity of a modified solution (*PhN-FR-A5-S*) with a surfactant-free *FR*-solution (*PhN-FR*), Fig. 5 and 7, and a surfactant free *FR*-solution after plasma surface pre-treatment (*PTI*), Fig. 4 and 6.

It turned out that the selected wood species reveal a different impact of surfactants on wood flaming resistivity - the resistivity of *Mahogany caoba* (*Swietenia macrophylla*) against the flaming pyrolysis and combustion was increased, the resistivity of *Mexican white cedar* (*Cupressus Lusitanica*) against the flaming was reduced, and the resistance of *Tzalam* (*Lysiloma bahamensis*), against flaming was not substantially altered.

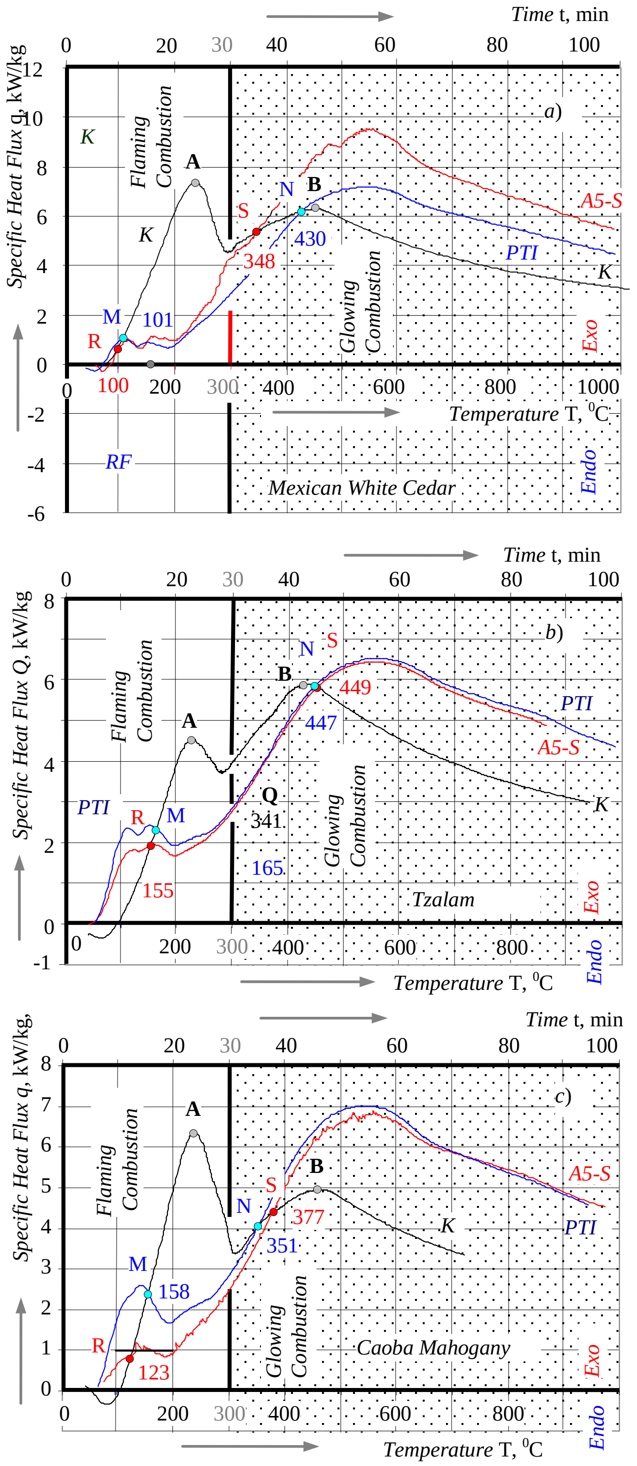

Fig. 4. *Criterion of wood pyrolysis and combustion behavior established on simultaneous thermal analysis (STA) - specific heat flux q spectra (per unit surface and mass losses) of bare wood sample (K), plasma-aided flame retarded sample (PTI) and plasma-aided flame retarded sample with FR-solution modified by anionic (5 vol. %) and siloxane surfactant or super spreader (0.1 %) (A5-S):* ***a*** *- Mexican White Cedar (Cupressus Lusitanica);* ***b*** *- Tzalam (Lysiloma bahamensis);* ***c*** *- Mahogany Caoba (Swietenia macrophylla).*

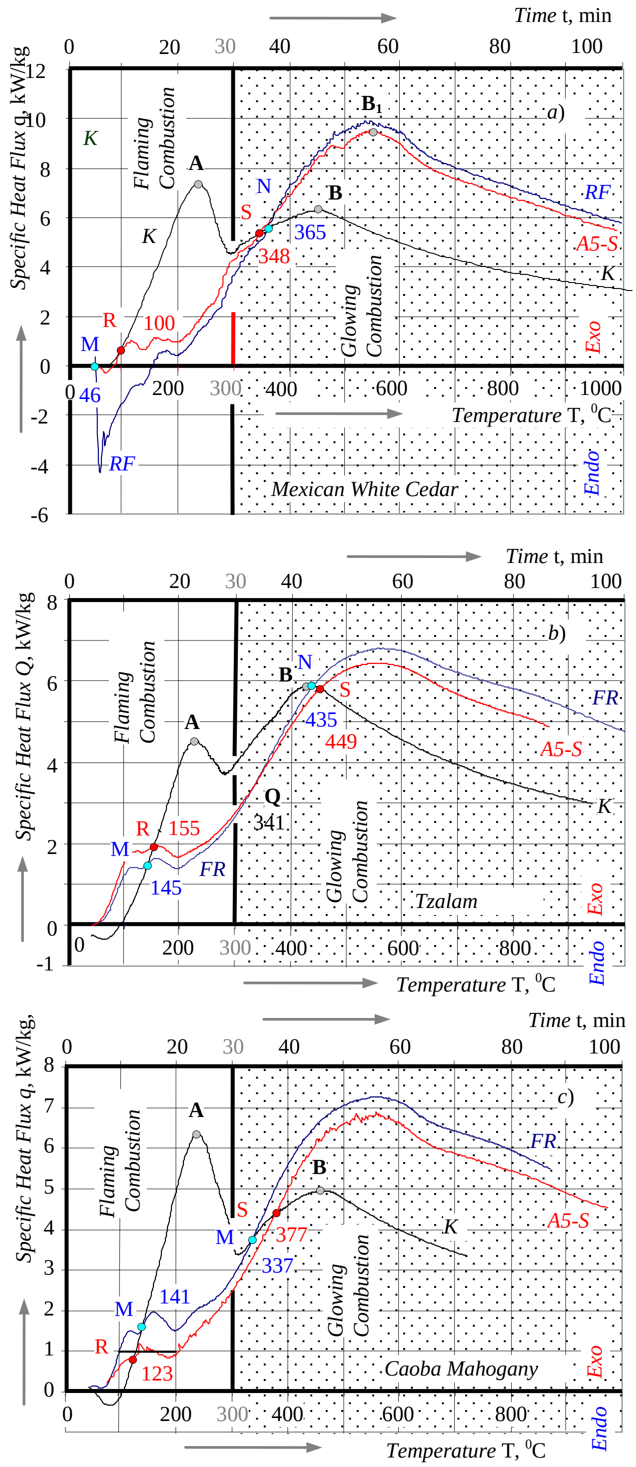

Fig. 5. *Criterion of wood pyrolysis and combustion behavior established on simultaneous thermal analysis (STA) - specific heat flux q spectra (per unit mass losses) of bare wood sample (K), flame retarded sample (FR), and plasma-aided flame retarded sample with FR-solution modified with anionic (5 vol. %) and siloxane surfactant (0.1 %) (A5-S):* ***a*** *- Mexican White Cedar (Cupressus Lusitanica);* ***b*** *- Tzalam (Lysiloma bahamensis);* ***c*** *- Mahogany Caoba (Swietenia macrophylla).*

Conclusion

The application of *STA* (*TGA* and *DSC*) allows evaluating the wood pyrolysis under heat influence by setting pyrolysis stage, temperature ranges and characteristic temperature peaks. Simultaneous thermal analysis defines and illustrates the impact of the used surfactants on *flame retardancy of wood*. The influence of surfactants on the flame pyrolysis and combustion resistivity refers to some of the unique behavior of wood. There are variations in wood behavior determined mainly by its heterogeneous and complex composition. Thus, in *Caoba mahogany* surfactants increase the flame combustion resistance by extending the temperature range while in *Mexican white cedar* negatively affects this resistance and in *Tzalam* surfactants almost have no influence on flame retardancy.

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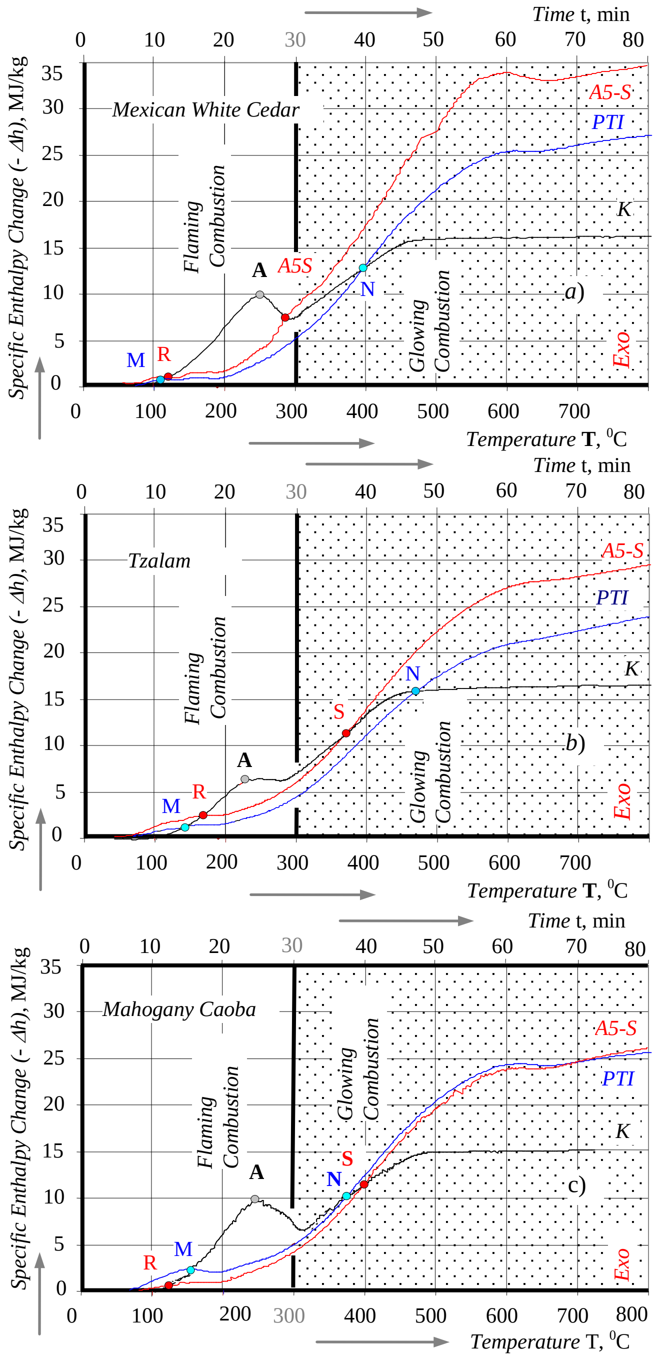

Fig. 6. *Criterion of wood pyrolysis and combustion behavior established on simultaneous thermal analysis (STA) - specific enthalpy change (-Δh) spectra (per unit mass losses) of bare wood sample (K), plasma-aided flame retarded sample (PTI) and plasma-aided flame retarded sample with FR-solution modified with anionic (5 vol. %) and siloxane surfactant (0.1 %) (A5-S):* ***a*** *- Mexican White Cedar (Cupressus Lusitanica);* ***b*** *- Tzalam (Lysiloma bahamensis);* ***c*** *- Mahogany Caoba (Swietenia macrophylla).*

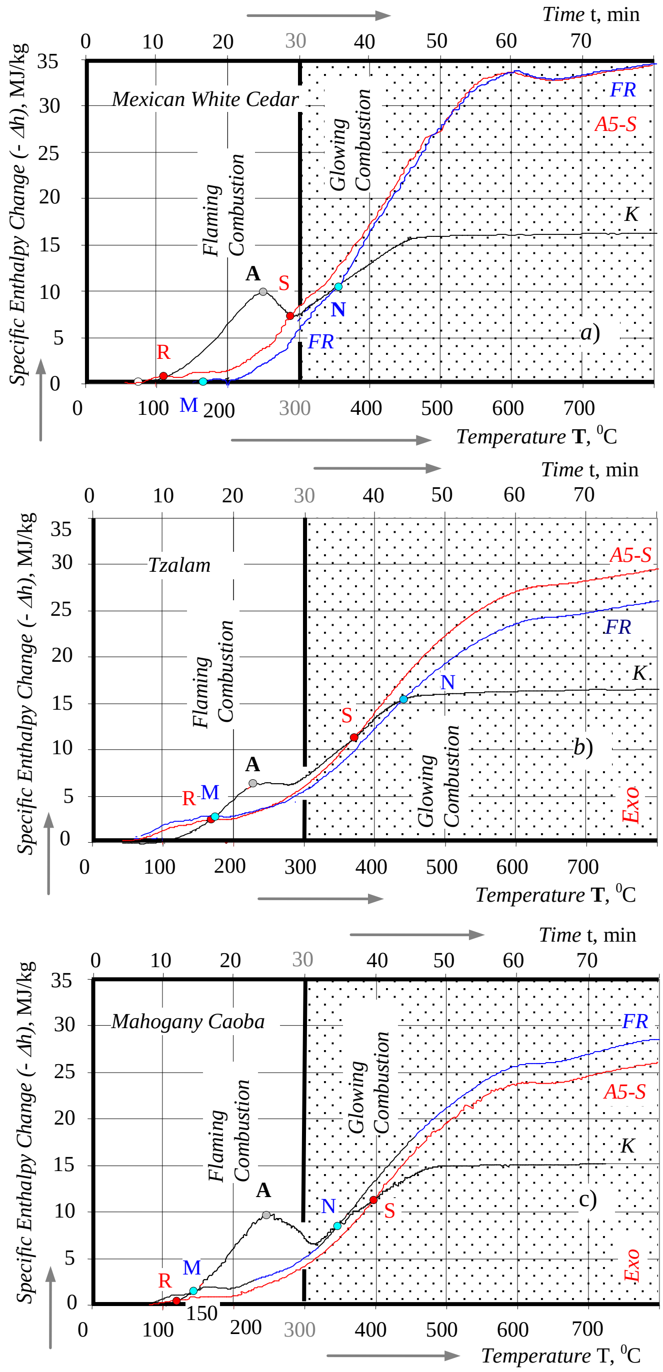

Fig. 7. *Criterion of wood pyrolysis and combustion behavior established on simultaneous thermal analysis (STA) - specific enthalpy change (-Δh) spectra (per unit mass losses) of bare wood sample (K), flame retarded sample (FR) and plasma-aided flame retarded sample with FR-solution modified with anionic (5 vol. %) and siloxane surfactant (0.1 %) (A5-S):* ***a*** *- Mexican White Cedar (Cupressus Lusitanica);* ***b*** *- Tzalam (Lysiloma bahamensis);* ***c*** *- Mahogany Caoba (Swietenia macrophylla).*

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