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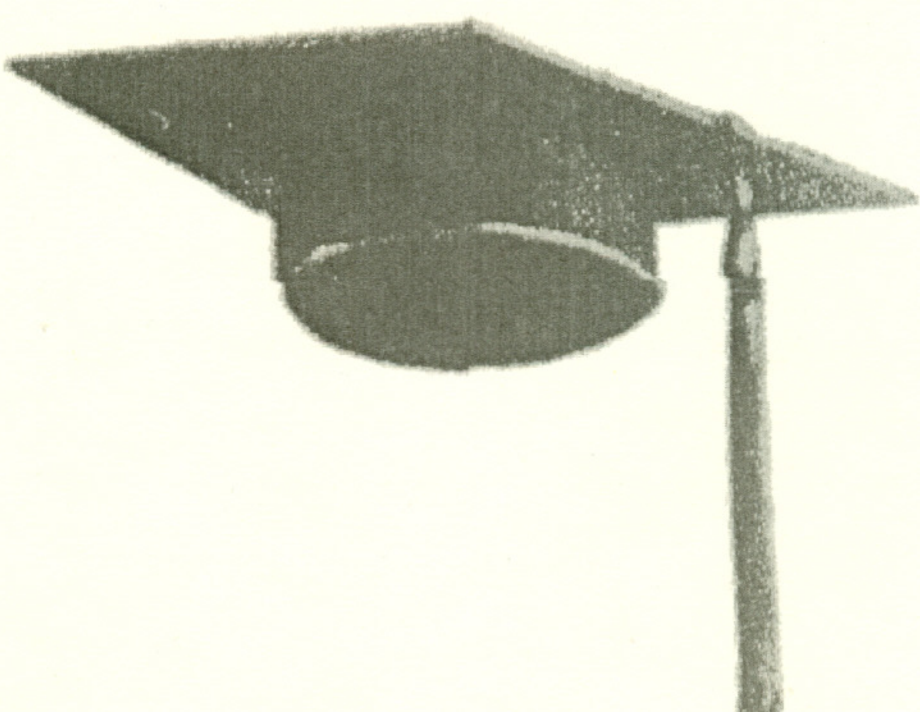
June 4-7, 2008, Sozopol, Bulgaria

eds. Nikolay Kolev, Lubomir Dimitrov,
Elena Helerea and Marieta Olaru



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PART I

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PLASMA AIDED IMPREGNATION TECHNOLOGY FOR FLAME RETARDED WOOD PRODUCING

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Abstract: This paper was developed as part of an investigation intended to analyze the thermal degradation reactions of flame retarded wood produced by different capillary impregnation technologies: flame retardant capillary impregnation, surfactant and spreader aided capillary impregnation, and cold plasma aided capillary impregnation. Two basic thermal methods of analyzing the degradation of wood are emphasized: thermogravimetric analysis (*TGA*) and differential thermal analysis (*DTA*), and differential scanning calorimetry (*DSC*). The decomposition of dry pine (*Pinus silvestris*, Bulgaria) wood was studied in a static air atmosphere at temperature below 600°C – the overflash fire point. The process of general thermal degradation for flame retarded wood is discussed and is followed by specific knowledge of the literature on thermal analysis studies of wood and its components.

Keywords: cold plasma surface activation (functionalization), differential scanning calorimetry, flame retarded wood and wooden products, flammability, ignitability, plasma aided technology, phosphor containing flame retardant, spreader, surfactant, thermogravimetric analysis

1. Introduction

A new fire protection technology, technical means and impregnating water solutions containing flame retardant (FR) under the form of phosphorous and nitrogen compounds have been developed, and their industrial approval has been achieved. Plasma-aided technology for capillary impregnation of porous media with flame retardants has been developed for wood, textile, and open pore foamed polymers. The plasma-aided capillary impregnation may take place in industrial conditions or on situ with the client, no matter where and how the object to be fire protected is located. A system of plasma applicators has been created to produce cold technological plasma through barrier electrical discharge at atmospheric pressure and room temperature. After plasma-chemical activation, or functionalization, that influences substantially the capillary activity of the porous material and its ion activity and capacity, an impregnation with fire-protective solutions is carried out within an open time of two to three hours. The plasma aided technology is patent protected and may be applied with different water solutions containing flame-retardants.

Flammability or "reaction to fire" is very imprecise term, however, it normally includes: ignitability, flame spread, and heat release rate.

However, it should be emphasized that two other important parameters which occur during a real fire, namely: smoke production and toxicity of gaseous products of thermal degradation and combustion, have to be taken into consideration when evaluating the influence of wood preservatives and modification on its flammability.

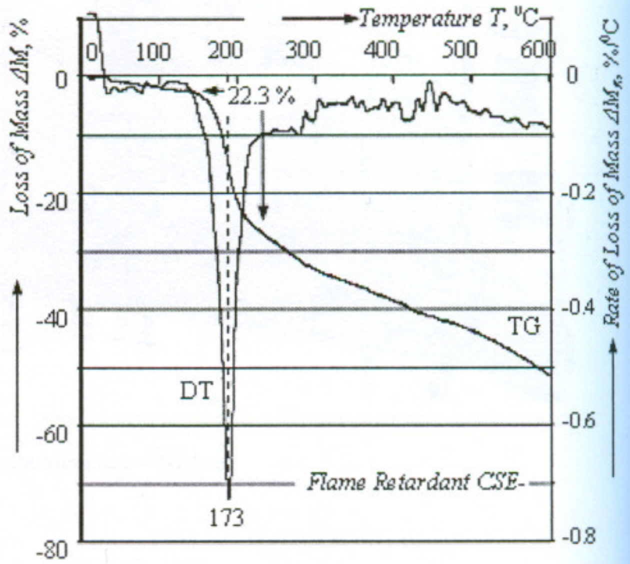


Figure 1. *TG* and *DT* thermograms of the flame retardant CSE-96 used (Interiorprotect, Ltd., Bulgaria).

Fire tests can be divided into the following main groups: a) fire resistance tests concerning the ability of *structure* to withstand the effects of fire and prevent its spreading; b) non-combustibility tests allowing pointing to *materials* which burn and those which do not; c) ignitability tests enabling to determine the ease with which *materials* ignite; d) reaction to fire tests determining such important parameters of *materials* as heat release rate, flame spread, etc; e) smoke production tests determining the ability of *materials* to produce smoke when on fire; f) tests for toxicity of

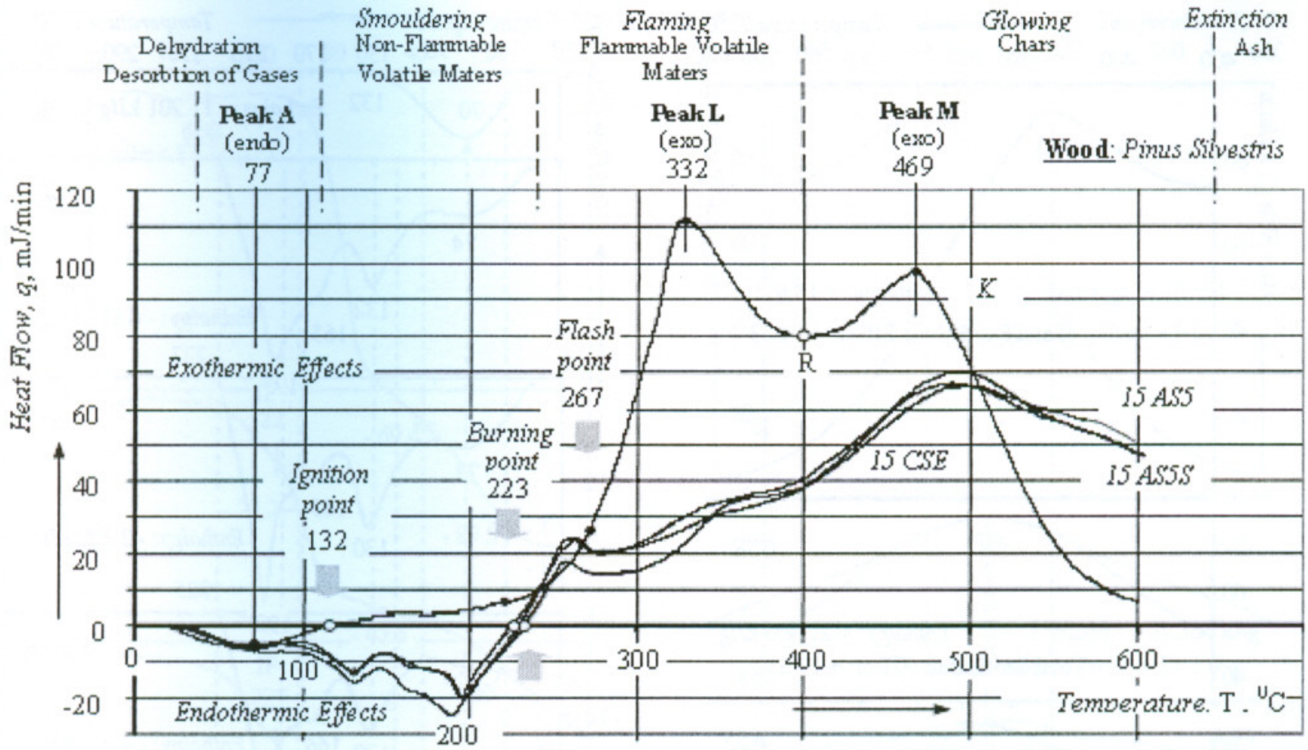


Figure 2. *DSC* thermograms (heat flow, *Q*, versus temperature, *T*) of natural pine (*Pinus sylvestris*) and flame retardant woods, produced by different plasma aided technologies: *K* - natural wood; *15CSE* - plasma aided capillary impregnation; *15AS5* - plasma and surfactant aided capillary impregnation; *15AS5S* - plasma, surfactant, and spreader aided capillary impregnation.

gaseous products released during thermal decomposition and combustion - they provide qualitative and quantitative analytical data on gases emitted by *materials* involved in the fire.

Thermal analyses - thermogravimetric analysis (TGA), differential thermal analysis (DTA), and differential scanning calorimetry (DSC) - give basic information on the mechanism of pyrolysis and combustion as well as data on the effect of wood treatment and modification. However, the correlation between results of thermal analysis and real fires has not been found yet.

The task of the present work consists in revealing the specific impact of the flame retardant, plasma-chemical surface functionalization, surfactants, and spreader on the thermal destruction and combustion of flame retarded wood.

2. Experimental Result and Discussion

The plasma aided flame retardation process involves three tools which are used for exerting direct impact on the thermal degradation of flame retarded wood: *the first one* consists in impregnating natural pine wood (*Pinus sylvestis*) with a 30-percent water solution of *FR-CSE* - sample *CSE*; *the second one* consists in adding 5 vol.% of anionic surfactant (*AS5*) to the impregnating solution *CSE-96* - sample *AS5*; *the third one* consists in performing cold plasma surface pre-treatment for 60 s in the plasma of dielectric barrier discharge (10 kHz) burning in air at atmospheric pressure and room temperature - sample *15CSE, 15AS5,* and *15AS5S*. The thermal degradation of all RF wood samples is compared with the basic sample made of natural pine wood (*Pinus sylvestris*). The thermal destruction of the *FR-CSE* used, which contains about 13.5 mass% (as P_2O_5), is investigated separately.

Experimental conditions for all runs were as follows: thermogravimetric (*TG*), derivative thermogravimetric (*DTG*), and differential calorimetric (*DSC*) analyses were completed by means of *Perkin-Elmer*'s equipment in air; the heating rate was 10 ^{0}C/min and a heating range from 26 ÷ 36 ^{0}C to 600 ^{0}C was used. All runs were done at least twice.

3. Conclusion

The experimental data analysis shown in Figures 1-5 has indicated that the proposed technology of plasma-aided capillary impregnation not only allows introducing larger amounts of flame retardant *CSE*-96, but also exerts a substantial impact upon the chemical interaction between the flame retardant and the wood matrix.

Acknowledgement

The financial support of the National Science Fund, Ministry of Education and Science of Bulgaria, for the Research Project VU-TN-09/2005 and 08027ni-1/08 are gratefully acknowledged.

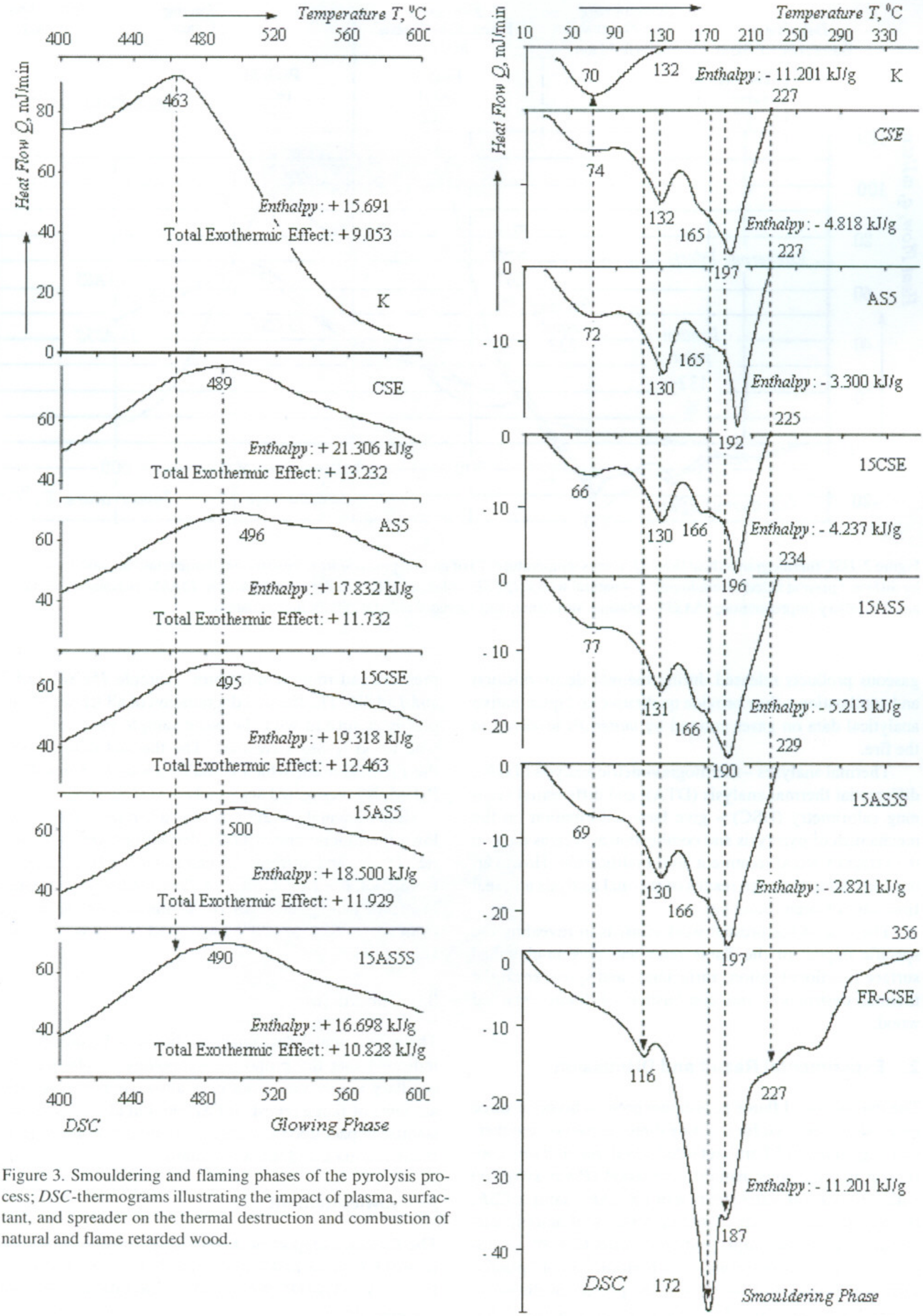
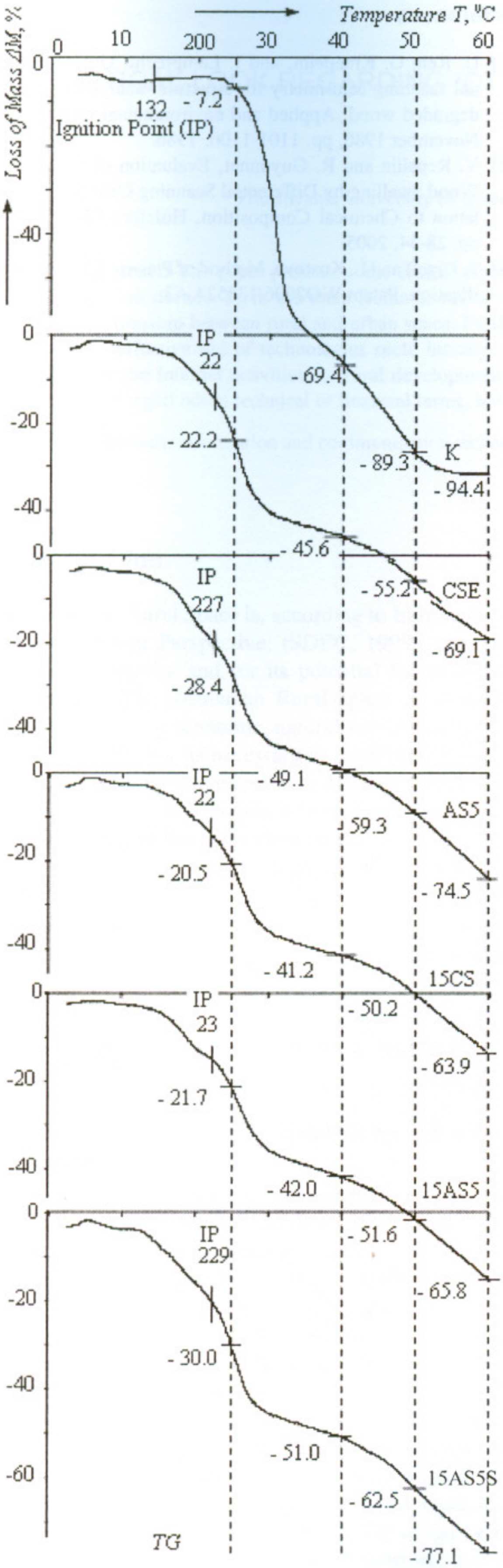


Figure 3. Smouldering and flaming phases of the pyrolysis process; *DSC*-thermograms illustrating the impact of plasma, surfactant, and spreader on the thermal destruction and combustion of natural and flame retarded wood.



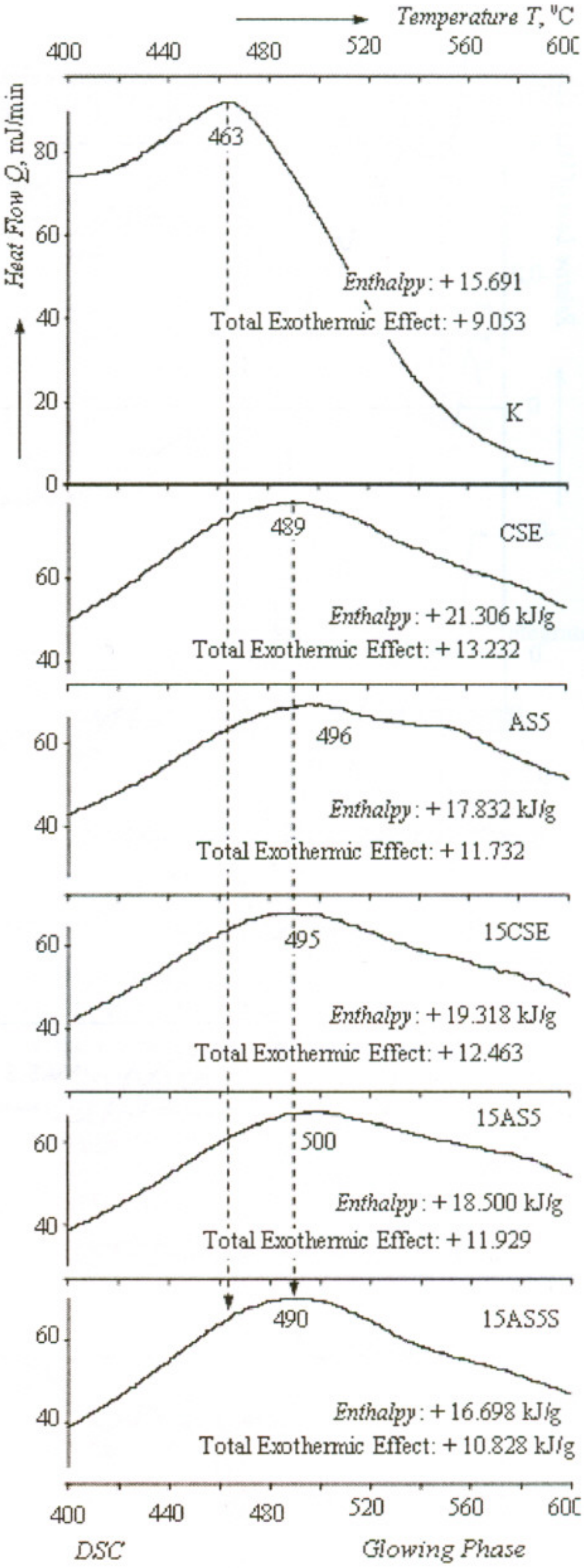


Figure 4. Glowing phase of pyrolysis process – *DSC*-thermograms; and *TG*-thermograms of all pyrolysis processes of natural pine wood – *K*, and different flame retarded woods – CSE, AS5, 15CSE, 15AS5, and 15AS5S.

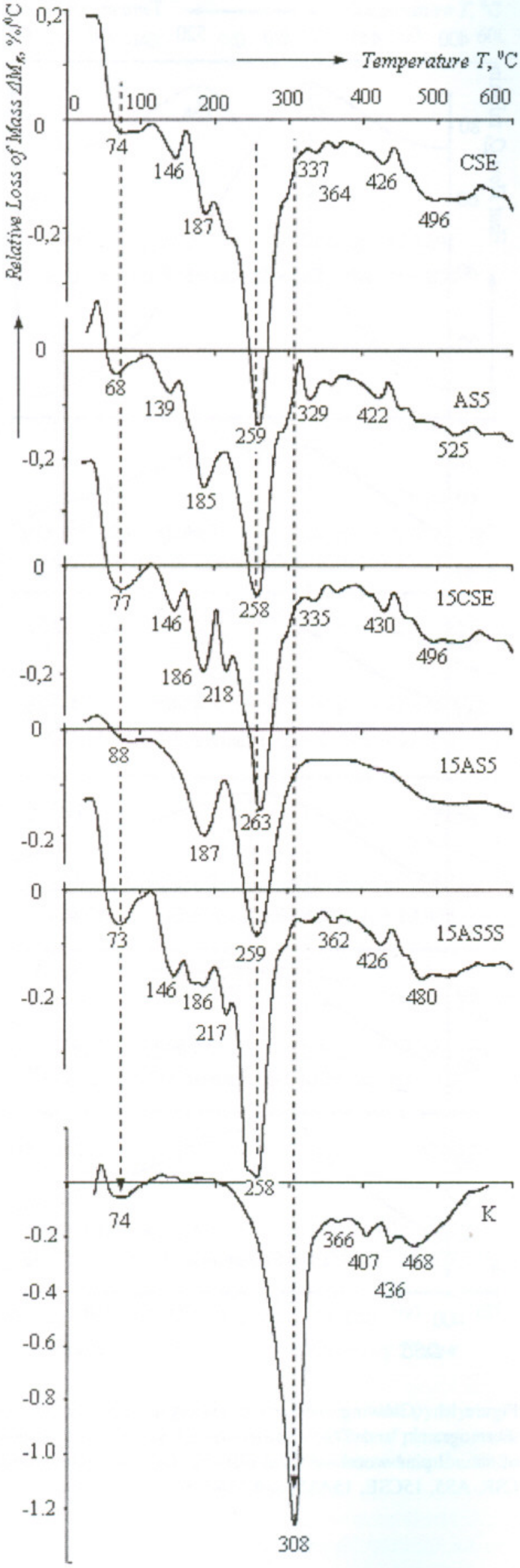


Figure 5. *DT*-thermograms of pyrolysis process.

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