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Characterization of Wood Composite Materials by Thermo-Gravimetric and Differential Scanning Calorimetry Methods

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Abstract
The paper aims to highlight the importance of using the 2 methods (thermo-gravimetric, differential scanning calorimetry, by characterization of wood composite materials (Oriented strand boards, Wood chipboard, Medium density fibreboard). Wood composites are widely used in the wood industry. By using th thermo-gravimetric method, the mass of composite sample changes by varying the work temperature (10 °C- 300 °C). By using the differential scanning calorimetry, the heat flow is measured depending on the change temperature (20 °C - 300 °C) at a certain moment. The thermograms obtained from the experiments indicate the deviation of the thermal energy released by the sample for the reference sample.

Keywords
Differential scanning calorimetry, thermo-gravimetric, oriented strand board, wood chipboard, medium density fibreboard

1. Introduction

Lignocellulosic composite materials fall into four categories, on the basis of the physical properties of the wood used in the manufacture of plywood, chipboard panels, oriented wide-chips panels, wood fiber panels. Improving the properties of the composite material can be achieved by means of: density adjustment, variation of the type and amount of resin and additives, in order to increase water and fire resistance.

At the European level, the consumption of composite materials increased until the year 2000, followed by a stagnation in the consumption of chipboard and plywood, and a significant increase in oriented strand board (OSB), medium density fibreboard (MDF) products. Consumption of wood-based boards decreased by 17.2% in North America and by 3.7% in Europe.

The composite materials are used for the furniture industry, musical instruments, civil engineering, having a series of advantages over other materials:

- resistance to high temperature;
- corrosion resistance;
- very good durability;
- higher strength and rigidity in relation to metals.

Recently, there has been a significant increase in the production of OSB, due to market demand. In Europe, the production of OSB is about 4 million m^3/year, carried out in the following new factories: Agglo, Kronobourgas, Isoroy, Kronofrance, Fritz, Egger, Glunz, Kronotex, SmartPly Europe Ltd, Kronospan, Kronopol Ltd, Nexfor Ltd.

The manufacturing process of OSB makes it economical:

- ensures the maximization of the use of forest resources (small diameter trees are used, which have low commercial value);
- it is a product characterized by uniformity;
- the orientation of the face chips gives high resistance along the panel.

By using long chips (75-110 mm long, 15-25 mm wide, 0.3-0.7 thick), the way of layering and orienting the chips, the adhesives used, determines the high strength of OSB boards (good internal cohesion, rigidity and water resistance).

OSB boards are classified into four categories: OSB/1 (general use indoor-dry), OSB/2 (load-bearing in dry conditions), OSB/3 (load-bearing in humid conditions), OSB/4 (for heavy duty inhumid conditions)

Poplar, pine, birch, maple are recommended as wood species for the manufacture of OSB. Adhesives used based on phenol-formaldehyde resins in liquid or powder form, are used for faces. PMDI (polymeric diphenyl-methane diisocyanate), isocyanate adhesives are used for the core as they form stronger bonds than phenol formaldehyde adhesives. An important property of these adhesives is that they harden al low temperatures (pressing time decreases). These adhesives provide good water and heat resistance.

Wood chipboards are obtained through the agglomeration of wood particles with adhesive, under the influence of pressure and temperature.

Wood chipboards have the following advantages:

- higher valuation of small wood varieties (sawdust, wood dust) and of some inferior wood species (willow, alder, poplar);
- the degree of anisotropy of the structure in all directions is reduced, because of the division of wood into chips.

In addition to advantages, wood chipboards also have disadvantages:

- water absorption and swelling in high thickness;
- the faces of these boards have no aesthetic value.

In Europe the main producers of wood chipboards are Germany, France, Italy, Spain, Russia, Poland, Ukraine, Belgium, Turkey, the Czech Republic.

The kinds of raw material used in the manufacture of chip board are: lobes, log heads, legs, scrap from the manufacture of furniture.

The characteristics of the boards are influenced by: the kind of raw material, moisture content, density, structure, chemical composition, swelling and shrinkage coefficients.

The technological flow of wood chipboard manufacturing includes peeling, chip shredding-cutting, particle sorting, drying, gluing, carpet forming, pre-pressing, hot pressing, formatting, cooling.

The wood kinds that cannot be cut into chips are transformed into wood chips, from which the chips required for the manufacture of wood chipboards are then cut. The optimal sizes of the wood chips are 18-25 mm long, 15-40 mm wide, 4-8 mm thick.

The particles used in the manufacture of the boards have different humidity (U = 12 - 140%). The humidity should be therefore reduced to a value between 2-8% depending on the adhesive applied and the structure of the boards made. Humidity reduction is achieved in drying installations using temperatures> 250 º C. Particle sorting may be carried out before and after drying. The purpose of the sorting is to provide the particle fractions required for the technological flow. The sorting may be performed with mechanical sieves or in air stream. The gluing process is influenced by the following factors: the characteristics of the particles subject to gluing, the characteristics of the adhesive used and the functional parameters of the glue application machines. The application of the adhesive is a continuous process which requires a thorough check of the chip mass and the adhesive amount. The adhesives used to glue the particles are urea-formaldehyde, phenol-formaldehyde, melamine-urea-formaldehyde, MDI (isocyanate type).

The main characteristics of the carpet which influence the forming technology as well as the characteristics of the boards are:

- the structure of the carpet, the orientation of the chips in the forming plane, the degree of compaction of the structure, the structure symmetry, the mass per unit area, the particle size composition per unit area, the loosening coefficient, the moisture of the chips in the carpet structure.

The most important parameters are the pressure, temperature, and pressing time. The temperature of the press trays varies between 200 - 220 º C. The specific pressing pressure varies between 2 - 4 MPa. Cold pre-pressing is carried out for the purpose of reducing the pressing time, decreasing the thickness

of the carpet. After cold pre-pressing for this carpets, pre-heating can be performed to increase the carpet temperature. Steam or hot air preheating is used.

Fiberboard results from the agglomeration of fibers and bundles of wood fibers and other lignocellulosic materials in the presence of a synthetic adhesive, under the action of pressure and temperature.

The production of MDF (Medim density fiberboard) has increased at a fast pace representing one of the most popular products.

During 2010, there were 57 % increases in capacity in Eastern European countries, 45 % in South American countries, between 21 and 30 % (North America and Asia). The largest producers of wood fiber boards are Canada, Italy, Spain, Malaysia, France, South Korea, Brazil.

The preparation of the raw material consists of peeling, chopping, sorting, storing and washing the wood chips. The optimal sizes of the wood chips are -30 25-30 mm long, 15-25 mm wide, 5 - 10 mm thick. By means of the Defibrator type installations, the plasticization of the wood chips with saturated steam is carried out. The heat treatment of the wood chips is conducted with saturated steam at 6 - 10 bar, at a temperature of 190 º C, for 3-7 minutes. Defibration is performed for fiber production, which have a large contact surface for gluing. After defibration, the fibrous material has a moisture between 50 - 120% and must be dried at a moisture of 5 – 15%. Drying is carried out with hot air or directly with flue gases at a temperature of 120-150 °C. The speed of hot air shall be between 20 - 40 m/s. Drying can be performed in installations with direct or indirect heating. After drying, the fibrous material is sorted. The amount of fibers resulting from sorting is stored in horizontal silos. In these silos the fibers are measured and dosed in order to form the carpet. The importance of carpet formation is materialized by the formation of a continuous layer on a support. In this case, air is the transport and formation environment.

Pressing is important for achieving M.D.F., and as important parameters are temperature (180 – 210 °C) and pressure (0.5-5 MPa).

[1] note the ways of performing differential scanning calorimetry as well as dynamic differential scanning calorimetry. Dynamic differential scanning calorimetry uses a sine wave while differential scanning calorimetry generates a sawtooth wave.

[2] underlines that the thermal system was analyzed, considering a thermal resistance between the test vessel and the base board. From the thermal analysis it was found that the calibration constant necessary to determine the heat capacity is given strictly in terms of temperature amplitude ratios.

[4] show in the study that the difference in heat flow is measured compared to a reference material as a function of temperature. Sodium chloride dipolyphosphate and tripolysphate in the concentrations used affect the incubation time.

[6] emphasize in the study the fact that reverse and non-reverse heat flow exchanges showed that enthalpy relaxation took place in dry wood. The structure of dry wood has a rigid amorphous state, due to the interactions between the wood components.

[7] underline in the study that rectification is a treatment which reduces the swelling of wood and increases its resistance to fungal attack. In this study, differential scanning calorimetry was applied in order to determine the fiber saturation point. The influence of temperature and duration of heat treatment on wood swelling was investigated.

[6] point out that the thermal properties of solid and ground wood at different humidity conditions have been investigated by modulated differential scanning calorimetry. The transitions of ground wood occurred at lower temperatures than those of solid wood at a similar moisture content.

[8] note in the study that mangrove wood is recognized as a potential biomass resource. The thermal characteristics of mangrove wood were studied by conducting thermogravimetric analysis. Volatile substances, fixed carbon and ash content were found to be 55.66%, 21.6% and 7.24% respectively.

[9] presented in the study the results of a thermogravimetric analysis of four thermally modified wood species. Thermally modified wood was used for the tests before and after the extraction process. The extraction process had no effect on the thermal characteristics of any of the wood species.

[10] outlines in the study a special method of thermogravimetric analysis. This analysis was used to the mass percentage of wood flour and polypropylene copolymer in the composition of the composite-plastic material (WPC).

[11] point out in the study that the thermogravimetric method was a useful tool for rapid estimation of components in both solid wood and wood attacked by brown rot.

[12] show in the study that the decomposition of hemicellulose, cellulose and lignin takes place in a limited temperature range. The following wood species were taken into consideration: *Castanea sativa*, *Eucaliptus globulus*, *Quercus robur*, *Pinus pinarth*, *Pinus sylvestris*. The FTIR spectra obtained from the assessed gases helped to classify the wood species according to the degradation areas.

[13] describe in the study the thermal properties of oriental beech sawdust (*Fagus orientalis*) treated with 0.25% solution of Adolit KD-S, 1%, Wolmanit CX-8 solution and Tanalit-E solution 4.70%. These experiments were carried out using thermogravimetric analysis under argon atmosphere. The results were compared with untreated wood (control). The experiments have shown that treatments with the respective solutions decreased the maximum degradation temperature and increased the amount of residual carbon compared to the control sample.

[14] emphasizes in the study the effects of glycerol pre-treatment and thermal modification on poplar wood. These effects were examined using thermogravimetric analysis. The total mass losses of the heat-treated samples before and after impregnation with glycerol were studied. The study underlines that pre-treatment with glycerol is useful for the thermal stability of wood.

[17] highlighted in the study the thermal properties of beech wood treated with salts and their mixtures in aqueous solutions at a concentration of 3%. Salt mixtures containing phosphates have remarkable effects on the thermal stability of beech wood, while NH_4Cl salt has lowered the decomposition temperature.

2. Materials and Methods

Differential scanning calorimetry analysis allows the establishment of: solidification, crystallization, or melting behavior, degree of crystallization, thermokinetic analysis.

The differential scanning calorimetry was performed using DSC 200 F3 MAIA equipment. The weight of the samples are between 15-20 mg, were heated at a constant rate 10 ºC/min, up to 200 ºC under nitrogen atmosphere (Figure 1).

Fig. 1. DSC 200 F3 MAIA equipment

Thermo-gravimetric method analysis allows the determination of : mass changes, stability at certain temperatures, compositional analysis.

The thermal analysis was studied using the STA 449 F3 Jupiter analyzer instrument. The temperature range used was from 25 to 500 °C. The heating rate was 10 °C/min under nitrogen atmosphere at a gas flow rate of 20 ml/min (Figure 2).

3. Results and Discussion

By dilatometry scanning calorimetry analysis in the case of sample 11 of melamine faced chipboard it is noted that at a temperature of 105.8 °C, the process of heat absorption (endothermic reaction) of

34.98 J/g occurs. At a temperature of 169.8 °C, the process of heat release (exothermic reaction) of 7.67 J/g occurs. At a temperature of 239.3 °C, the heat absorption process (endothermic reaction) of 0.1817 J/g occurs.

Fig. 2. STA 449 F3 Jupiter equipment

By dilatometry scanning calorimetry analysis in the case of sample 12 of melamine faced chipboard it is noticed that at a temperature of 93.7 °C, the process of heat absorption (endothermic reaction) of 37.35 J/g occurs. At a temperature of 159 °C, the process of heat release (exothermic reaction) of 43.47 J/g occurs. At a temperature of 239.3 °C, the process of heat absorption (endothermic reaction) of 1.954 J/g occurs (Figure 3).

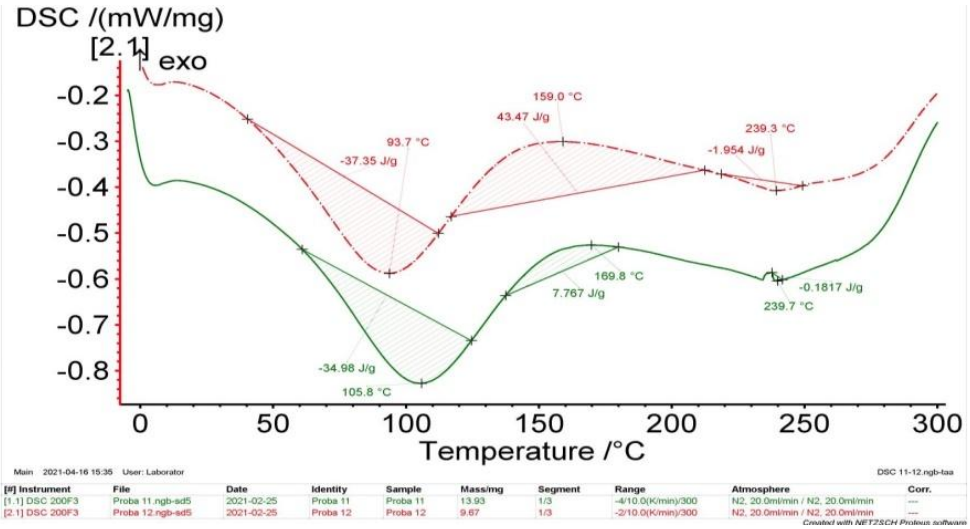

Fig. 3. Dilatometry curves for samples 11 and 12 of melamine faced chipboard

By dilatometry scanning calorimetry analysis in the case of sample 4 of OSB, it can be noticed that at a temperature of 97.1 °C, the heat absorption process (endothermic reaction) of 25.24 J/g occurs. At a temperature of 172.3 °C, the process of heat release (exothermic reaction) of 1.652 J/g occurs.

By dilatometry scanning calorimetry analysis in the case of sample 5 of OSB, it can be noticed that at a temperature of 72.7 °C, the heat absorption process (endothermic reaction) of 14.7 J/g occurs. At a temperature of 146.5 °C, the process of heat release (exothermic reaction) of 41.52 J/g occurs (Figure 4).

By dilatometry scanning calorimetry analysis in the case of sample 6 of MDF, it is noticed that at a temperature of 97.3 °C, the heat absorption process (endothermic reaction) of 25.24 J/g occurs. At a temperature of 155.3 °C, the heat release process (exothermic reaction) of 23.07 J/g occurs. At a temperature of 227.6 °C, the process of heat absorption (endothermic reaction) of 3.555 J/g occurs.

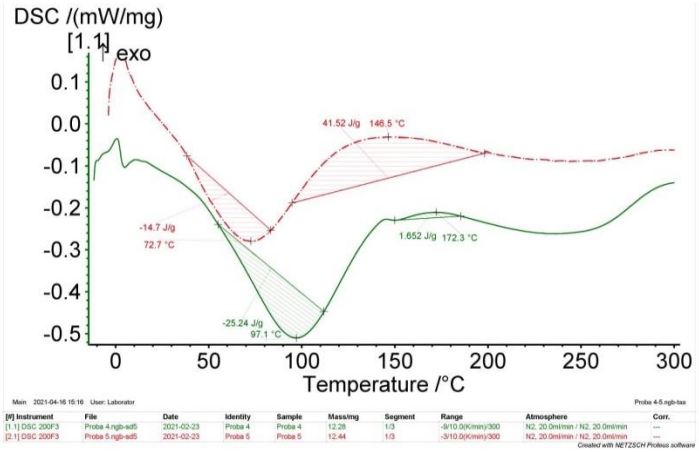

Fig. 4. Dilatometry curves for samples 4 and 5 of OSB

By dilatometry scanning calorimetry analysis in the case of sample 10 of MDF, it is noticed that at a temperature of 93.7 °C C, the heat absorption process (endothermic reaction) of 11.42 J/g occurs. At a temperature of 151.4 °C C, the process of heat release (exothermic reaction) of 11.42 J/g occurs. At a tempearature of 228.3 °C, the heat absorption process (endothermic reaction) of 5.186 J/g occurs (Figure 5).

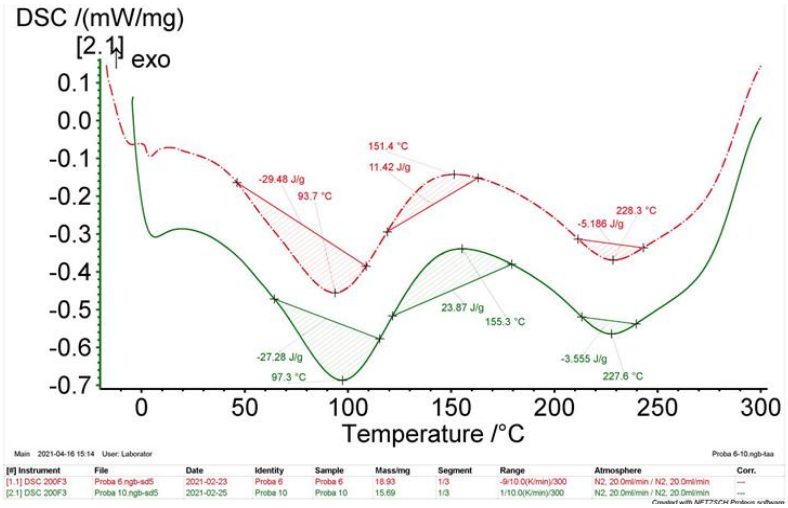

Fig. 5. Dilatometry curves for samples 6 and 10 of MDF

With thermo-gravimetric analysis, due to the working principle that has a built-in analytical balance which measures real-time mass changes in the samples taken, the calculations are accurate.

For sample 11 of melamine faced chipboard, analyzing thermo-gravimetric, it is noticed that up to 100 ºC, 1.69% of the mass has been lost, and in the temperature range 100-250 ºC, 6.96% of the mass has been lost. In the last temperature range 250-300 °C, 14.32% of the mass was lost (Figure 6).

For sample 2 of OSB, analyzing termo-gravimetric, it is noticed that up to 100 °C, 0.76% of the mass has been lost, and in the temperature range 100-250 °C, 1.36% of the mass was lost. In the last temperature range 250-300 °C, 11.13% of the mass was lost (Figure 7).

4. Conclusions

The temperature variation must cause a change in the mass of the sample, so thermo-gravimetric is limited to decomposition and oxidation reaction and the physical processes analyzed may be evaporation, sublimation, and desorption.

Through thermo-gravimetric we obtain information about the mass change of a material, but the origin of these changes must be investigated with additional techniques.

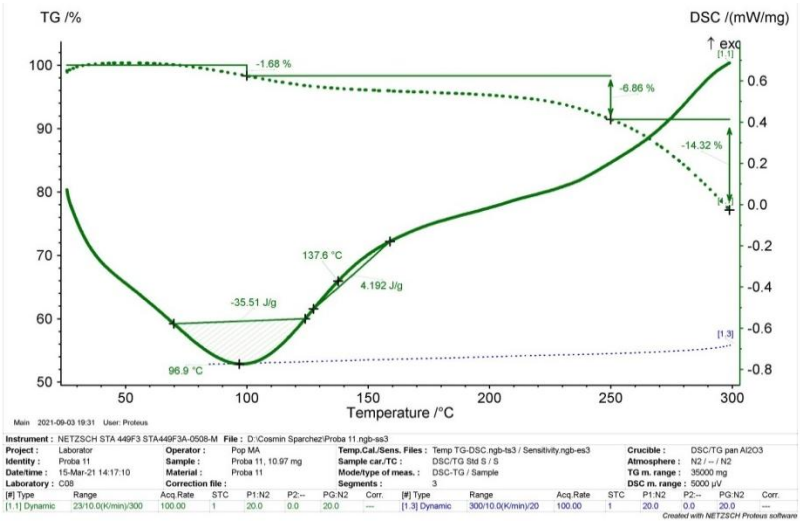

Fig. 6. Thermo-gravimetric analysis for sample 11 of melamine faced chipboard

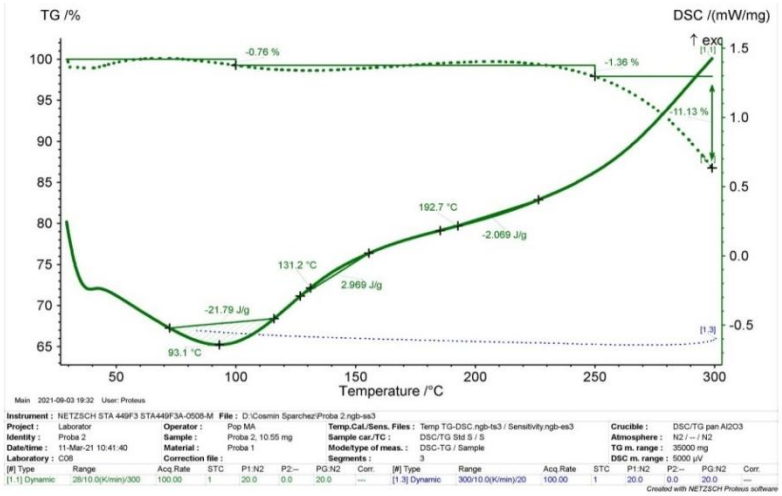

Fig. 7. Thermo-gravimetric analysis for sample 2 of OSB

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