TESTING OF ADHESIVES CAPACITY FOR SOME SYSTEMS BASED LIGNOCELLULOSIC COMPOSITES BY ASSESSING THE MECHANICAL PROPERTIES

TESTAREA CAPACITATII ADEZIVE A UNOR SISTEME PE BAZĂ DE COMPOZITE LIGNOCELULOZICE PRIN EVALUAREA PROPRIETĂŢILOR MECANICE

UNGUREANU Elena[1], TROFIN Alina[1], JITĂREANU Doina [1], POPA I.V. [2]
e-mail: elnungureanu@yahoo.ro

Abstract. *The lignin stands out by a very large range of applications in extremely various domains. Choosing to use the impregnation of some slips of filter paper in the case of the method for assessing the adhesive capacity of the resulted composite structures is justified by the necessity of pointing aut the power of interaction of the utilized adhesives with the cellulose fibres from the structure of the wood. In order to dispose of the dificullty concerning the interaction of the substrate with the utilized reagents there have been used in all the cases reference samples of the substrate which have undergone identical treatments excepting the utilized adhesive.*
Key words: *adhesive systems, furan resins, furfuryl alcohol, lignin, filter paper.*

Rezumat. *Lignina se remarcă printr-o gamă foarte largă de aplicaţii în domenii extrem de diverse. Opţiunea pentru utilizarea prin impregnare a unor benzi de hârtie de filtru în cazul metodei pentru evaluarea capacităţii adezive a structurilor compozite create, are ca justificare necesitatea evidenţierii capacităţii de interacţiune a adezivilor utilizaţi cu fibrele celulozice din structura lemnului. Pentru a se elimina neajunsul interacţiunii substratului cu reactivii utilizaţi în toate cazurile s-a recurs la raportarea faţă de probe martor ale substratului ce au fost supuse unor tratamente identice cu excepţia adezivului investigat.*
Cuvinte cheie: *sisteme adezive, răşini furanice, alcool furfurilic, lignină, hârtie de filtru.*

INTRODUCTION

For a material to perform as an adhesive it must have four main requirements:

- It must "wet" the surfaces - that is it must flow out over the surfaces that are being bonded, displacing all air and other contaminates that are present.
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- It must adhere to the surfaces - That is after flowing over the whole surface area it must start to adhere and stay in position and become "tacky".

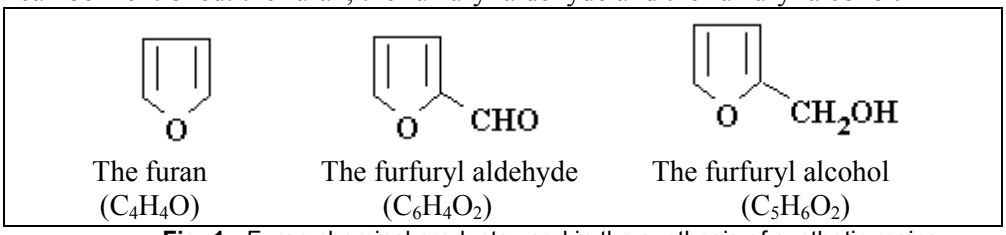
[1] University of Agricultural Sciences and Veterinary Medicine of Iasi, Romania
[2] "Gh. Asachi" Technical University of Iasi, Romania

- It must develop strength - The material must now change its structure to become strong or non-tacky but still adherent.
- It must remain stable - The material must remain unaffected by age, environmental conditions and other factors as long as the bond is required (Popa et al., 2007).

As an adhesive, lignin should behave similarly with the phenolformaldehyde resin due to its polyphenolic structure. These properties are characteristic to native lignin, whereas the technical lignins in order to be transformed into insoluble resins have to be additionally crosslinked (Ungureanu, 2011).

The catalysts or accelerators are chemicals added in small amounts to increase the rate of chemical reaction in the curing or hardening process. Maleic anhydride was used as crosslinking agent.

The furan resins represent an important class of synthetic resins which have as a starting point chemical substances having a furan type structure. Among the basic chemical products used for the synthesis of such synthetic resins there can be mentioned: the furan, the furfuryl aldehyde and the furfuryl alcohol.



The furan	The furfuryl aldehyde	The furfuryl alcohol
(C_4H_4O)	($C_6H_4O_2$)	($C_5H_6O_2$)

Fig. 1 - Furan chemical products used in the synthesis of synthetic resins

With respect to the furfuryl alcohol, it is known the fact that it, can be obtained in industry by the hydrogenation of furfuryl in the presence of the selective nikel Ramy catalysers, platinum oxyde, which favours the hydrogenation of the aldehyde functional group and of the furan nucleus (Yelle et al, 2004; Meister, 1996).

Assessing the adhesive characteristics of the furfuryl alcohol and of various furan resins, by impregnating support materials followed by the development of some reticular reactions is not a recent technique but rather a permanently developed perfected and up-to-date technique (Ungureanu, 2011).

MATERIAL AND METHOD

The following materials have been used:

- Wheat straw lignin (L_1), offered by Granit Recherché Dévelopement (relative humidity (%) - 5.00, ash (%) – 2.30, pH in suspension- 2.70, solubility in acids (%)- 1.00);
- Furan resin BioRez91ME (R_1) (resin produced by Trans Furan Chemicals, with a broad curing spectrum ranging from 130°C to 200°C , relative humidity-36%);

- Furfuryl alcohol (aspect – oily liqhid; colour – yellowish colourless; density, g/cm^3 – 1,1296; boiling point, ^{0}C – 171,750; refraction index, n^{20}s – 1,4845; toxicity – 50 cm^3/m^3 aer), (Trans Furan Chemicals bvba);
- Maleic anhydride (Fluka) MA.

Work procedure*:* Lignin solutions necessary for adhesive formulation were obtained by dissolution of lignin in furfuryl alcohol (FA). It was prepared lignin solutions of different concentrations, as: 20%, 30%, 40%, 50%. The performed tests concerning the solubility of lignin in FA showed a almost complete dissolution of lignin in FA.

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The alcoholic lignin solution was thoroughly mixed with a hydrophilic furan (prepolymer) BioRez 91ME with a solid content of 64% in a weight ratio 1:1. The catalyst (maleic anhydride) can be dissolved directly in furfuryl alcohol before lignin addition or it is possible to be added as the last component (powder) before adhesive application and mixed in well.

The testing procedure based on paper filter impregnation is presented in figure 2.

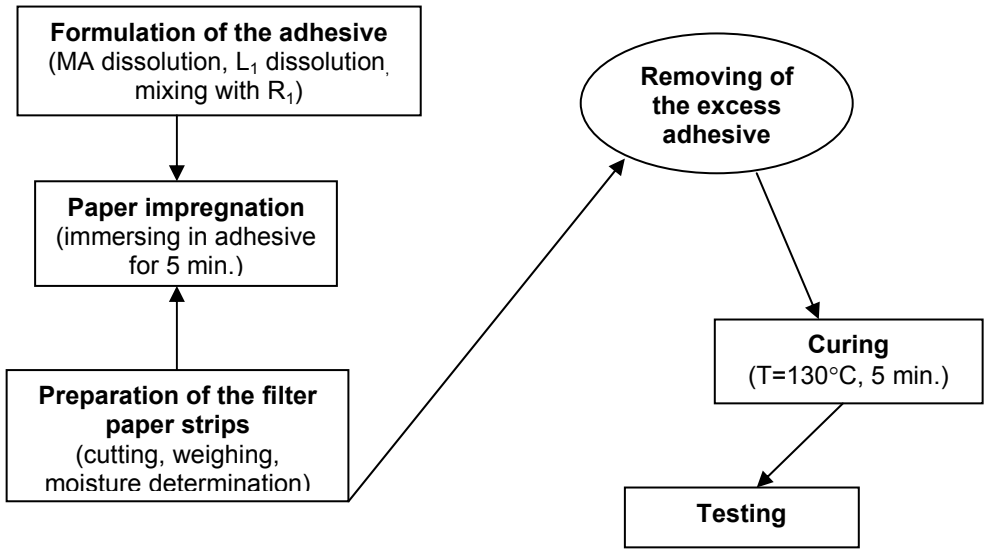

Fig. 2 - Testing procedure for evaluation of adhesive systems - based on paper filter impregnation

The impregnation was followed directly by curing. Curing was done in a hot-air oven at 130°C for 5 min. The oven dry mass of each sample of strips was determined so that adhesive uptake (wet and dry) could be determined. Finally, the samples were conditioned at 23.0 ± 1.0°C and 50.0 % ± 2.0% relative humidity until constant mass was achieved, before being tested.

The tensile strength and elongation at break of each sample were measured using an Instron Universal machine, with a cross head speed of 10 mm per min. A comparison of properties was made with the control resin. A sample of the filter paper, not impregnated by resin, was tested as an indication of the properties of the paper

substrate. The tensile strength and elongation at break of each sample was measured.

A standard procedure has been used to determine the bending stiffness of impregnated paper strips by measuring the resistance to slight bending (Tappi Test Method T-535 cm-85 Stiffness of paperboard – resonance length method).

RESULTS AND DISCUSSIONS

In the first trials, the filter paper strips (80 g/m^2) were of 200 mm long and 80 mm width, with the length being cut along the machine direction of the paper. For paper tensile strength test have to use 15 mm wide strips. Tensile strength test method is very specific about sample cutting. That means to use a special cutter for 15 mm wide samples after the curing and conditioned. However, the extra cutting process proved to be a source of micro cracks in edge of the impregnated paper specimens.

In the second series of trials we use for impregnation filter paper strips of directly 15 mm width. The filter paper strips were impregnated with different adhesive products and their mixtures, by completely immersing in adhesive for 300 s. The excess resin was removed from each strip by running the sample through a 0.25 mm gap. The edge defect was removed from each strip by suction with filter paper No crakes on the strip edges appear in this case. The appearance of the breaking zone are normally.

Fig. 3 – Testing of mechanical properties on the filter paper impregnated with adhesive lignocellulose systems

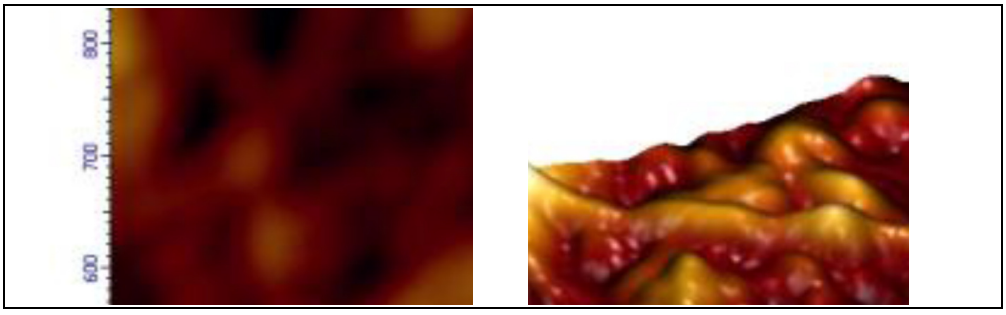

Fig. 4 - 2D-image (left) and 3D-image (right) for filter paper treated

The experimental data have a good dispersion and follow a normal distribution.

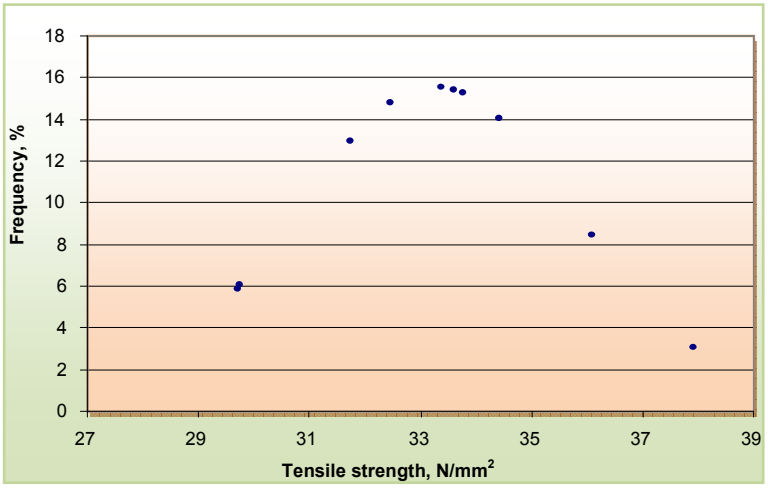

Fig. 5 - Testing for distribution of the dispersed particles

By adding lignin in resin formulation a cross-linking process occurs, aspect demonstrated by the insolubility of the final product and by increasing of the tensile strength.

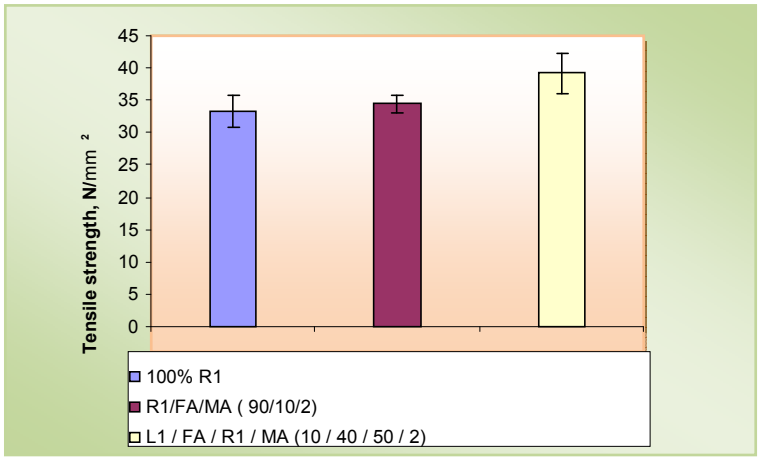

Fig. 6 - Tensile strength for the samples impregnated with adhesives

For addition levels up to 20% lignin ($L_1$10%, $L_1$15%, $L_1$20%.), the tensile strength of the resins was equal or better than that observed for the pure furan resin. The low concentration (10-40% L_1/FA) alcoholic solutions from unmodified lignin demonstrated a high stability on over test duration.

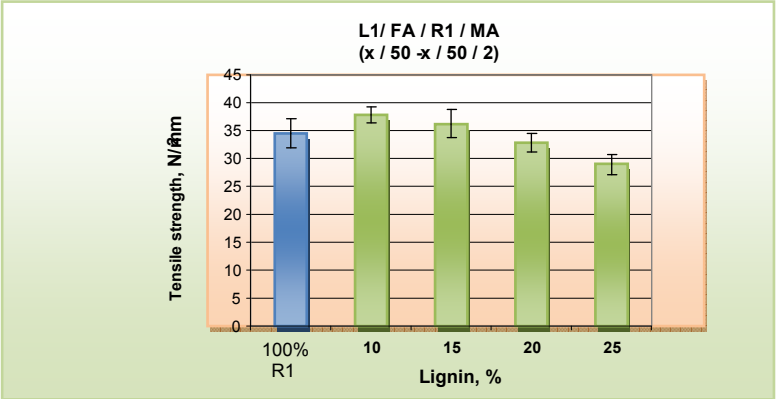

Fig. 7 - The evolution tesile strength depending on the content of lignin

CONCLUSIONS

1. The procedure developed proved to be a very useful tool in evaluation of adhesive system because:
   - give reproducible results with good dispersion (normal distribution);
   - it is sensible to chemical modification of lignin;
   - permitted us a relatively complete evaluation of adhesive;
2. The weak points of the procedure are:
   - impossibility to test adhesives with high viscosity (it was not possible to test a formulation adhesive with 25% lignin);
   - strong dependence of experimental results of solubility of lignin.
3. The use of furan products (furfuryl alcohol and furan resins), together with the cellulose fibers from the structure of paper and implicitly of wood contributes to the achievement of new adhesive system having superior proprieties and which are likely to be applied in various domains.
4. On the one hand, as the concentraton in the adhesive grows, the level of impregnation improves and on the other hand the level of retention lowers.

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