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Green gluing of tropical wood Part III: X-Ray Photoelectron Spectroscopy (XPS) and Fourier Transform Infrared Spectroscopy (FTIR) analysis of Frake and Ayous green wood's Glue line.

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Abstract:

We have recently succeeded to bond two tropical African woods Ayous and Frake in the green state by the glulam technique using a one component (1C-PU) polyurethane adhesive. Durable bond line was achieved and succeeds to meet the structural standard requirement.

Scanning electron microscopy performs on the glue line show a good embedding of the glue on the wood fibers. X-ray photoelectron spectroscopy also referred to as Electron spectroscopy for chemical analysis (ESCA) and Fourier transform infrared spectroscopy were used to investigate the interaction of both tropical woods with adhesive. Unlike theoretical expected urethane functional group, xps analysis indicated a high proportion of non hydrolyze urea group and hydrogen bonding of nitrogen (N) at 399,5 eV and 400,5 eV respectively. This foreseen that polyurea polymers and hydrogen bond are the most probable bond to strengthen the glue line

Key words: Green gluing, Tropical woods, Sanding, 1C-PU, XPS, FTIR, Polyurea

1- Introduction

Wood is one of the most abundant materials in the Congo large forest reserve. It has remarkable technological potentials, which explains its use throughout in various forms: massive timber, composites or massive reconstituted wood. In civil engineering structures, composites and reconstituted sawn timber (glulam), generally obtained by gluing, are the most suitable products used when high mechanical performances are required.

Process of woods bonding is usually achieved through the application of an adhesive on wood surface. Among the different mechanisms involved in the bonding of woods, two synergistic effects seem essential to achieve a good bonding strength: the penetration and anchoring of the adhesive in the void spaces of the wood surface and the occurrence of a strong interaction or chemical reaction between the adhesive and the major constituents of wood. This latter condition is governed by the surface properties that depend not only on the bulk composition, but also in the surface building process. In this study, X-ray Photoelectron Spectroscopy (XPS) also referred to as Electron

Spectroscopy for chemical analysis (ESCA) and Fourier Transform Infrared Spectroscopy (FTIR) which have proved to be two suitable investigation methods to characterize the composition of a material surface have been used. These methods are powerful analytical and non-destructive techniques which have already been used for the analysis of the surfaces of natural or synthetic polymers [1], natural and textile fibers [2],[3],[4], [5]; and in the characterization of wood surfaces and wood reactions [6]. They were used to elucidate the mechanism of bonding of these woods with a one-component polyurethane adhesive (1C-PU)

2- Materials and methods

2-1. Samples preparation

Beams of two unseasoned tropical woods respectively ayous and frake were recently obtained by gluing six wood lamellae (single and mixed) with a one component polyurethane adhesive (1C-PU) [7]. Specimen for XPS and FTIR analysis were prepared by cutting samples of $10 \times 10 \times 2 \text{ mm}^3$ from mixed and single wood beams. Cutting was performed such that all samples contained glue line.

2-2. Samples analysis

XPS analysis was performed using an ESCALAB VG 220i-XL spectrometer fitted on a microlab system from Vacuum Generators and equipped with a condition source of non monochromatized $\text{Mg K}\alpha$ anode X-rays source with an excitation energy of 1253,6 eV. K

Kinetic energy measurements were made using a hemispherical electrostatic analyzer ($r = 150 \mu\text{m}$) and a detector pass energy of 150 eV. Thin samples having a thickness of (2mm) or less were mounted onto an indium sample older and introduced in the working chamber maintained at a pressure of 10^{-7} Pa. Under these conditions, the order of the magnitude of the X-rays penetration is estimated within 1-10 nanometers. Electrons from the inner part of the sample are recaptured by the charging effect of the surface. Infrared spectroscopy (IR) is a nondestructive analytical technique to determine, identify or confirm the presence of functional groups. In this case, this technique has been used to confirm the observations made using the XPS analysis highlighting the presence of functional groups on the surface characteristics of materials and possibly the chemical reactions on the substrate-polymer interface. The FTIR measurement were performed using an infrared spectrometer Fourier transform Magna IR 550 coupled to an MCT detector cooled with liquid nitrogen. The accessory "ATR" is placed in the sample compartment. A microscope allows focusing on the desired wood analysis area.

3- Results and discussion

Figure 1(A) and (B) shows the XPS survey of ayous and fraké glue line. As expected, the spectrum is dominated by carbon, oxygen and nitrogen peaks, since polyurethane is highly reactive with hydroxyl groups to form polyurethanes. The high-resolution spectrum of the carbon peak shows the presence of different chemical states, or classes, of carbon in the wood joint. Other elements such as silica appear as traces in glue joints of the two species.

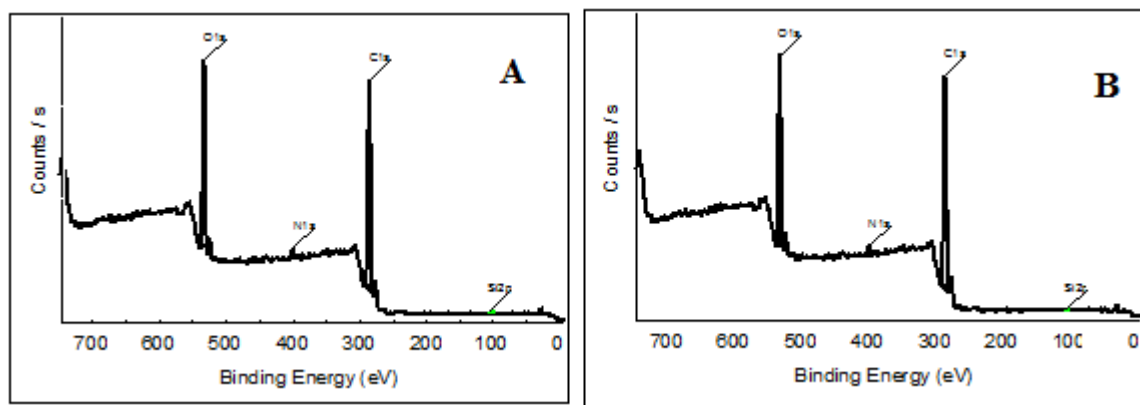


Figure 1: Survey of ESCA spectra for : (A) Ayous glue joint and (B) Fraké glue joint

The binding energies of different chemical elements present in the two species are summarized in Table 1.

Table 1: Binding energy of different chemicals elements on ESCA (XPS) survey spectra

Wood species	Binding energy ($\pm 2eV$)		
	C _{1s}	O _{1s}	N _{1s}
Ayous glue joint	284,89	532,01	399
Frake glue joint	284,68	532,01	398,57

3-1. Peak Assignments

In order to get more information, O 1s, C 1s and N1s peaks have been expanded and deconvoluted into their component peaks (figure 2)

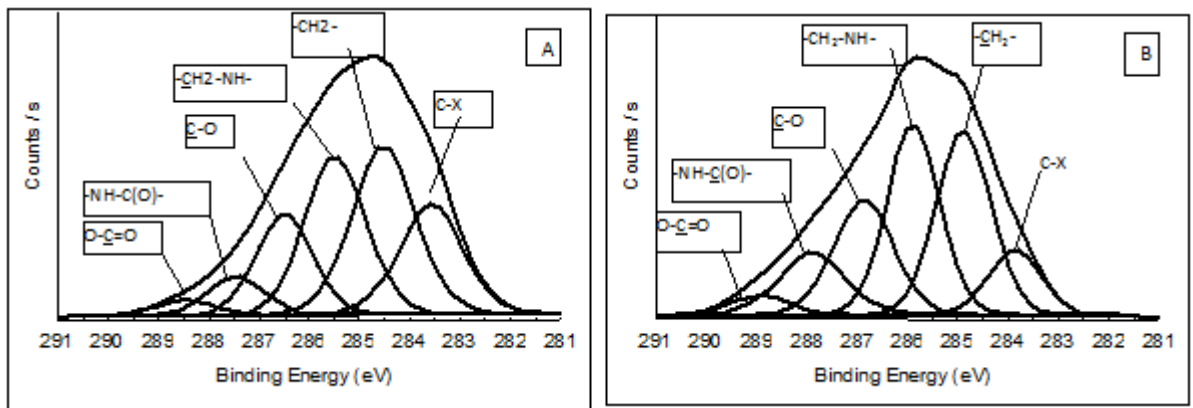


Figure 2: Expansion and deconvolution of C 1s peaks obtained from ESCA: (A) Ayous glue joint; (B) Frake glue joint

Deconvoluted high resolution spectrum of the C_{1s} peak of Ayous and Frake glue joint are characterized by six main peaks with five assigned peaks (Figure 2) : C-C or C-H at 284.5 eV, -CH₂-NH- at 285,8 eV, C-OH at 286.6 eV, -NH- C(O)- at 287,6 eV, O-C = O at 288,5 eV. The peak à 283,5/284 eV are shifted though low binding energies due to inhomogeneous charges and cannot being assigned with sure. O_{1s} is composed of three main peaks with two peaks assigned (Figure 3): O-H at 532.5 eV, H₂O /O-H at 533 eV and O-X non assigned. N_{1s}) shows three peaks (Figure 4): N-X at 398,5 non assigned, NH-C(O) at 399,5ev and NH-C(O) (hydrogen bonds) at 400,5eV.

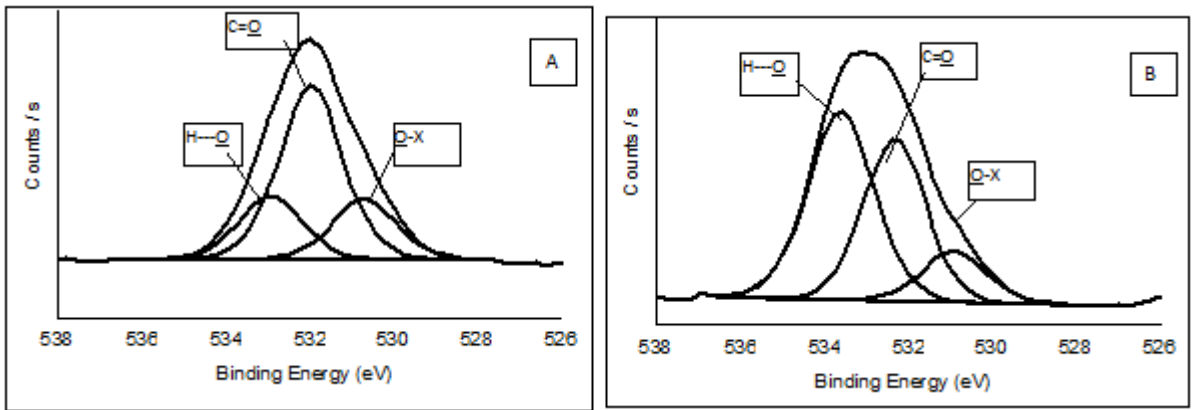


Figure 3: Expansion and deconvolution of O1s peaks obtained from ESCA: (A) Ayous glue joint; (B) Frake glue joint

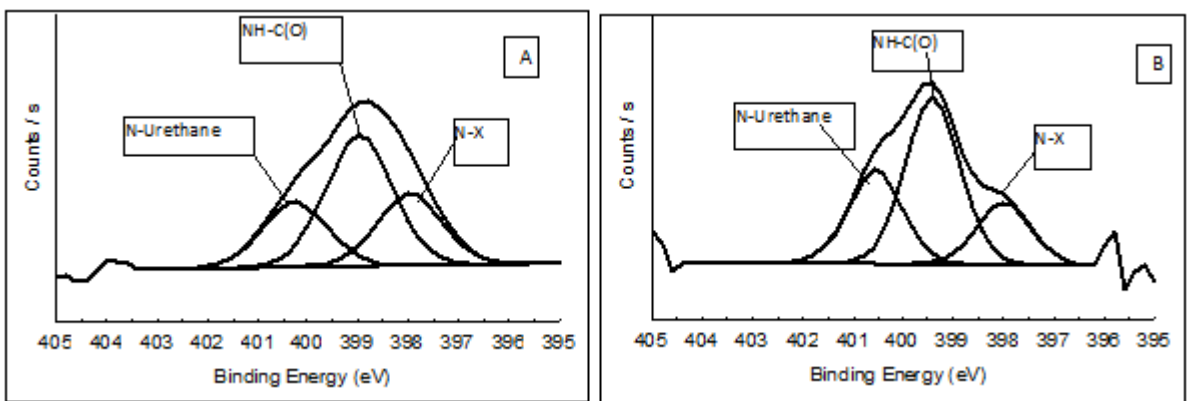


Figure 4: Expansion and deconvolution of N1s peaks obtained from ESCA: (A) Ayous glue joint; (B) Frake glue joint

Different proportions of carbon, oxygen and nitrogen chemical have been evaluated and reported in figure 5, 6 and 7.

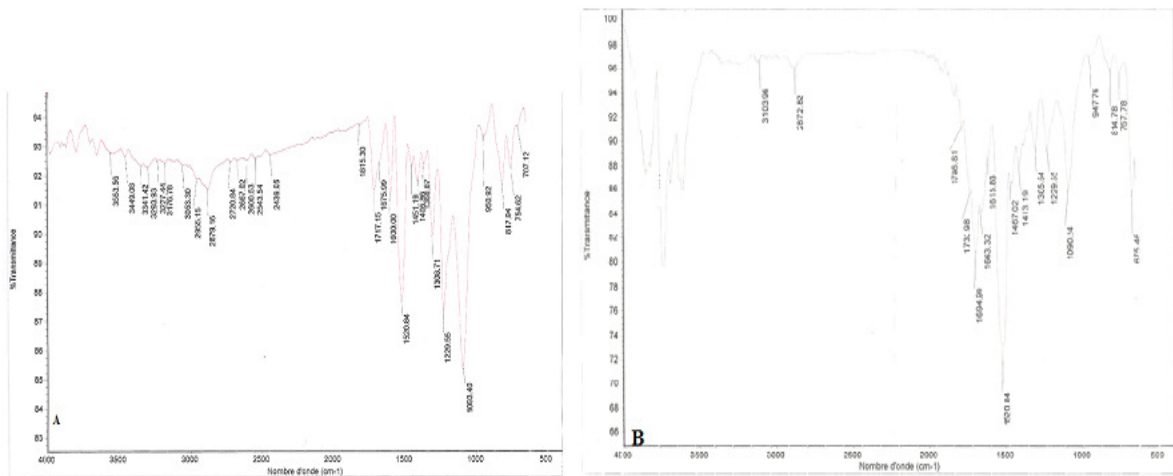


Figure 5: FTIR spectra of ayous joint (A) and fraké joint (B)

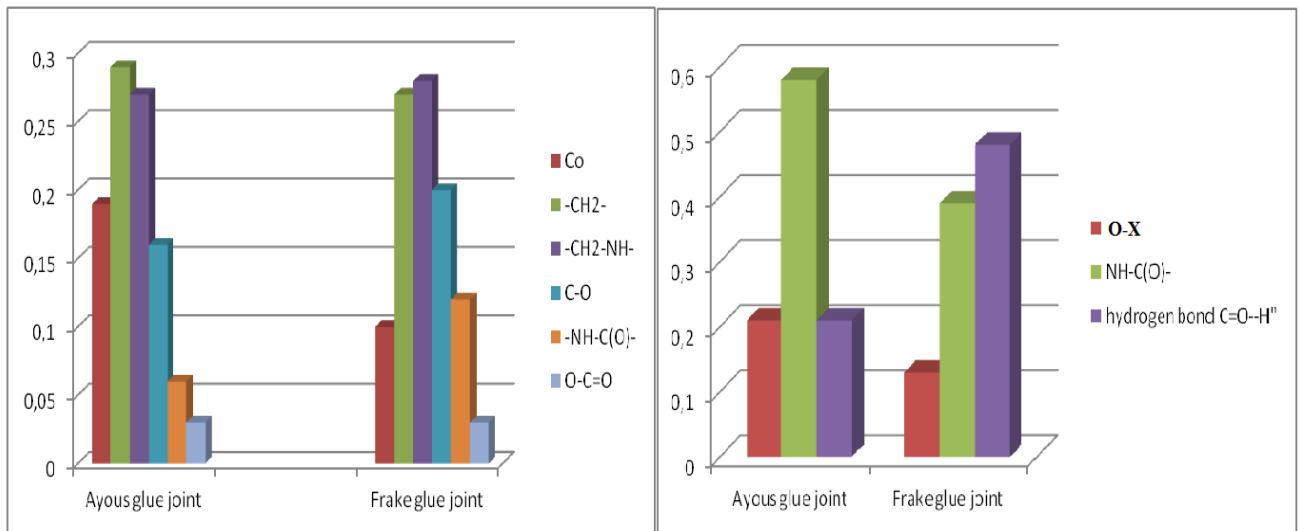


Figure 6: Proportion of different elements chemical states

(a): C1s ; (b): O1s; (c): N1s

Figure 6, 7 and 8 shown a high proportion of hydrogen bonds in wood glue joints. Hydrogen bonds have already determined the cristallinity of wood fibers. The established cristallinity denotes of high mecanical performance. And this could be explained by a high contribution of hydrogen bonds in gullam adhesion product

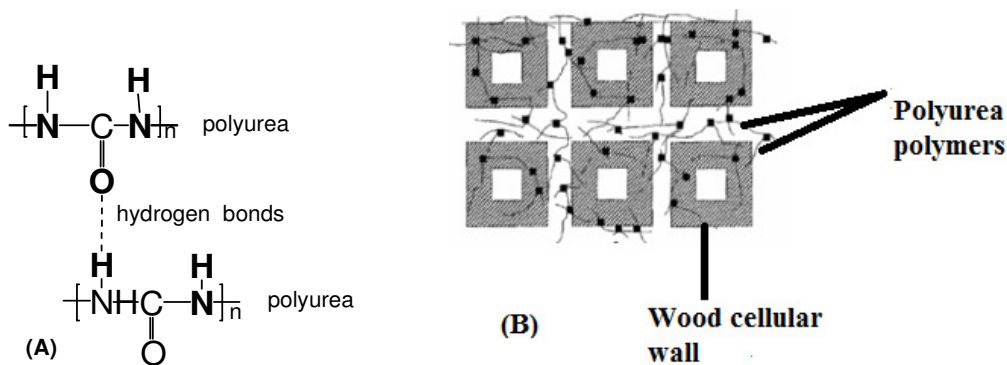


Figure 9: A: Hydrogen bonds between polyurea polymers; B: modèle of interdiffusion between fibers cellular wall and polyurea polymers adapted from Phanopoulos et al (2003)

Apart of hydrogen bonds the great quantity of $-CH_2-NH-$ functions observed particularly the $-NH-C(O)-NH-$ functions in glue joint (figure 9) denote of the formation of high quantity of polyurea polymers. Earlier work done by [8]Frazier and J.Ni (1998) have shown that the gluing of wood with isocyanates occurs through the reaction of isocyanates with water present in the wood, to give urea and biuret species that can form a cross-linked polymer matrix. This should certainly contribute to reinforced wood-adhesive adherence and adhesive cohesion. The trend of the results obtains are in accord with the model proposed by frazier and J.Ni for the curing of the isocyanate-wood glue bond which was one where pMDI penetrates deeply into wood cells and into the middle lamellae between the cells and reacts with the water present to form urea linkages (both linear and branched) and which form an entangled, interpenetrated network to give a strong, mechanically interlocked bond. C. [9]Buckley & al. (2002) shown that the more resistant glue joints (non hydrolysable) those obtained by interdiffusion and chemical reaction of glue (covalent bonds formation) towards wood cell wall.

-NH-C(O)-NH- and hydrogen bonds exhibited by XPS are confirmed by the ATR-IR analysis as shown in Figure 5. FTIR of ayous and frake glue joints are displayed in Figure 5. peaks of interest have been assigned according to amides with hydrogen bonds stretching band detected at 1694,99 and 1675,99 cm^{-1} [10], [11],[11] for ayous and frake glue joint respectively.

Conclusion

X-ray photoelectron spectroscopy also referred to as Electron spectroscopy for chemical analysis (ESCA) and Fourier transform infrared spectroscopy were used to investigate the interaction of both tropical woods with adhesive. Unlike theoretical expected urethane functional group, xps analysis indicated a high proportion of non hydrolyze urea group and hydrogen bonding of nitrogen. This foreseen that polyurea polymers and hydrogen bond are the most probable bond to strengthen the glue line.

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