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Review of Existing Methods for Evaluating Adhesive Bonds in Timber Products

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Abstract

Gluing is an integral part of the majority of production processes in the timber industry. The effectiveness of adhesive application, glue bond development and glue penetration into the wood structure is becoming more and more important as more structural glued timber products are used in construction and other applications. The continued increase in utilisation of mass timber products (MTPs) such as CLT, glulam and LVL in tall timber buildings requires an accurate and in-depth understanding of adhesive roles and their performance effectiveness during the life span of any of those products in relation to the type of loading applied, environmental effects (e.g. RH and temperature) and in-service condition of elements (e.g. exposure to major wet events and degradation from decay). This review aims to provide a comprehensive summary of existing imaging and other visualisation methods used to assess the glue line properties and examine the performance of glue lines in relation to factors such as species, product type and environmental conditions during manufacture and in-service life.

Keywords: glue line thickness, glue penetration, adhesion, timber species, image analysis, SEM, light microscopy, µCT, contrast agent, chemical imaging

1. Introduction to adhesive effectiveness

Gluing is an important and integral part of the majority of timber processing and new wood product designs and developments. Investigations into the effectiveness of the gluing process, formation of the glue line and structural strength of the product relies on an in-depth understanding of glue properties, the interaction between glue and timber species and type of product. This book chapter aims to review existing image analysis and other visualisation methods used to investigate the effectiveness of the gluing process and glue line formation in timber products at the cellular level.

Wood is a non-homogenous medium with a complex cellular structure and a range of chemical compositions that varies with type (hardwood vs. softwood), species, density and surface preparation. High extractives content, sensitivity of extractives to a high temperature environment and the surface characteristics of species affects the adhesion bond formation and strength. Adhesive bonding is currently considered to be mainly a combination of three mechanisms: mechanical interlocking (through wetting of the wood surface, glue penetration and

distribution and), physical attraction and covalent chemical bonding [1, 2]. **Figure 1** shows a diagram of glue bond development and importance of the mechanisms leading to development of a strong glue line and good glue penetration into wood cellular structure.

The natural resinous and oily extractive content of some timbers in combination with different drying methods, or added chemicals during processing could affect the adhesive performance due to possible changes in wood surface chemistry. Adhesion is a surface phenomenon and its penetration varies with different wood surfaces, extractive contents and spatial scales of surfaces. The actual measurement and assessment of mechanical interlocking of glue in the wood industry requires more investigation and is still developing [1]. The physical attraction between the wood and adhesive in many adhesive types is considered as the primary bonding process through developing van der Waals forces and hydrogen bonds [1]. The initial surface wetting and absorption into the wood structure are fundamentally important in development of a strong adhesive profile though physical attraction. The covalent bonding between wood fibre and adhesives occur through electron sharing however, in waterproof adhesives where there are sufficient intermolecular physical attractions this electron sharing system may not be necessary [1].

The development of adhesive bonds requires wetting of the surface, flow of the adhesive into the wood structure and penetration into lumens and cell walls of wood (for some adhesive types only). A good wetting process requires formation of a low contact angle with the surface. The flow however refers to spread of adhesive liquid over the wood surface and better coverage of the surface will lead to better bond development. Penetration is the movement of the adhesive into the depth of the wood structure [3]. The overall penetration of adhesive in wood follows Darcy's law where liquid volume flow (Q in $m^3.s^{-1}$) is defined as function of the specific permeability of wood (K in m^2) and area perpendicular to the liquid flow (A in m^2), length in flow direction (L in m), dynamic viscosity of the adhesive liquid (η in Pa. s) and pressure gradient (ΔP in Pa) [4].

$$Q = K \times \frac{A}{L} \times \frac{1}{\eta} \times \Delta P \tag{1}$$

Permeability plays an important role in glue bond development and low permeability of wood types (such as Douglas-fir heartwood) can cause very low penetration of resin into radial and tangential surfaces while high permeability of the wood surface can lead to bond line starvation [5, 6].

Adhesive penetration into wood can be categorised into: gross penetration and cell wall penetration [6] (see **Figure 2**). In gross penetration the adhesive flows into the wood porous structure filling the lumens, this type of penetration occurs in most of the resin types at low viscosity. The cell wall penetration which is the flow of the adhesive into the cell walls of woody structure is only achieved with resins with small molecular weight (MW) components. A previous study identified that a MW of 3000
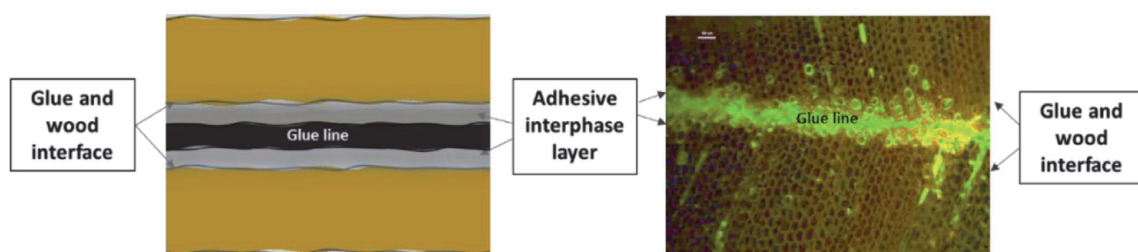

Figure 1.
Adhesive bonding mechanism including the glue and interaction between glue and timber.
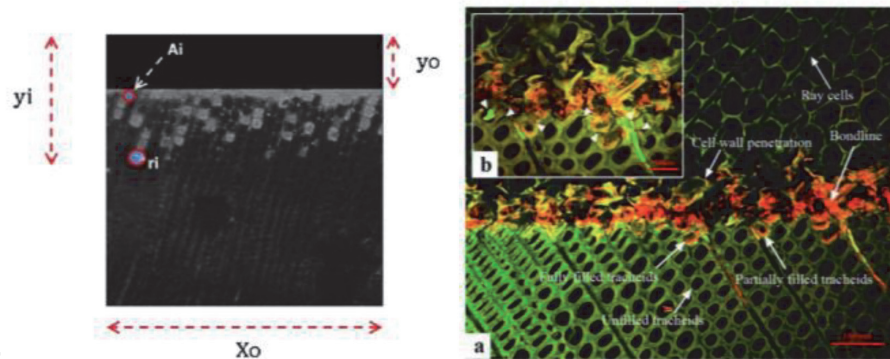
Figure 2.
(Left) Imaging of effective penetration (EP) and maximum penetration (MP) of gross penetration of into structure [8] and (right) penetration of urea formaldehyde (UF) into wood cell walls shown in green colour [9].

for polyethylene glycol (PEG) is a critical factor at room temperature to achieve cell wall penetration in Sitka spruce. Higher temperatures could cause larger MW due to the changes in cell wall polymer and intermolecular volumes [6, 7]. Both effective and maximum penetrations are critical in achieving a good adhesive bonding [4, 6].

Effective penetration (EP) is defined as the total area of adhesive in the interphase region of the glue line divided by the width of glue line. Maximum penetration (MP) is measured as the average value of five distance measurements taken of the five most remote adhesive objects observed in the image field [8].

$$EP = \frac{\sum_i^n A_i}{X_0} \ and \ MP = \frac{\sum_i^5 (y_i + r_i - y_0)}{5} \tag{2}$$

As shown in **Figure 2(a)**, A_i, X_0, y_i, r_i and y_0 are the area of adhesive studied in μm^2, width of image or maximum rectangle determining the area in μm, centroid of adhesive object i, mean radius of object i and reference y coordinate in μm [8].

Resin viscosity, molecular weight (MW), molecular weight distribution, resin solid content and surface tension are properties of an adhesive that affect the bond development and adhesive penetration [5, 6]. Literature suggests the lower MW leads to deeper penetration of adhesive into wood structure in comparison with higher MW resins [10]. Operational factors such as open processing time, pressing time, temperature and consolidation pressure also affect the glue bond development and penetration [4–6]. Adhesive interaction with the wood surface is a function of length scale and defined as interlocking/entanglement and charge. The interlocking or entanglement interactions occur over a longer length scale in comparison with charge interaction which happens in the molecular or nano-length scale [4, 11].

Adhesive penetration is defined by the maximum depth of adhesive in wood, the thickness of the glue bond and the surface condition that affects the penetration measurement and direction. In softwood, tracheids filled with glue are visible in an interconnected zone while the process in hardwoods is more complex. In hardwood, the vessels are filled with glue but they are usually scattered and not close to the actual glue line. In softwood samples the maximum glue penetration is usually measured by trigonometric relations between the uni-directional tracheids near or around the borderline (see **Figure 3**). Hass et al. [12] applied a new approach in defining adhesive penetration in hardwood samples (beech) by considering pore space, ray distribution, viscosity of adhesive and bond line morphology. The study showed that the position of measurement has direct and significant effects on bond line characteristics [12]. The anatomical structure of samples, its wood type, adhesive type and their properties (viscosity for example) could all affect the penetration measurement and its replicability for various studies and products.
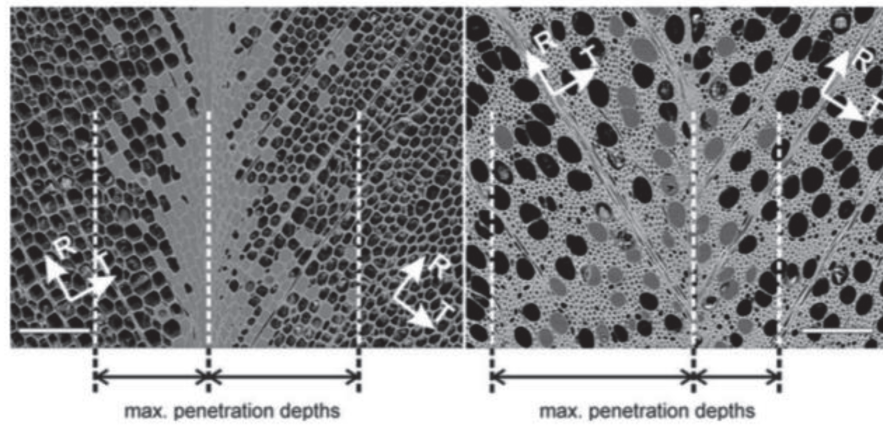
Figure 3.
SEM imaging of maximum adhesive penetration in (left) softwood (spruce) and (right) hardwood (beech). The length of the scale bar in images is 200 um [12].

2. Adhesive systems and types

The adhesive type used in each application need to be suitable for the end use of the product as well as being compatible with the wood type and characteristics and bonding conditions [3]. Wood adhesives can be categorised by their structural, semi-structural and non-structural applications as well as the strength and durability of the bond they develop.

Polymer formation can be used to categorise adhesive types. Linear polymers develop links that are like strings of beads; polyethylene and polypropylene are two linear polymers. The other type of polymer develop branches of linear chains and the properties of formed polymers changes intensely as the branches change. As the density and length of polymers developed changes the melting point, flexibility and strength of adhesive bond changes as well [3, 13].

2.1 Self-adhesion

Self-adhesion in wood products can be achieved in certain conditions however for better strength usually adhesives are needed. Hydrogen bonding, auto-cross-linking, wood welding and rotational welding are two of the self-adhesion types. Hydrogen bonding and auto-cross-linking of the lignin and hemicellulose components in high-density fibreboard requires little to no adhesive. In high moisture and temperature conditions, softening of lignin and the hemicellulose content of wood leads to hydrogen bonding of hemicellulose joining fibres and lignin to develop chemical bonds. Through vibrational welding at high temperature and cellular distortion, bonds are developed. The bond developed has shown good strength properties in dry environments, but the strength is very low in wet conditions. Rotational welding happens when a wood dowel is driven into another piece of wood. Self-adhesion can be improved by modifying the environment (moisture, heat etc.) as well as the addition of chemicals for better bond development [14–18].

2.2 Formaldehyde adhesives

Formaldehyde adhesives (resorcinol formaldehyde (RF), phenol resorcinol formaldehyde (PRF, urea formaldehyde (UF) and Mixed Urea Formaldehyde (MF-including melamine urea formaldehyde MUF) are usually water borne resins and the curing procedure involves polymerisation and loss of water. The loss of water in

the bond line delays the reaction of adhesives with wood due to the reduction in the wettability and movement of resin. This will limit the collision required for polymerisation process and heat transfer.

Thermosetting phenol-formaldehyde (PF) or UF are the polymers used more commonly in structural veneer-based wood product applications. For exterior veneer based wood product applications usually PFs are used and UFs are mainly used for interior applications [1]. Formaldehyde adhesives develop a rigid bond and do not creep due to combined development of polymeric chains and cross-linking groups [3, 13].

2.3 Isocyanates in wood

Isocyanates in wood (Polymeric Diphenylmethane Diisocyanate, Emulsion Polymer Isocyanates, Polyurethane Adhesives).

Isocyanates are used in wood adhesion due to their reactive characteristics to compounds with reactive hydrogen. These adhesives however can react very fast with wood moisture which will compete against the required reaction with the hydroxyl group in wood's cellulose and hemicellulose and phenol and hydroxyl groups in lignin sections. The other drawback of these adhesive is their high reactivity level with the human body that could cause safety concerns during the gluing process. The most used type of isocyanates is polymeric diphenylmethane diisocynate (pMDI) in manufacturing oriented strand board (OSB) [3, 19]. PUR adhesives are also now widely in use for a wide range of application in timber products including glulam and cross laminated timber (CLT).

2.4 Epoxy resins

Epoxy resins are compounded with ketimines that assists with releasing the curing agent when the adhesive is exposed to moisture. Similar technology is already used in coating products [1]. Epoxies are produced with a range of curing times which can influence the degree of cure and mechanical strength of adhesive layer [3].

2.5 Polyvinyl and ethylene acetate (PVA) and dispersion adhesives

These are waterborne adhesives that are cost effective and do not require a heat curing operation and they are mainly used in furniture construction. These adhesives commonly exhibit good flow into the cell lumens that are exposed to glue however due to the high molecular weight they do not usually penetrate into wood cell walls [3]. Polyvinyl acetate (PVA) is commonly used for wood gluing in non-structural and furniture making however it lacks water resistance and has low load bearing properties.

2.6 Bio-based adhesives (protein Glues, tannin adhesives, lignin adhesives)

The protein driven from wheat grain (gluten) can react with aldehydes in a similar way to urea. Gluten has a high level of amine groups (lysine and arginine) which react similarly to the ones in melamine and phenols [20]. The availability of gluten from grain is an advantage for its application in wood adhesion. However, the powder form of the gluten limits its applicability to be used in current industrial manufacturing operations.

Lignin has a phenolic structure which makes it a potential replacement for phenol in phenolic resins used for wood adhesion [21]. Lignin based adhesives can be considered in two major categories including phenol formaldehyde and

formaldehyde free adhesives. Initial investigations into using unmodified lignin in phenolic adhesives showed a reduction in glue strength and an increase in press time, so chemical modification of lignin has been suggested as a solution [22]. The use of Kraft lignin and polyethylenimine (PEI) for development of a formaldehyde free adhesive showed a very high shear strength and water resistance in the glue developed [23].

2.7 Miscellaneous composite adhesives

In this group of adhesives depending on the role of timber in the composite there are three different product types known including: wood-fibre cement boards, wood-plastic composites and wood filler for plastics [3]. Wood-fibre cement products use the plant fibre to reinforce the panels and reduce the possibility of fracture development, this field is still under further studies. Wood-plastic combination is used to reduce the product weight for industries such as automotive industry. These products require good polymer- fibre interaction otherwise exposure of fibre to moisture and under stress the interface can fail.

2.8 Construction adhesives

These adhesives are used in construction for attaching floors and wall coverings which provide better rigidity in comparison with using nails or screws only. These adhesives do not require fast curing as the nails or screws hold the connection together while curing is completed. These adhesives have high molecular weight with minimum amount of solvent and are applied at room temperature. They are used to cover the gaps in the connected joints. The adhesives used in construction are usually elastomers to provide some deformability for small range displacement so the bond lines do not fail/crack as timber expands or shrinks. These adhesives are not designed for larger scale movements due to their high molecular weight and require checks and maintenance of the joint over time [3].

2.9 Hot melts

These adhesives develop bonds quickly and are used for furniture, cabinet making, windows and edge banding of laminates. The high viscosity of these adhesive limits the wetting properties of them [3].

2.10 Pressure sensitive adhesives (PSAs)

These adhesives are high molecular weight polymers that have application in decorative laminates, tapes and labels. They are usually used in gluing plastics to wood and have different ranges for products used in indoor and outdoor conditions. Their low flow characteristics makes the pressure application important so that the force applied creates required deformation in the elastomeric adhesive [3]. In these adhesives debonding or failure of the bond developed can happen under large dimensional changes and is dependent on the stress–strain properties of the adhesive.

2.11 Contact adhesives

These adhesives are polymers in solvent and are applied on gluing surfaces and left for the solvent to evaporate before the two surfaces are brought together and pressed. These adhesives are used in bonding wood to plastic laminates.

2.12 Polymerizable acrylic

These adhesives are expensive and are not commonly used in bonding wood. Structural acrylic and cyanoacrylate instant adhesives are two types of these adhesives that can be used for gluing wood. They require a smooth surface and are used in products that require rapid curing and high strength bonds. They are used in products such as electronics assembly or decorative layers in panel manufacturing [3].

2.13 Film adhesives

These adhesives are used in applications where the use of liquid glue is difficult or limiting for the type of product. They are applied using an applicator such as fibreglass mat or tissue paper in applications such as gluing very thin layer wood veneers [3].

2.14 Formulation of adhesives

Adhesives are developed for specific applications where altering the properties of the adhesive can be required. Parameters such as surface roughness, moisture level in wood structure, cost, time between application and curing, type of production process and conditional changes (season) can influence the customisation required for formulation of adhesives [3].

3. Techniques used in imaging wood products

The conventional microscopic imaging has been the most common and primary tool for capturing wood structure using optical waves and high magnifications to clearly capture the structure of the sample. Modern optical microscopes can generate images with 1500 times magnification with a 0.2 um limit in spatial resolution. The light transmitting through transparent sections and reflecting on the other parts of object helps in differentiating the structural components and developing clear images of the surface. Use of bright, dark, polarised, phase difference and fluorescence enhances the imageability of objects depending on the differences between the structural components [24]. It is not easy to create perfect images of a natural object using optical wavelengths due to variations in optical waves passing through the lens of the microscopes [25]. However, use of digital multimedia and digital processing technology to enhance the light microscopic system allows the collection of imaging outputs, improves the image quality and provides more image post-processing options.

3.1 Microscopy (visible light, UV, IR)

Using light microscopy, the position and thickness of adhesive in lumens and other parts of the wood structure, can be captured and measured depending on wood species and adhesive types used [26]. Light microscopy can be used in generating 2 and 3D images of structures. 3D imaging of a sample's volume can be achieved by further processing of either microtome sectioning and stacking of images using visualisation software or using optical techniques (such as light-sheet microscopy, confocal laser scanning microscopy or optical projection tomography) to mount the images taken into volumetric models [27–29]. Florescence microscopy is used to separate wood cells from the glue line [30] using short wavelength light to brighten sections of the sample [24]. Fluorescence microscopy uses a high voltage

mercury lamp [24] that can improve the image quality by permitting the light excitation to irradiate the specimen and separate the weaker re-radiating fluorescent light from the brighter excitation light (**Figure 4**) [31].

The fluorescence presence in the adhesive needs to be sufficient to be able to capture a clear image of the bond and glue line (see **Figure 1** for more information). In order to enhance the image quality fluorescence staining can be used however sufficient power is required to produce fluorescent light [24]. Specific optical filters (each has a certain wavelength) are needed to generate images with suitable wavelength of light emitted resulting in maximum fluorescent light outputs. An aqueous mix of 0.2% of acridine yellow which is absorbed by wood but not the PF glue is used to enhance the fluorescence presence in the imaging process [31]. However the staining methods used in the literature are limited and require extra experimental steps for sample preparation, and majority of reported staining trials applied stain to the resin prior to gluing. The application of stain in industrial scale production line can be costly and cause undesirable colour changes in the product [32]. Combining images taken in visible and florescence modes (FM) can enhance the visibility of the glue line. Mahrdt et al. [32] used a merging method to combine images taken in visible and fluorescence modes using gentian violet and brilliant sulphaflavine dyes respectively (**Figure 5**). The merging technique improved the post-processing required for quantifying glue line parameters by allowing
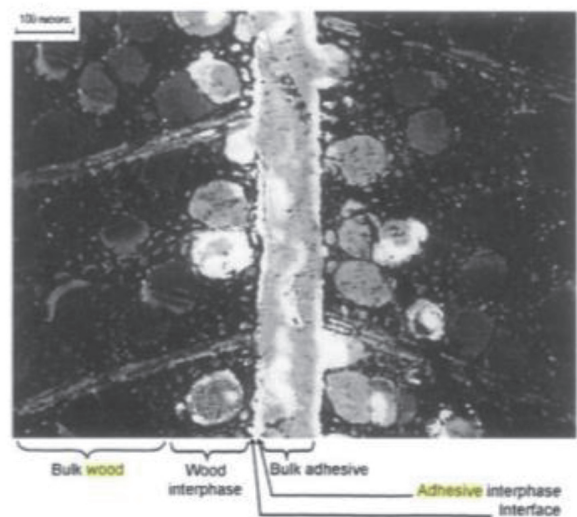

Figure 4.
Adhesive bond line fluorescence microscopy of an epoxy glue showing regions of glue penetration [30].
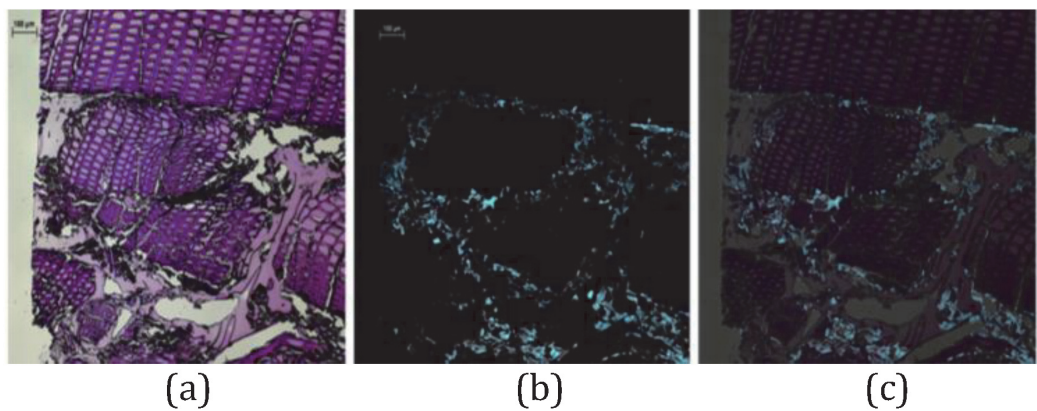

(a) (b) (c)

Figure 5.
Image of cross section and glueline using (a) visible, (b) fluorescence modes and (c) merged image [32].

semi-automatic analysis of images. The most common disadvantage associated with using FM is the limitation in capturing depth which is an important parameter in glue line investigation [33].

Based on the optical path used the fluorescent microscopy can be a transmission fluorescence microscopy (TFM) or an epifluorescence microscopy (EPI) [24]. TFM usually uses a dark field passing light through a condenser to excite the sample to emit fluorescent light in different directions. TFM provides generation of strong fluorescent light in low magnifications however once the magnification increases the fluorescent light reduces making the method more applicable for larger specimens [24]. EPI uses the objective lens as a well corrected condenser first then as a light gatherer to form an image (see optical path illustration in **Figure 6**).

The images produced by TFM are usually darker in comparison with images taken by EPI. Some of the other advantages of EPI systems are increased fluorescent light intensity, reduction in light loss, no loss of fluorescence intensity [24].

Imaging of adhesive penetration into wood based composites using EPI is shown in **Figure 7** [34]. The maximum penetration depth in fibres showed a rising trend as the moisture content increased. A study on the effects of moisture curing of polyurethane on the segment content (hard and soft content) of adhesive bonds showed that increasing hard phase content increased the intermolecular interactions, liquid viscosity of adhesive,and the soft phase glass transition temperature [35]. This study showed that glue bond line thickness increased as hard phase percentage increased while the hard mass percentage had a negative relation with the effective glue penetration (**Figure 8**).

Penetration of PF resin into poplar was quantified using EPI imaging at 2, 6 and 10% moisture content [34]. For imaging, a 50% phenol formaldehyde (PF) resin solution in water was sprayed onto the uniform oriented strand board. The mixture made of poplar and 2% potassium carbonate (K_2CO_3) as the catalyst was added to the strand boards [34, 36].

Using ultraviolet (UV) microscopy can generate better resolutions and higher magnification for images of glue lines. UV incident light microscopy imaging (UVLMI) was used to study the glue line in LVL samples of beech shown in **Figure 9** after cyclic temperature and RH conditioning [37]. The images taken showed a clear illustration of the modified melamine–formaldehyde resin (MUF) and PUR penetration in the LVL structure. High-resolution episcopic microscopy
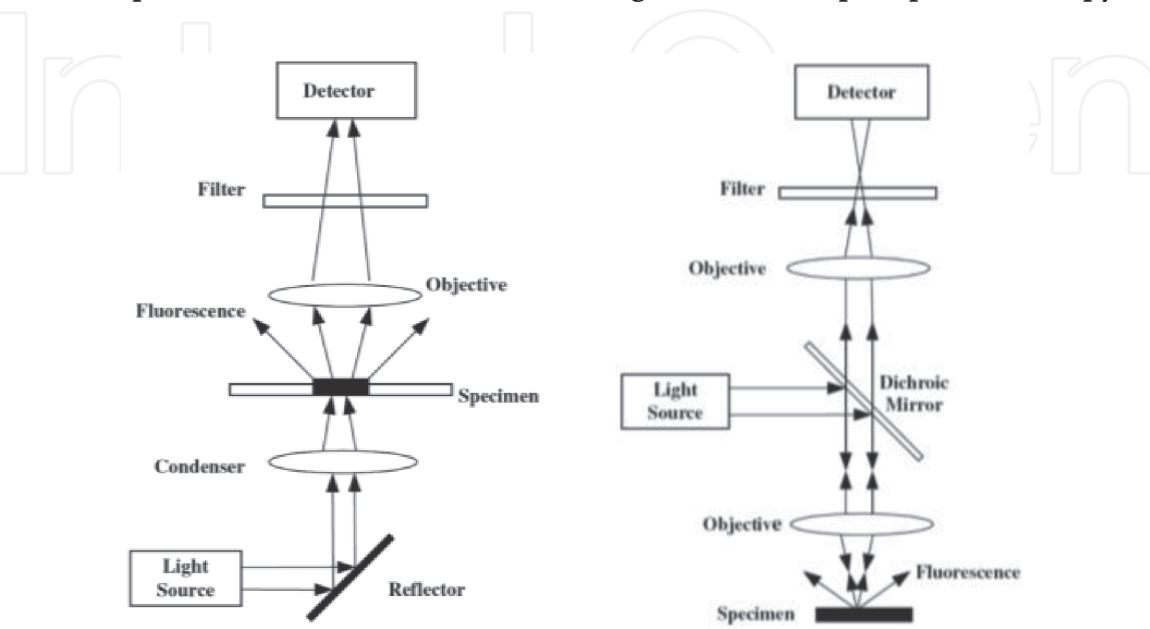

Figure 6.
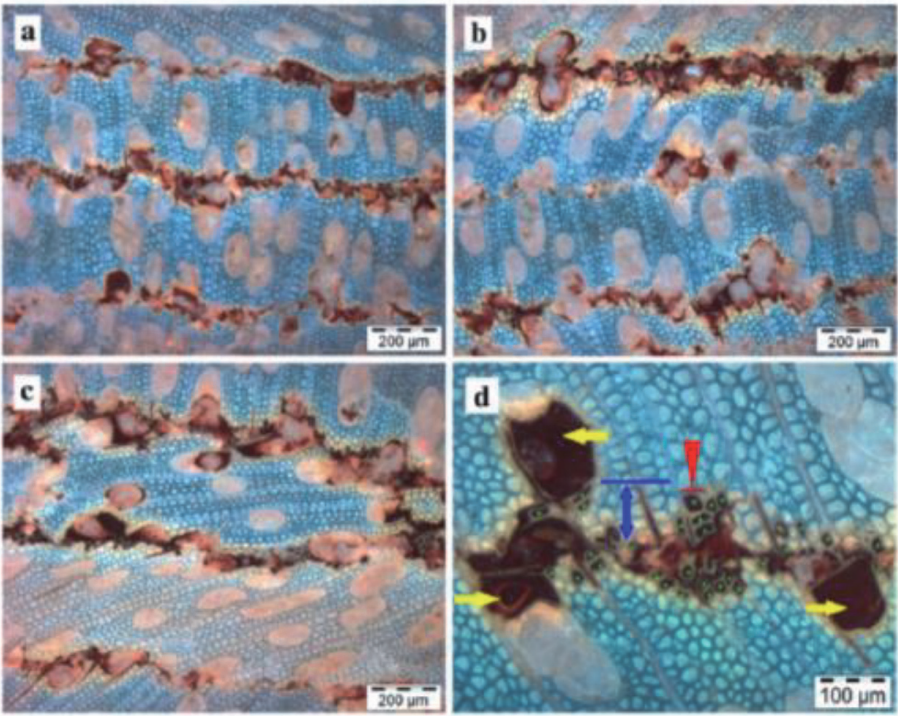*Optical path differences between (left) TFM and (right) EPI [24].*

Figure 7.
Penetration of PF resin into poplar strands at different moisture contents of (a) 2%, (b) 6%, (c) 10% and (d) the discontinuity of glue in 6% moisture content samples [34].
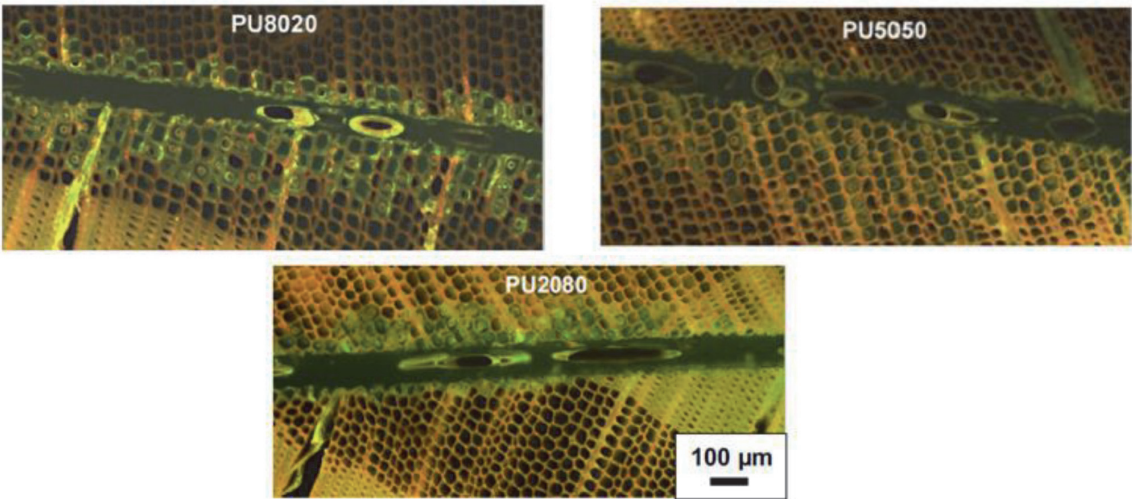

Figure 8.
Florescence microscopy imaging of PUR bond line and penetration development for top: PU8020 with 53.5%, middle: PU5050 with 65.4% and bottom: PU2080 with 72.5% hard phase mass [35].

((HREM) uses microtome sections taking images of thin layers of the sample block (1–5 μm thick). The images are then stacked using visualisation software to develop a 3D model of the sample block. A florescence stereomicroscope and a digital camera are used to capture the dye mixture embedded in the sample block in visualising the structural components [29]. The HREM method has been successfully used in various medical applications providing better resolution imaging in comparison with X-ray computed tomography, MRI, and optical projection tomography (OPT) [28, 38]. Samples require detailed processing of dehydrated tissues prior to HREM imaging using 4% paraformaldehyde (w/v) for 1–2 hours under vacuum and then incubated at 4°C overnight before being washed by ethanol and embedded in embedding solution (**Figure 10**) [29].
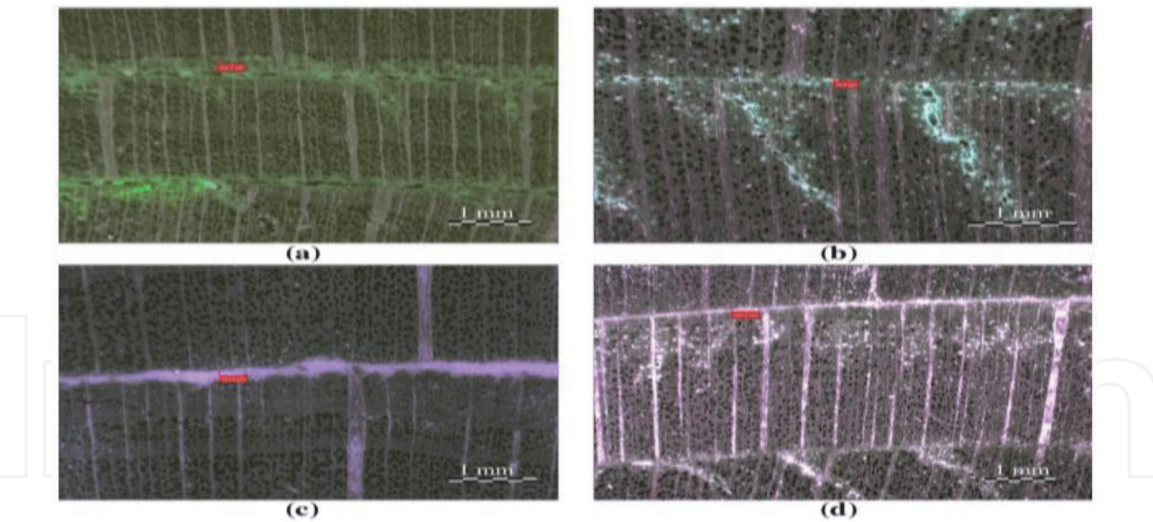
Figure 9.
Images of layered structure of LVL specimens conditioned in different temperature and relative humidity [37].
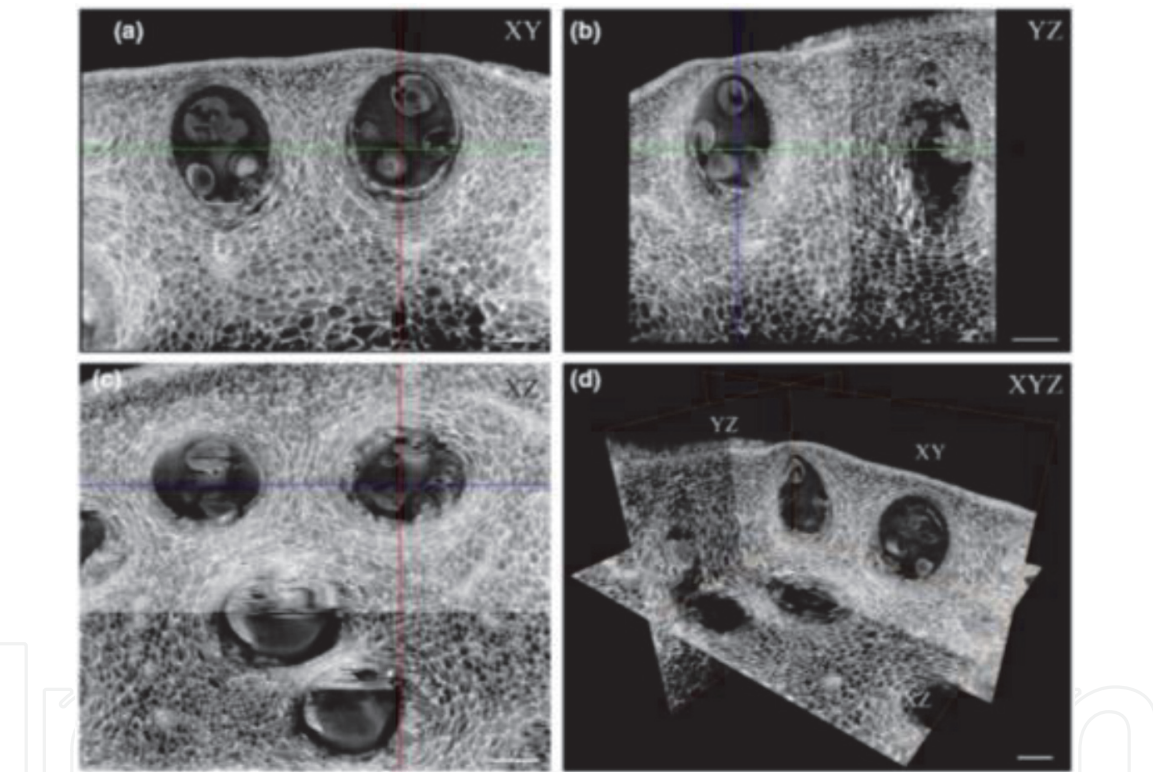

Figure 10.
Section images of mature Citrus limon *fruit using HREM [29].*

3.2 Electron microscopy (SEM, ESEM, transmission electron microscopy (TEM))

To enhance the spatial resolution of microscopes, the use of electron beams is becoming a more preferred technique replacing the visible light source. Electron microscopy can reach a maximum of more than 300 million times magnification. Moving electrons can act similar to optical wavelength variation. The concept of detecting the energy release and wavelength as electrons move are the foundation for development of transmission electron microscopy (TEM) in 1938 and scanning electron microscopy (SEM) in 1952 [24]. Imaging with a higher level of magnification and in greater depth is possible using electron microscopy. However the grey

colour of the images produced limits the imaging of different glue lines and sample preparation and imaging in a vacuum environment are some of the limitations for electron microscopy [24, 33].

3.3 X-ray computed tomography (XCT)

Computed tomography (CT) was initially developed in 1970s for medical imaging, the CT scanner provides a range of image sizes including: large up to 250–500 µm or smaller µ-CT (min spatial resolution of 50 to 325 nm) and n-CT (min spatial resolution of 200 nm). For smaller size samples, reduction of the X-ray flux is required which would increase the required scanning time [27]. X-ray computed tomography (XCT) is used in microscale to image the cellular structure of samples in 3D mode providing spatial details of the complex xylem network [39]. XCT uses radiation that penetrates into the sample and then detects photons after being traversed through the specimen. XCT uses two different mechanisms to develop images of the structure: absorption contrast and phase-contrast tomography. In absorption contrast methods, the differences in material linear attenuation coefficients, µ, affects the absorption contrast resulting in sufficient contrast to map out the edges of cells in the sample structure. The linear attenuation coefficients are dependent on material density and atomic number (Z). Phase-contrast tomography uses photons incident beam phase shift at an interface between two different materials. Phase contrast tomography is suitable for materials with low absorption contrast such as soft material with a lower atomic number [40]. In wood adhesive penetration detection, the similarities between the attenuation PF and cellobiose (chosen polymer in this study to represent cell walls in wood structure) showed that XCT is not a suitable detecting method if the PF glue was tested without any contrast agent added [41]. However, this may not apply to all glue types and timber products depending on wood species, their characterises and densities (**Figure 11**).

The lack of x-ray absorption contrast by the adhesive and the wood cells makes the use of micro CT (µCT) technology in bond line imaging and adhesive penetration challenging. The insufficient differences between the natural density of wood and the glue types used require addition of an enhancing agent to the glue for the purpose of imaging. Heavy metals have been used as contrast agent for imaging of glue line in wood products however their limited tag mobility and phase separation of those components made use of them less effective in studying the glue line properties [42]. The scanning is dependent on the absorbed contrast based on a materials linear attenuation coefficient (µ) differences. The linear attenuation coefficient is a function of material density and elemental composition. Based on Beer–Lambert's law, µ is calculated using the transmitted (I) and initial (I_0) radiation intensity and the material thickness (d) [43]:

$$I/I_0 = exp\,^{(-\mu d)} \qquad (3)$$

Considering the differences between adhesives used in the timber industry and the density of commercial timber species, achieving the required contrast between the two materials will require addition of contrast agents to the wood or adhesive used. The use of contrast agent additives has been studied for wood adhesive penetration studies before [33, 40, 41, 44, 45] . From a range of tested contrast agents used with PF (see **Figure 12**) iodine was selected as the preferred agent, improving the clarity of differences between adhesive and cell walls. Rubidium was also tested as a contrast agent in combination with PF. RbPF (added in form of RbOH as a contrast agent to PF with molar ratio of 14.3) showed significantly clearer differences between the glue and cell walls. Use of Rb as contrast agent produced clear images and
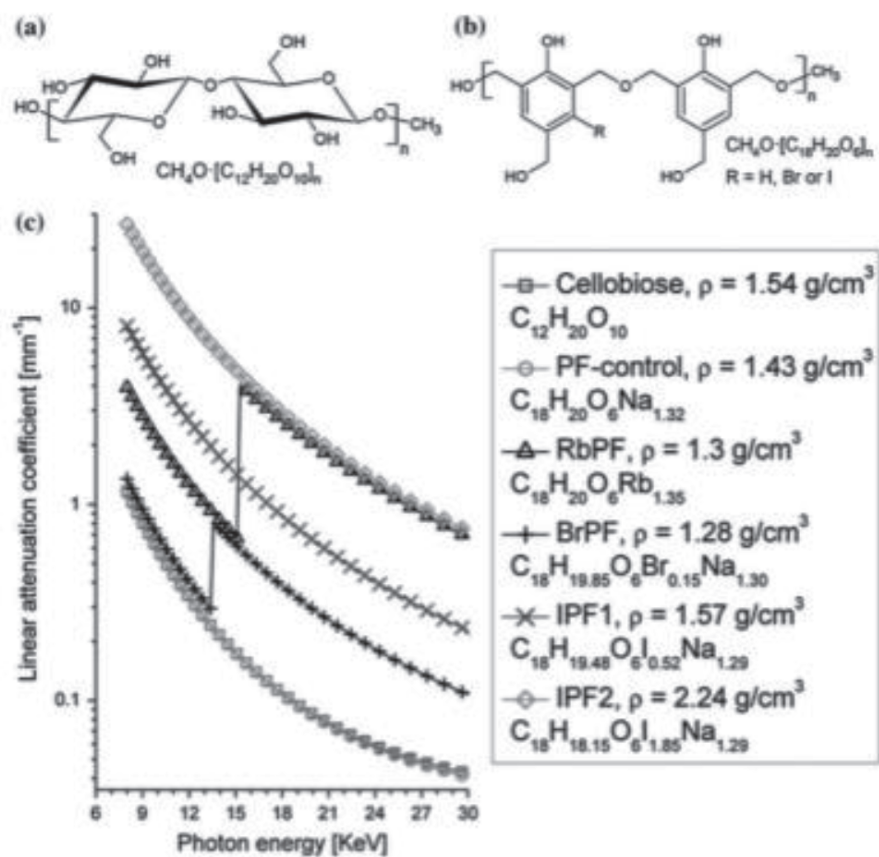
Figure 11.
Linear attenuation recorded for PF- control, brominated (BRPF), iodinated (IPF1 and 2) and rubidium (RBPF) adhesives and cellobiose as representor of wood cell walls polymer [41].
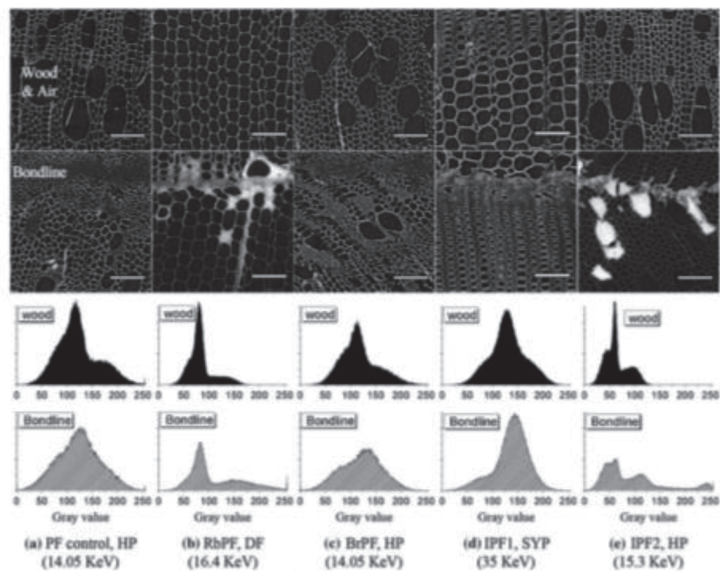

Figure 12.
Results of imaging adhesives with different contrast agents added [41].

adhesive with Rb travelled easier in species with higher permeability such as oak and poplar while Douglas-fir showed lower movement of Rb in sample structure. The images taken from samples also showed that Rb travelled into the wood structure independently of the adhesive and penetrated further than the adhesive which indicated the complexity of using Rb as a contrast agent [33].

Use of x-ray micro tomography (XMT) in capturing the PF adhesive penetration in Douglas-fir samples improved the clarity of glue penetration in the sample

structure however, the grey-scale results did not illustrate clearly the interface between cell walls and resin filled lumens (**Figure 13**).

For clarity and image quality using XMT, samples need to be dried. The excess moisture - at saturation point can reduce the cell wall contrast and image quality. However, the oven drying of wood samples below FSP could increase the risk of cell wall collapse. Combining electron energy loss spectroscopy (EELS) or energy dispersive X-ray analysis (EDXA) with electron microscopy has shown advantages in quantifying the glue line parameters and depth. EELS uses an electron probe targeted with high capabilities in imaging dark modes (these couple weakly with the optical excitation making imaging more difficult in comparison with light mode) to map different locations of the sample [46]. The SEM-EDXA is suitable in investigating if the adhesive penetrated into the cell structure (**Figure 14**) [47].

Iodinated (IPF) and brominated (BrPF) PF resins were used in µCT imaging of the glue lines in wood composite panels. The study of glue viscosity and MW (after curing) showed no changes in BrPF. However, the MW of glue before curing increased as percentage of added contrast agent increased. The IPF had slightly lower viscosity compared to control PF samples. The powder density determined for BrPF was lower than controlled PF samples. Further testing of tagged adhesive using fluorescent micrographs and energy-dispersive spectroscopy (EDS) elemental
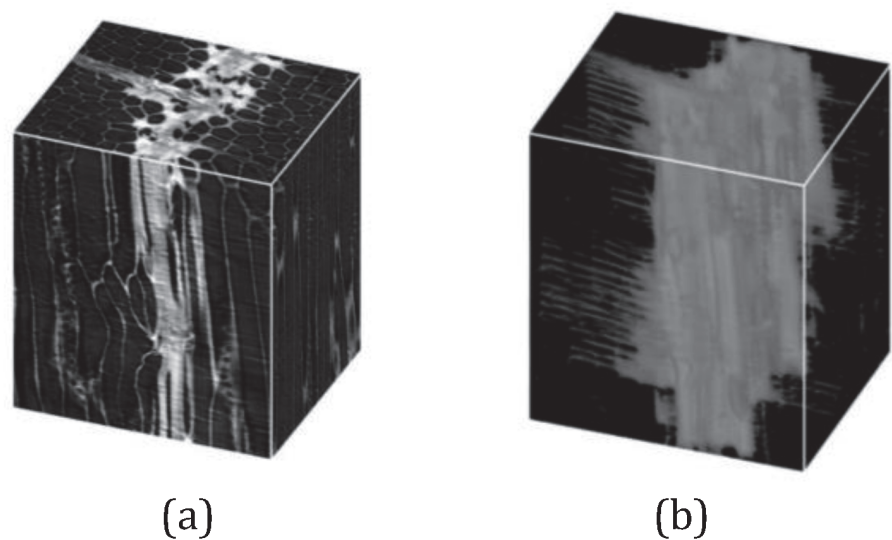

(a)   (b)

Figure 13.
3D images of Douglas-fir with phenol–formaldehyde adhesive in transverse surface on top (a) with the cell wall details and (b) removed cell wall details [33].
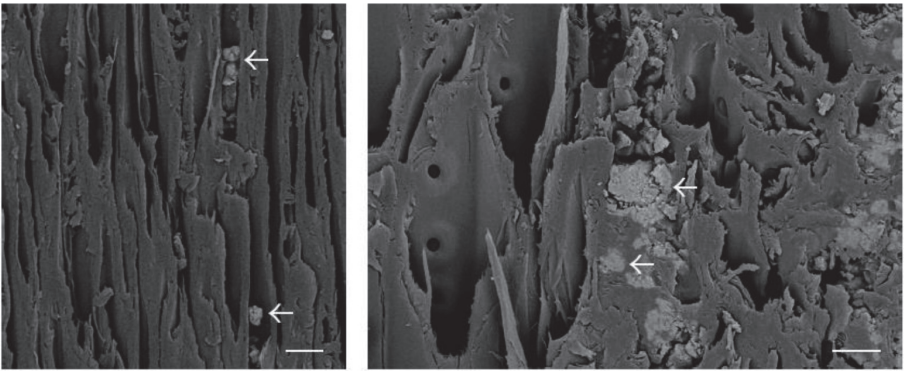

Figure 14.
EDX spectrum and backscattered SEM imaging of OSB using zinc borate as a contrast agent, arrows show the zinc borate particles in lumens of wood cell [47].

maps by Modzel [48] showed the presence of Rb in cell-walls in sections with no RbPF glue presence (**Figure 15**) [43].

In a study by Kamke et al. Iodinated PF (IPF) was made by adding 10% 3-iodophenol to plywood adhesives following commercial adhesive synthesis manufacturing process. The final adhesive had 39.5% weight iodine as the contrast agent to enhance the x-ray imaging results. Even though the viscosity of adhesive at 25C was 930 cP which could have changed the curing properties of the glue it did not compromise the behaviour of the bond. Images from this study are shown in [41]. The images generated with ionising radiation (called tomograms or slices) are obtained from the translation and rotation of the source and detectors. In this imaging technique, the attenuation coefficient of x-ray or gamma rays are recorded. In a non-homogenous medium such as wood structure it detects the attenuation coefficient depending on the quantum energy of the ionising radiation and the chemical composition of the sample [49]. The computer tomography (CT) method provides the option of imaging the full 3D structure of the sample including bark, knots, heart and sapwood borders etc. [44]. The scanning parameters measured by ionising radiation vary with wood species, size of the specimen, level of contrast in density for different defects, end use of scan information, speed of scanning required for imaging [49].

3.4 Magnetic resonance imaging (MRI) or nuclear magnetic resonance (NMR)

MRI or NMR was developed in 1970s for medical imaging and it was used later in the 1980s in imaging plant material [27, 50]. MRI is a non-destructive method to develop images of plant structure and has been used as an in vivo method to determine the water content and moisture movement in plant structure [51]. Images of a *Quercus serrata* branch were taken using MRI at different signal intensity specification to detect different components (shown in **Figure 16**) including water, annual ring, inner rings [52]. Although no studies have been reported in the literature on the use of MRI or NMR for studying adhesive bonds, both these technologies have considerable potential for this purpose.

Thermal technique uses the temperature to map out the surface of the object applying an active or passive heating procedure. The cyclic loading in structural timber for example can be used as an active source of heating to detect the effects of defects in structure on the mechanical strength and performance of products [49].

The passive heating process usually applies for detecting knots direction, slope of grain, moisture distribution and rupturing in wood structure. The passive heating process does not create the destructive effects on the samples due to low thermal stress however it requires fast recording methods to generate accurate images.
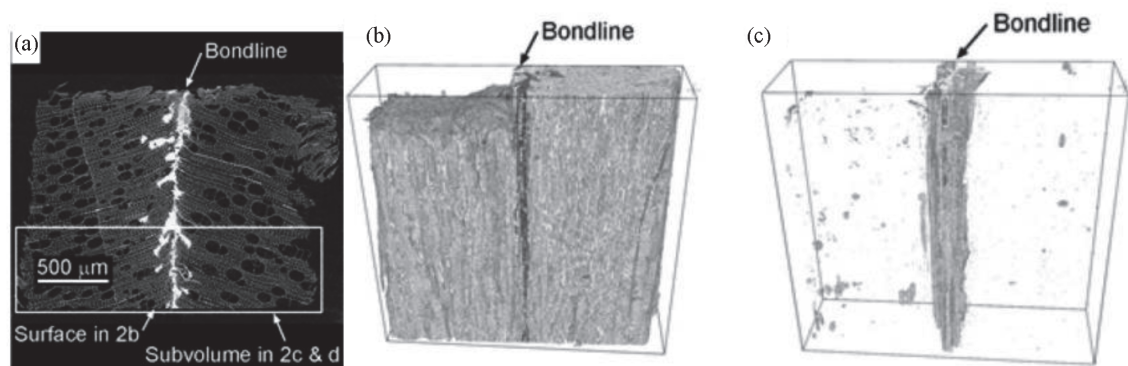

Figure 15.
X-ray images of poplar glued using iodinated PF, (a) cross section, (b) wood and bond line in 3D, (c) 3D bond line without wood sections [40].
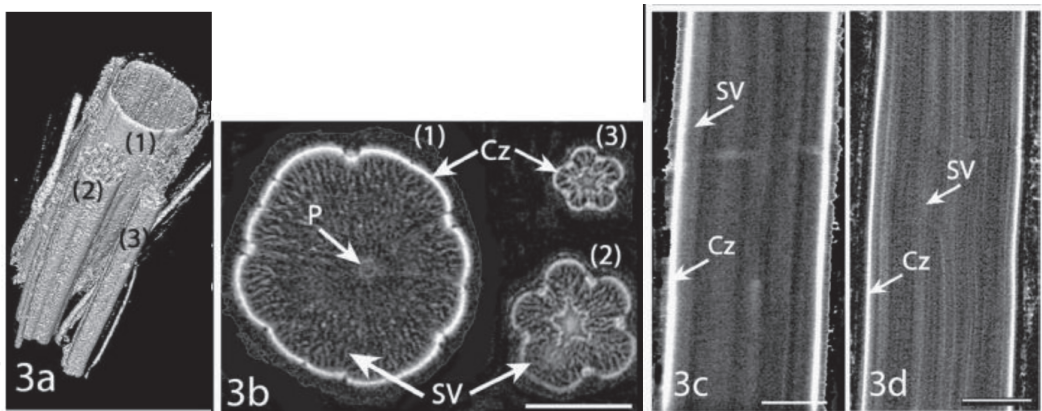
Figure 16.
Images of Quercus serrata *branch using MRI, (a) Surface-rendered image reconstructed from a 3D-FSPGR dataset. Bolts 1 to 3 correspond to the numbers in* **Figure 3b**. *(b) Proton density weighted crosscut images. TR, 2000 ms; TE, 25 ms. (c & d) A proton density weighted (TR, 2000 ms; TE, 20 ms) and a T1 weighted (TR, 540 ms; TE, 20 ms) radial image, respectively. Pixel size 156 μm. — Scale bar = 1 cm [52].*

Infrared cameras are the most common types of detecting systems using electronic detection of infrared emitted from the sample [49]. These methods are used in determining the integrity of the surface and sub-surface, for example in wood based composites [53].

Scanning thermal microscopy (SThM) uses two different modes to detect the changes in chemical composition of samples and map out the adhesive penetration in the structure using a thermal probe. The thermal contrast microscopy (TCM) measures the changes in temperature of the sample surface. Conductivity contrast microscopy (CCM) also measures the sample's surface conductivity as the temperature is kept constant [54]. Images of wood phenol-resorcinol-formaldehyde-adhesive bonds (PRF) using SThM are presented in **Figure 17**.

In microwave techniques, Dielectric properties of the sample structure are used to image the structure in microwave imaging methods. This method is commonly used for assessment of wood products after drying and gluing, for detecting any internal detects such as knots, spiral grains, discontinuation in structure of logs, lumber and wood based composites [49].

Ultrasonic methods are low cost methods for detecting the adhesive content of wood products with a high sensitivity to delamination, however achieving imaging with high resolution is difficult and coupling pressure dependency reproducibility
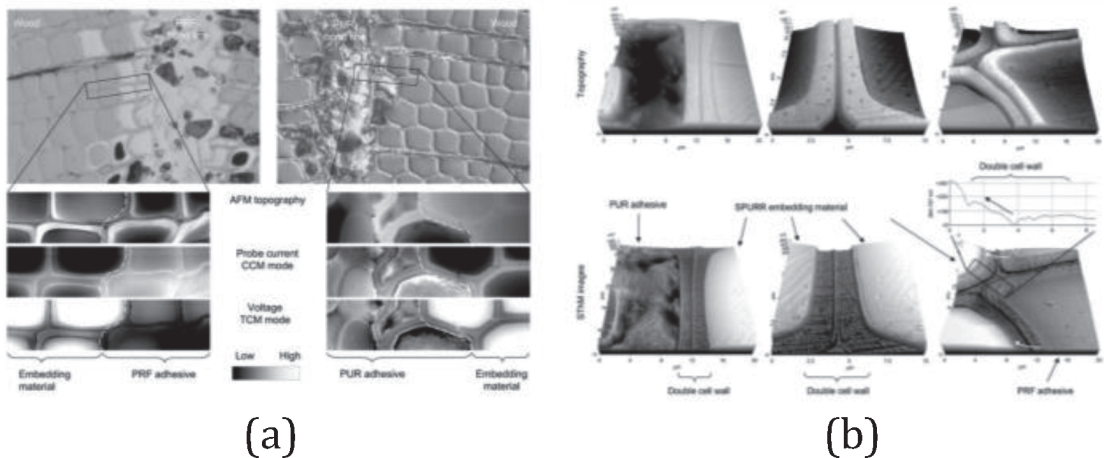

Figure 17.
(a) Images of adhesive bond in spruce samples using (top): Light microscopy and (bottom): SThM mapping the topography, probe current and voltage and (b) differences in topography for SPURR epoxy resin (Centre) and in contact with PUR adhesive (left) and PRF adhesive (right).

of this technique is poor [44]. There have been reported studies on the uncertainty around voltage values that the tranducer recorded (20–25% variation) where extra applied hand pressure is required for ultrasonic readings [55]. Air coupled ultrasonics systems however have shown promising results eliminating this variation which requires future investigation on its effectiveness in detecting complex glued sections (**Figure 18**) [44, 55].

FTIR imaging of chemical components (Fourier Transform Infrared) provides details of functional groups based on absorption, of chemical band intensity, band areas, and position. **Figure 19** shows the FTIR readings for wood and Polymeric diphenylmethane diisocyanate (pMDI) in uncured and cured conditions [56]. This study reported on the challenges in detecting glue formation in the cell walls using this method.

Chemical imaging is used to map out the intensity and distribution of different components in wood structure [57, 57]. The study used non-covalent interaction of
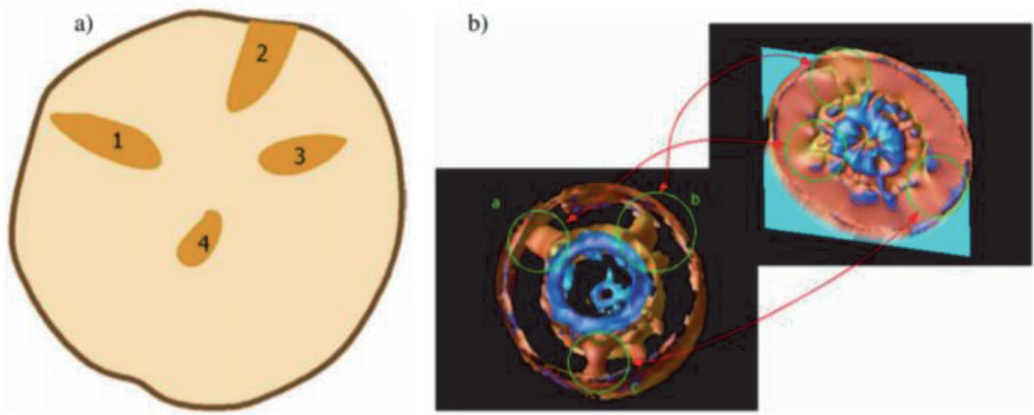

Figure 18.
Microwave technique used to detect knots in radial (a) and reconstructed image (b) [49].
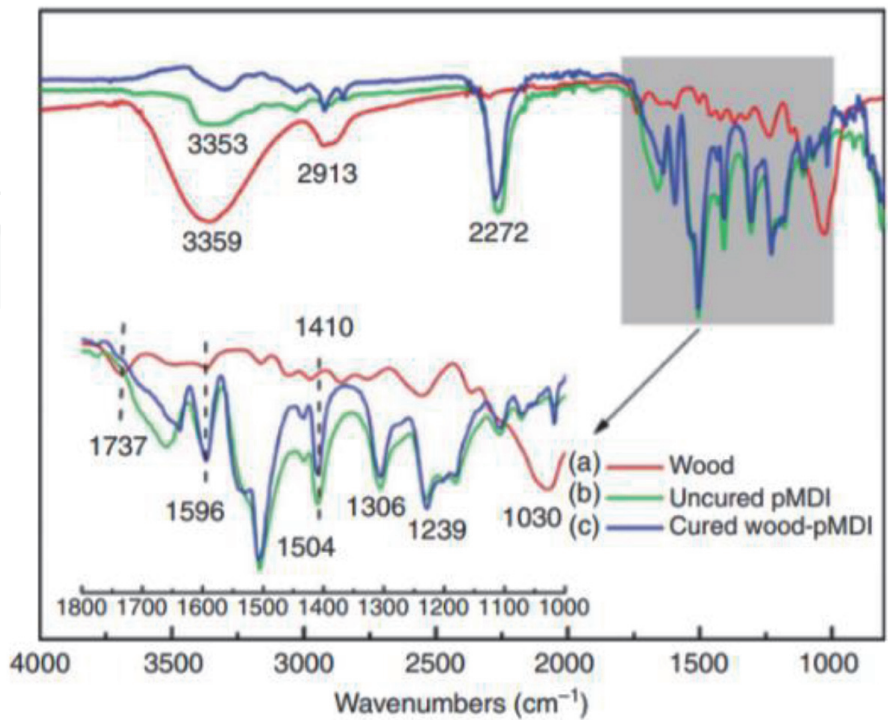

Figure 19.
Results of imaging using FTIR spectra of loblolly pine (Pinus taeda L.) wood and pMDI, and wood-pMDI [56].

acetylated nanocrystalline cellulose (AC-NCC) with polylactic acid (PLA) in material tested as an indicator for chemical imaging. **Figure 20** shows the readings for different chemical components in the samples including visible light image in left column, mCH2 absorption maps in middle column and integrated mC=O second derivative absorption peak areas in right column. The results showed that FTIR imaging can be used effectively to image the chemical links between a substrate and functionalised filler [57].

3.5 X-ray photoelectron spectroscopy (XFS)

As discussed earlier, X-ray computed tomography (XCT) was used after adding the bromine substituted phenol formaldehyde (BrPF) to increase the X-ray
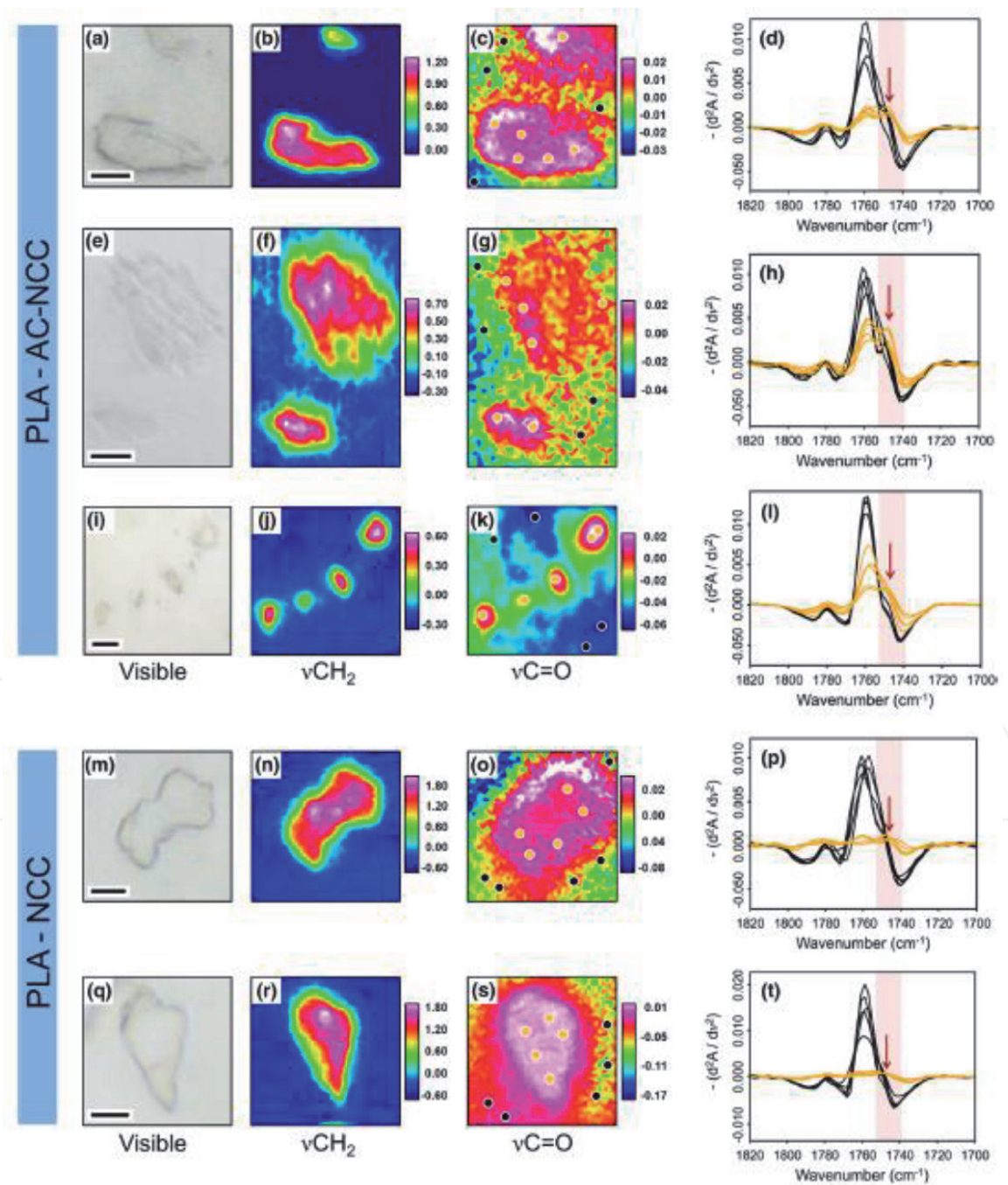

Figure 20.
Results of absorption map for different particles using chemical imaging and FTIR micro spectroscopy for composite materials [57].

attenuation and capture a brighter glue line which is distinguishable from wood cells [58].

This study also used X-ray fluorescence microscopy (XFM) to acquire images of the bond line [59]. This study investigated the required adhesive (BrPF) flow and infiltration to develop an effective glue bond in loblolly pine (*Pinus taeda*). Four materials including BrPF, BrPF-mixed with wood, wood and air in the porous structure of wood was observed in the images as shown in **Figure 21** (left image (b)). The study indicated some uncertainty around the wood composition with BrPF that was not included in the 3D reconstruction of the image. **Figure 21** shows the X-ray and X-ray fluorescence image and the sections in red circles that were not used in 3D reconstruction. The potential inclusion of wood cell walls infiltrated with BrPF or another material interface in the images could be a limitation in 3D imaging with XCT that could be overcome using XFM. Image on the right in **Figure 21** shows the consistency of XCT images.

The XCT method provides the option of visualising the flow of BrPF and penetration into the 3D wood structure over time. In sections with less clarity of BrPF mixed with wood, XFM provided a detailed observation for the glue presence inside the cell structure of the wood (**Figure 22**).

A study of PF in wood cell walls using XFM in combination with nanoindentation showed that using BrPF enhanced the cell wall matrix (see sections showed in **Figure 21**). The different molecular weights (MW) of BrPF tested showed that the lower MW had greater effects on preventing the softening of cell walls due to gained moisture. In **Figure 23**, the glue line is shown by arrows and the dashed white line shows the cells that were tested for mechanical strength using nanoindentation [59].

Nano indentation and scanning probe microscopy (SPM) imaging of year old Masson pine (*Pinus massoniana* Lamb.) showed the advantages that SPM could provide in investigating the cellular strength [60]. **Figure 24** shows the detailed images of cells and modified (PF resin (as thermosetting agent) impregnated samples at 15, 20, 25 and 30% treatments) cells. This method can be used in detecting the presence of glue in various sections of the cell wall and its effects on cell wall strength as the thickness of glue and the properties of bond line (thickness and depth of penetration) varies.
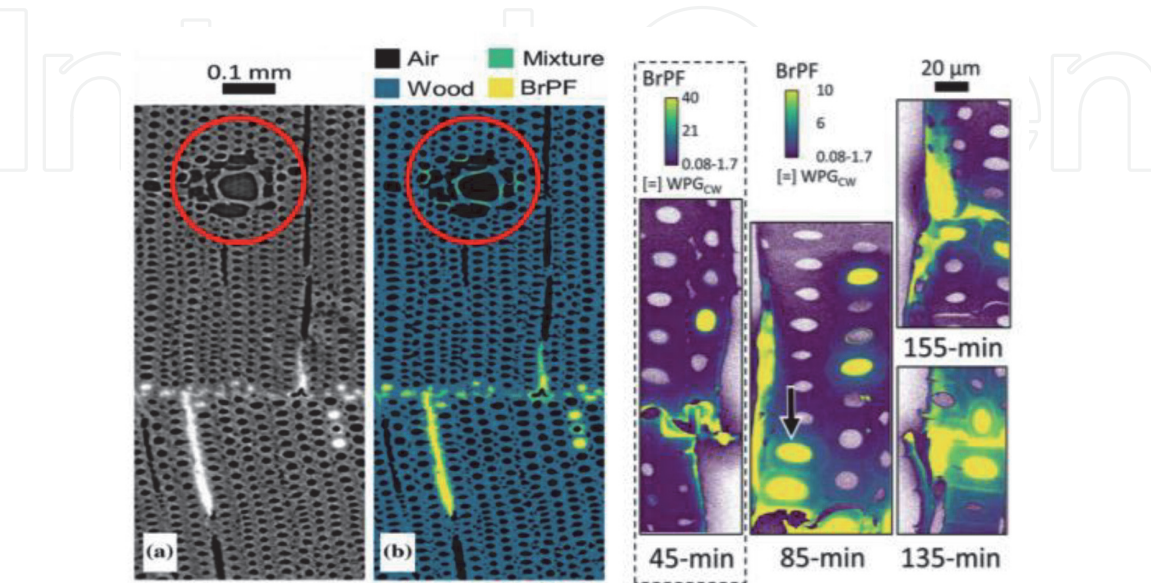

Figure 21.
Left- X-ray computed tomography (XCT) and right- X-ray fluorescence microscopy (XFM) images of glue line [58].
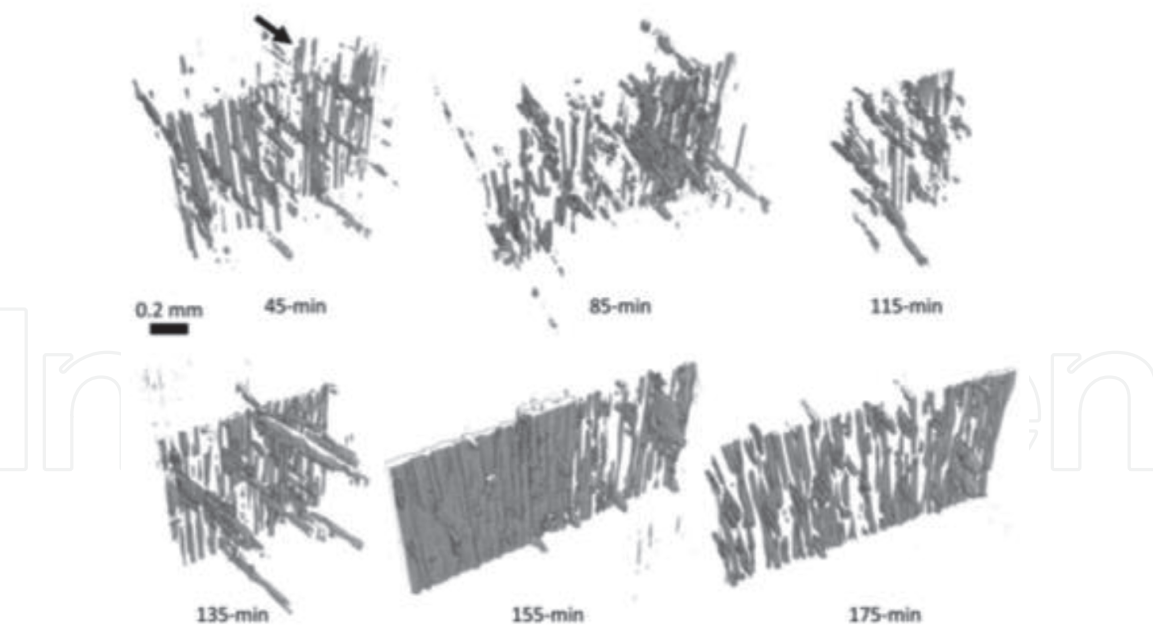
Figure 22.
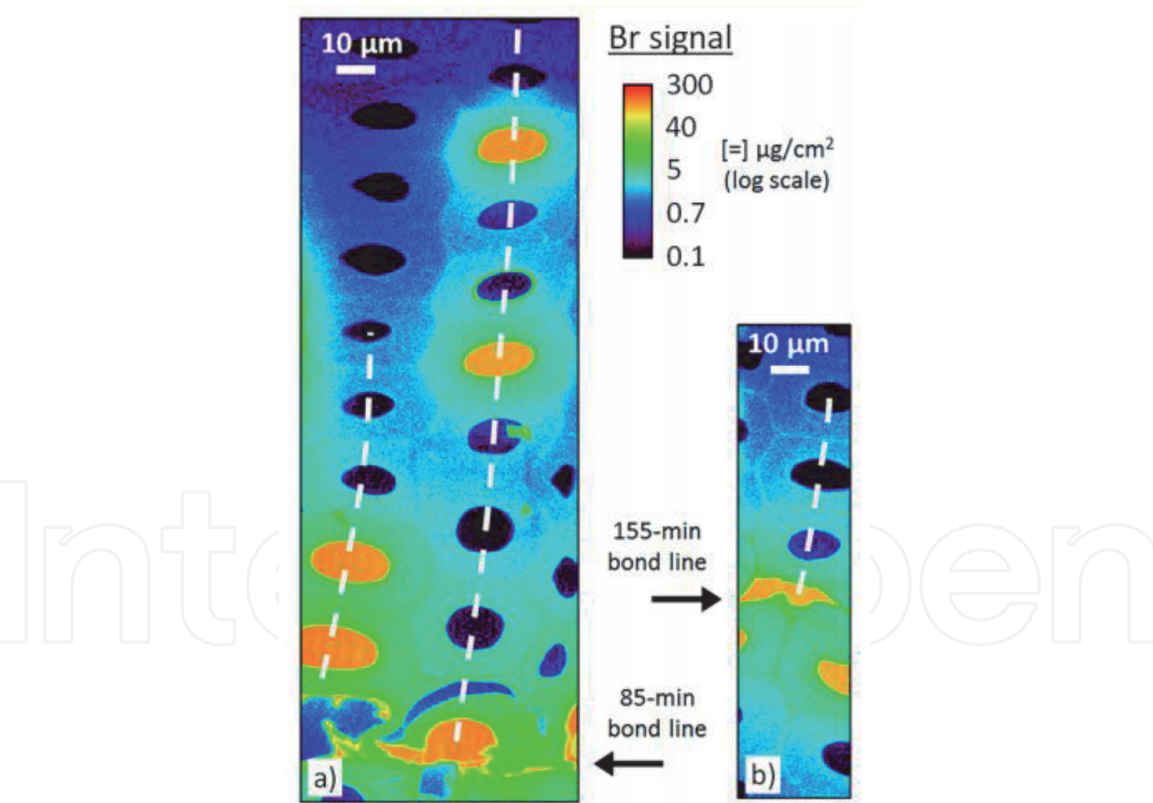*Flow of BrPF into wood structure over time [58].*

Figure 23.
XFM used to map Br penetration into wood cell structure, (a) after 85 min BrPF and (b) 155 min samples [59].

3.6 Epifluorescence microscopy (EPI)

EPI microscopy can provide imaging of the cell structure from the surface of samples used for imaging.

A study of adhesive penetration in particle board composites using EPI showed this microscopic method was effective for quantitatively assessing glue interaction
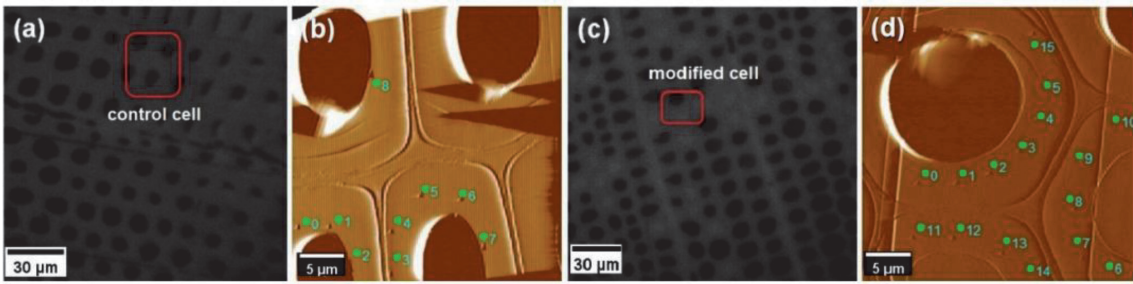
Figure 24.
Images of Masson pine (a and c) under microscope and (b and d) under SPM equipped with nano indenter probe [60].

with wood. The study determined adhesive filled fibre numbers (FFN), adhesive filled vessels number (FVN), maximum adhesive penetration depth in fibre (MPD_f) and in rays (MPD_r). **Figure 25(a)** shows the results of EPI microscopy in poplar samples at different moisture content (2, 6 and 10% MC) [34]. Penetration of resin was higher in higher moisture content wood. **Figure 25(b)** shows results of EPI imaging of three UF adhesives in poplar in radial and tangential directions. The images taken showed clear sections where glue penetrated into vessels, rays as well as showing the bond lines. Three UF adhesives were tested at different viscosities including UF I, 545 mPa.s < UF II, 745 mPa.s < UF III, 1644 mPa.s [36].

This study showed penetration into the radial direction was lower than penetration in tangential direction which could be related to the presence of pits (on radial walls) that facilitate the adhesive movement in tangential direction. Shear strength of specimens increased as penetration increased and was higher in adhesive with lower viscosity (due to better flow of adhesive) [36].

3.6.1 Confocal laser scanning microscopy (CLSM)

Confocal laser scanning microscopy (CLSM) provides an optical depth selection option to image the sections of a structure that are usually very hard to physically prepare. Imaging of glued sections using CLSM under FM provides a clear representation of the glue line and its borders in wood structure. **Figure 26(a)** represents the images taken from a coated wood surface in a study investigating the durability of the coating material used and penetration into surface cracks of radiata pine (*Pinus radiata*). The comparison with light microscopic imaging showed the advantages of using CLSM in showing the details of penetration into cell lumina and fine surface cracks [61].
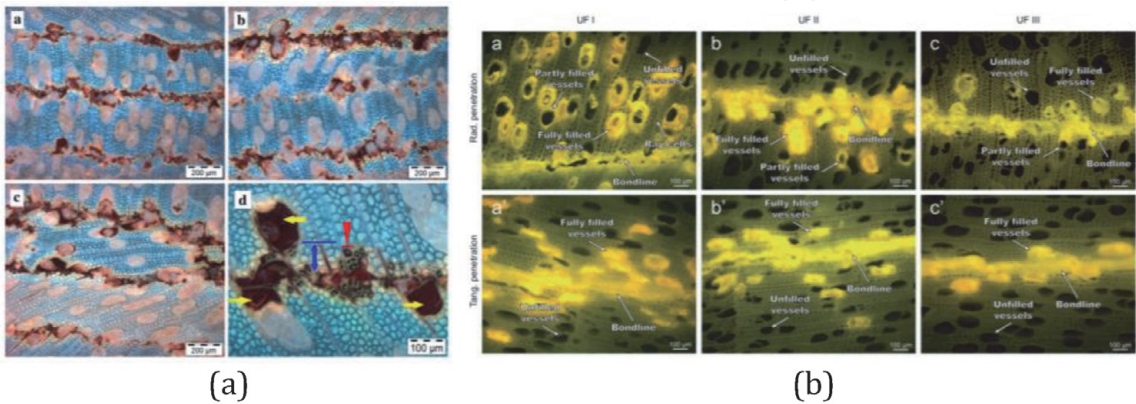

(a)   (b)

Figure 25.
(a) Images of UF penetration into wood structure using EPI [34] and (b) EPI imaging of adhesive penetration into different planes of poplar (a, b and c are in radial and a' b' and c' are in tangential directions) [36].
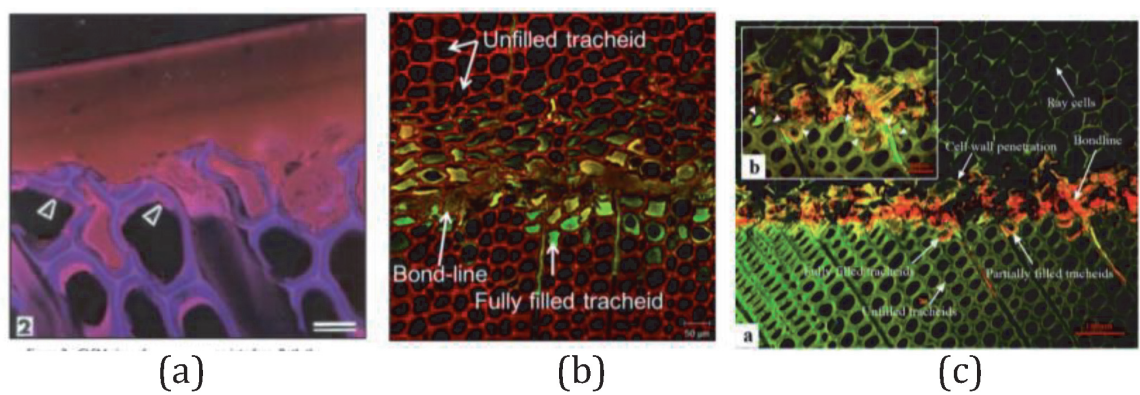
Figure 26.
CLSM used in (a) imaging coating surface and penetration of coating material into the wood structure (bar = 20 µm) [61], (b) urea formaldehyde (UF) resin penetration into wood structure [62] and (c) UF adhesive imaging showing wood structure and resin penetration [9].

The separation between urea formaldehyde (UF) resin and wood cells is not usually easy with common microscopic methods; **Figure 26**-right show images of UF resin using CLSM [9].

Figure 26(b) shows results of CLSM of UF resin penetration in radiata pine (*Pinus radiata D. Don*) veneer layers (2 mm thick) of plywood including fully adhesive filled and unfilled tracheids and the glue bond line developed [62].
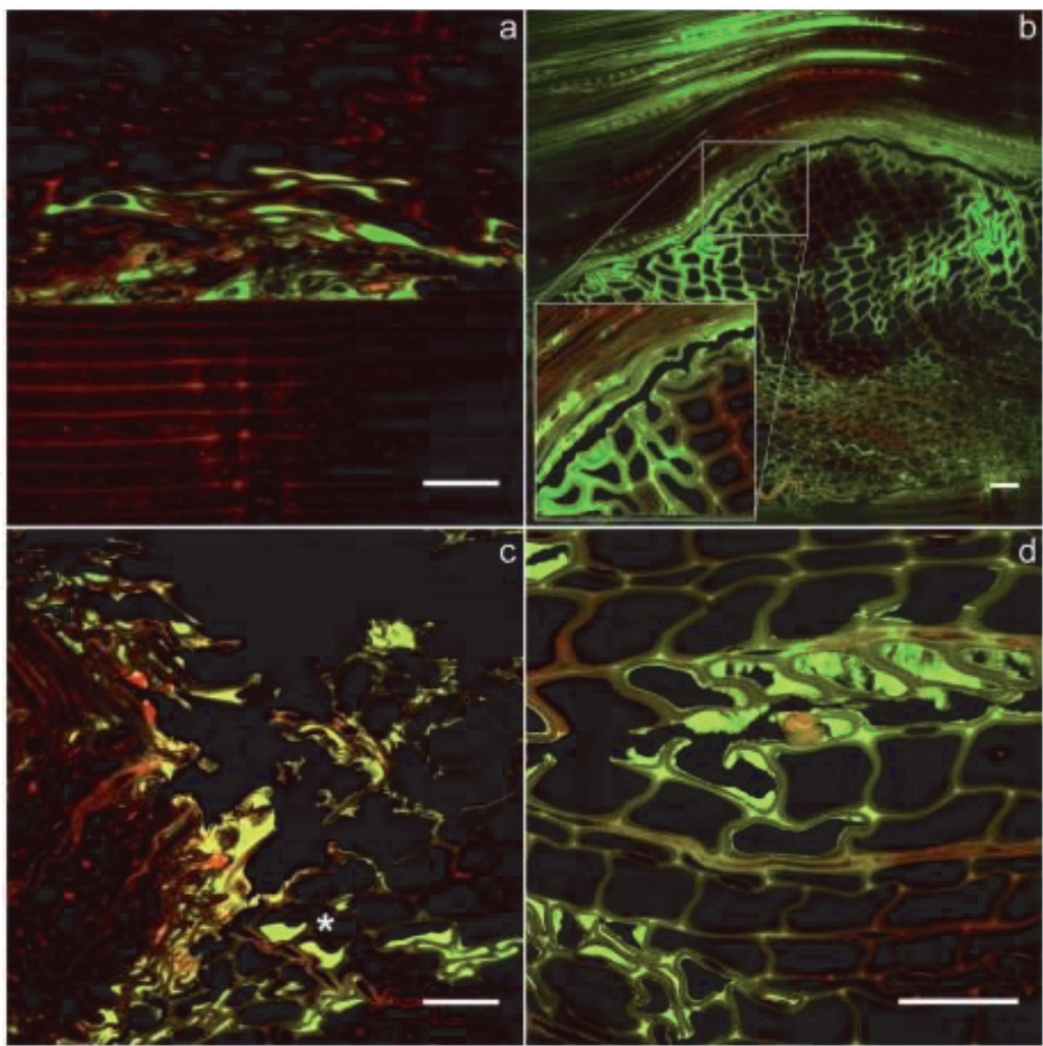

Figure 27.
Images of particleboard section glued using fluorescence-labelled UF resin, bar = 50 µm [63].

Figure 26(c) shows results of CLSM imaging of UF resin in samples of *Cryptomeria fortune* Hooibrenk. This study showed that UF resin penetrated in axial tracheids and ray lumens and cracks around the glue line. Imaging showed that glue penetrated easier in the direction of lowest resistance (shown in image by arrows) to the compression applied by the press machine [9].

CLSM has also been used in imaging different particle board samples targeting the UF resin component in board structure enabling the quantification of resin percentage in the total board area. This study which combined CLSM and fluorescence labelled UF (**Figure 27**) showed clear sections of cells and glue penetration into the structure of boards (made from Norway spruce (*Picea abies* (L.) Karst.) [63]. Singh et al. used [61, 64] confocal florescence micrographs to capture the adhesive penetration into the wood cell structure as well as detailed information on interlocking of cells by glue (see **Figure 28**). CLSM is a new imaging technique that
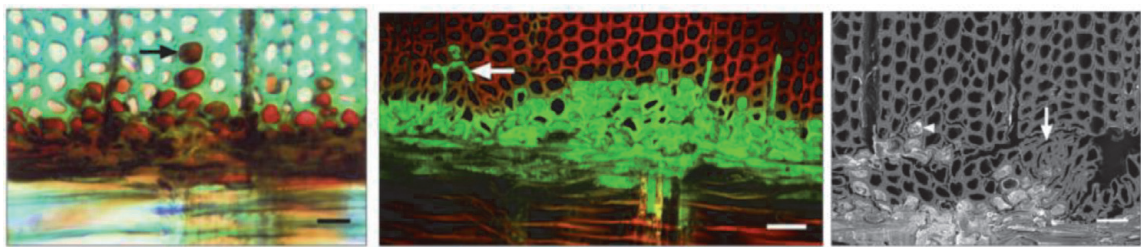

Figure 28.
Left- light microscopy image with high magnification, middle- image taken using confocal florescence micrograph and right- SEM image [64].
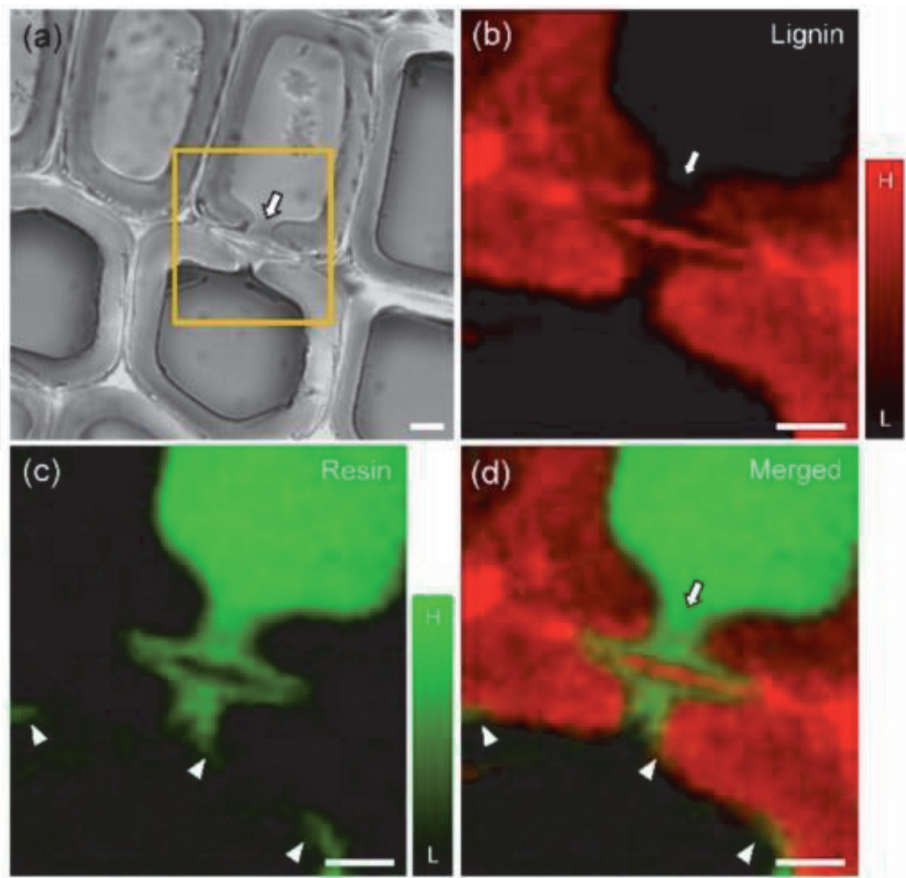

Figure 29.
Raman mapping on cross section of coated samples of Japanese cedar (a) Raman image (b) lignin, (c) alkyd resin and (d) merged information adrenal glands [65].

has capabilities in enhancing the contrast between the wood and coating or adhesive materials to improve the image quality and clarity.

3.7 Chemical mapping - FTIR microscopy

Chemical mapping and imaging of biological and biometric samples- FT-Raman spectroscopy- has recently been used in studying wood including coatings, degradation and modification of cell walls. Similar to NIR detecting systems, Raman spectroscopy uses the vibrational transitions [65]. However, Raman spectroscopy uses the changes in polarizability of functional groups while NIR uses differences in infrared absorption to detect chemical components. A study of Japanese cedar (*Cryptomeria japonica* D. Don) using confocal Raman microscopy showed clear details of the structure and chemistry of wood including distribution of alkyd resin and lignin (**Figure 29**). The study showed the great capability of Raman microscopy in detecting different chemical components of wood and wood coating materials at the cellular level of structure.

Similar work done by Wang et al. [60] showed the advantages that Raman spectroscopy can provide in studying the penetration of PF resin into the cellular structure of Masson pine. The images indicated the penetration of PF resin into cell lumens as well as into cell walls. The images also show the interaction of the PF with polymers in the cell walls. The nano indentation done on cell walls in combination with the Raman spectroscopy conducted showed that the added PF resin in the wood structure reduced the dimensional changes of the structure resulting from swelling and shrinkage [60].

4. Summary and future directions

The review focuses on existing studies and techniques used for visualising adhesive bonds in various timber products. This review highlighted the capacity, advantages, limitations and the potential of the different visualisation methods including Microscopy (visible light, UV, IR), Electron Microscopy (SEM, ESEM, transmission electron microscopy (TEM)), X-ray computed tomography (XCT), Thermal techniques, Microwave techniques, Ionising radiation, Ultrasonics, FTIR imaging of chemical components, X-ray imaging, Epifluorescence Microscopy (EPI), Confocal laser scanning microscopy (CLSM) and Chemical mapping. The majority of work published has focused on applications and types of wood used for specific products rather than developing/following a standard protocol in determining definitions for adhesive bond development and measurements. These studies are mainly qualitative and comparative only to samples tested in each study. The review suggests that there is a need for developing standard definitions for glue bond shape, thickness, penetration depth and scale in both 2 and 3D in order to be able to quantitatively assess the effectiveness of gluing process for various timber products. The effects of wood types, anatomical structure, gluing properties, curing mechanisms and glue types used for different applications/products need to be studied for different structural and non-structural timber elements. The review also highlighted the need for better definitions of glue lines, penetration in relation to wood grain direction and cellular details which could affect the penetration effectiveness depending on surface wetting properties, extractive content in cells and size of cells. The interaction between glue, cell walls and chemical extractives of different wood types before and after glue application and product manufacturing will need to be carefully studied and a summary of potential differences and similarities for each type of timber product can be developed for industry to access.

The existing literature shows the strength and capacity of different visualisation methods for analysing the adhesive effectiveness in different wood types, however more research is required to examine the potential of each method for a range of timber products and wood types (e.g. soft vs. hardwoods). The 3D imaging can provide detailed information on adhesive bonds in various conditions including where there is variation in MC, glue type (different curing requirements and curing time) and wood type (including low to high density and for soft and hardwood).

In the use of X-ray scanning where density differences between the wood and glue are the main visualisation factor, further studies are needed for investigating the effects of adding contrast agents (such as iodine) to adhesives and the potential effects on glue properties that could lead to bond line variations and changes in penetration patterns.

Further work is required to monitor potential effects of industrial processes and environmental conditional factors on bond formation and adhesive penetration during and after production and in the service life of timber elements specially for structural products such as glulam, CLT and LVL.

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