

Quantitative analysis of commercial coating penetration into *Fagus crenata* wood using X-ray Microtomography

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Abstract

Recently, coating has been used as economically friendly materials to enhance the dimensional stability and aesthetic properties of woody materials as a preservative. X-ray microtomography is a promising visualization technique for observing the coating penetration plots. The study aimed to determine the efficiency and applicability of X-ray microtomography for observing and assessing the distribution of coating materials in two- and three-dimensional visualizations. The coating assessments were investigated using X-ray images and plot profile to obtain the penetration depth. Some Japanese commercial coatings were used for the experiments. Two types of coating application such as spray and brush were performed. Each coated sample was scanned using X-ray microtomography, and the coating penetration depth was successfully visualized and quantified. The chemical content in the coating materials and its concentration have been proven to affect the coating penetration plots and amount. The X-ray images has successfully showed penetration plot and assessed the penetration value at the microscopic scale.

Introduction

The use of film-thin layers has become ubiquitous practice across much of the scientific and industrial sector in the recent times. The film-thin layer which is applied to an object or substrate is defined as coating. Synergistic action between the characteristics of the substrate and the covering material were obtained when coatings are used¹. Coatings can enhance physical, chemical, and aesthetic properties and lower the costs of the final product. Coating applications such as varnish, stain, and paint, are commonly used in the furniture industry.

The interactions between coatings and natural material like wood and the very complex formulations of coatings made predictions of their service life and performance more challenging². Coatings are used to obtain a synergistic action between the characteristics of substrate and the covering materials³. The well-known fact is that the coating performance, namely adhesion, depends on a hygroscopic characteristic of wood composite panels⁴. Therefore, the type of coating plays an important role on its performance such as film thickness which affects the abrasion resistance and the adhesion of the coating itself. Finishing characteristics of coatings on wood surfaces depend on many variables, of which the coating-film thickness is an important one⁵. In addition, the penetration of the coating into the wood is important for several reasons. Firstly, coating penetration can improve water repellency and dimensional stability and reduce the surface cracks. Second, the adhesion of the coating to the wood material will benefit from a certain degree of penetration as well such as mechanical entanglement or interlocking, adsorption interaction by polar and dispersive forces, and intermolecular diffusion. Third, coatings containing fungicides might have improved penetration of the fungicide because it penetrates together⁶.

The fundamental understanding of the interactions between wood and coatings is desirable. In a previous study, apart from the anatomical structure of the wood, coating penetration is mainly determined by the ability of the coating to flow into the lumina of either tracheid or ray-cells⁶. Most likely the flow of a coating through the wood capillaries will be influenced by many variables like viscosity, surface tension, drying rate of the coating and the diameter of the wood capillaries. Thus, the anatomical microscopic penetration is well-known recently.

X-ray is a non-destructive method and provides 3D images for internal structure material that has grown in application to wood. Previous study conducted by Ekaputri *et al*⁷ about the applicability of X-ray microtomography to visualize the coating penetration phenomena into the wood using two different X-ray target such as Cu and Mo, successfully visualized the Japanese commercial coating into Japanese beech wood⁷. In addition, the potential of X-ray measurement for quantitative analysis that observed the dissolved CsCl concentration in the wood during the drying process had been described by Tanaka and Kawai⁸. Dealing with coating materials, Bulcke *et al*⁹ have proposed a detailed coating analysis in and on the wood suggested layer thickness and penetration depth can be calculated using X-ray as a non-destructive technique. Therefore, X-rays might have the potential to quantify chemicals that penetrate deep into the wood three-dimensionally.

Accordingly, the main purpose of this paper is to introduce the effectiveness of the X-ray microtomography technique for quantitative measurement of 3D wood coating penetration. The applicability of X-ray microtomography to distinguish anatomical elements of wood and coating materials using some Japanese commercial coating was checked. The effect of the type of coating in the application method and chemical content was also discussed. In addition, the effect of anatomical features or structure were discussed.

Methods

Sample preparation and coating application

Four rotary veneers of *Fagus crenata* Blume with dimension 300 mm × 300 mm × 1.0–1.3 mm was prepared. The veneers were dried in a constant temperature oven for 24 hours at 60 °C. After cooling down, the veneers were cold pressed and hot pressed at 125 °C with 1 MPa pressure for 20 mins and 4 mins. The veneers were cooled down for 24 hours in plastic bag and cut into twelve veneers with dimension 100 mm². One veneer was used as a control (no coating applied), six were coated using some Japanese commercial coating with spray application, and two were coated using brush application. The coating specifications are listed in Table 1.

The coating materials were applied onto the veneer with a 2-coat application with spreading rate approximately 280 g/m², as suggested by Kinoshita¹⁰ as the typical spreading rate for topcoat on hardwood surface. The application method and the interval of each spray-type coating and brush-type coating was 30 mins and 1–2 days referred to the technical data sheet published by the manufacturer of the coating materials (Asahipen). After coating, all veneers were air-dried for 24 hours. The coating thickness was calculated using Equations 1, respectively, and is presented in Table 2.

Table 1
Coating specifications

Coating Specifications						
Material Name	Color	Application	Coating Base	Ingredients That Might Affect X-ray Interaction	Percentage (%)	Chemical Formula
Aspen Lacquer Spray	Brown (BR)	Spray	Oil-based	Titanium Dioxide	0.1–1	TiO ₂
	Gold (GL)			Brass (Copper/Zinc)	1.0–5.0	Cu/Zn
	Matte White (MW)			Amorphous Silica	1.0–5.0	SiO ₂
				Titanium Dioxide	1.0–10	TiO ₂
	Pink (PK)			Titanium Dioxide	1.0–10	TiO ₂
	Silver (SL)			Aluminum	1.0–5.0	Al
	White (WH)			Titanium Dioxide	1.0–10	TiO2
Outdoor xylem protective paint WOOD oil-based	Light Oak (LOO)			Amorphous Silica	0.1–1	SiO2
				Ferric Hydrated Iron Oxide	1.0–5.0	Fe(OH)O
		Brush	Oil-based	Cobalt 2-Ethylhexanoate	0.1–1	[CH ₃ (CH ₂) ₃ CH(C ₂ H ₅)CO ₂] ₂ Co
Outdoor xylem protective paint WOOD water-based	Light Oak (LOW)			Amorphous Silica	0.1–1	SiO2
				Ferric Hydrated Iron Oxide	1.0–5.0	Fe(OH)O
		Brush	Water-based	Titanium Dioxide	0.1–1	TiO2

Table 2
Coating thickness

No	Sample Color	Wood initial thickness (μm)	1st coat	2nd coat	Coating Thickness (μm)
			coated wood (dry) thickness (μm)	coated wood (dry) thickness (μm)	
1	Brown (BR)	1002	1007	1019	17
2	Matte white (MW)	995	1018	1032	37
3	Pink (PK)	1019	1022	1034	15
4	White (WH)	985	995	1020	35
5	Gold (GL)	998	1009	1033	35
6	Silver (SL)	1028	1037	1054	26
7	Light Oak oil-based (LOO)	980	997	1011	31
8	Light Oak water-based (LOW)	958	987	1001	43

X-ray microtomography scanning

The coated veneers were cut into dimension 10 × 50 mm based on FOV (field of view) of the apparatus which used lens L1080, FOV φ3.6 (diameter) × 2.8 (height) mm. All of X-ray specimens of all coating materials were scanned using X-ray microtomography apparatus (Rigaku Nano 3DX Fuji, Japan). The Mo X-ray target with the tube voltage 50 kV and the tube current 24 mA with an 0.1 mm thick aluminum (Al) filter (for generating X-rays with higher energy) was used. The samples were put in the sample stage of the apparatus with the distance from the center of the sample stage to the 8 mm lens, and the stage height was adjusted as the six veneer samples were all in the field of view (Fig. 1). The acquisition settings were binning 2 resulting in 2.2 μm/pixel, 6 s exposure time, angular step at 0.2° during the sample rotation through 180°. The binning level used was recommended by Oishi and Tanaka¹¹. After scanning, the CT reconstruction process was implemented using a computer connected with the apparatus.

Penetration analysis

The X-ray image stack obtained from each coating that was scanned after the reconstruction process had 1234 images with the resolution 1648 × 1648 pixels. The coating penetration analysis was performed using ImageJ software with processing the image stack obtained. Determination of each axis for three-dimensional penetration plot was carried out using VGStudio Max software with 3D image obtained as explained in Fig. 2. After image slicing, the grey value was analyzed using ImageJ to obtain the penetration depth value.

Results and Discussion

2D X-ray image visualization

The 2D cross-sectional images (tangentially sliced) of control samples and some coating samples are shown in Fig. 3. The coating distribution on and in the veneer was successfully observed using X-ray microtomography. The visibility of the coating that successfully visualized using X-ray microtomography could be caused by the high number of ions in its chemical composition, which is in accordance with the results of preliminary work by Ekaputri *et al.*⁷. The coating part and woody materials are visible and can be distinguished clearly. The coating that is visible on the surface of the wood implies the white line while the coating that is visible inside the wood vessels implies the white dots. This follows the untreated wood image results which didn't have any white lines or white dots. Each coating showed a different phenomenon. Some coatings showed a deep penetration into the wood vessels while some of them only appeared on the wood surface. As mentioned in the previous study, this image result didn't have any artifact and resulted in a good image quality that was decent for the next step which was quantitative analysis.

Preliminary of three-dimensional penetration plot

The use of a three-dimensional penetration plot is a convenient way to track the coating on- and inside the wood, especially in the vessel sections. The preliminary analysis can enable quantifying the coating penetration amount at some tangential and longitudinal point (axis) by slicing the image from the 3D image stacks and by calculating the penetration distance from the image and its grey value to form a 3D visual penetration plot. Figure 4 shows the 3D penetration plots obtained for each coating material. The color level from purple to red indicates the different penetration depth values, respectively. In addition, the average coating penetration amount of each coating had been calculated and explained in Fig. 5. The average coating penetration calculation shows a different value of each coating.

Regarding the coating thickness and penetration as shown in Fig. 4, it implied that the coating application that was used such as spray and brush application method did not really affect the penetration distance. The deep penetration was shown in Fig. 4b, 4c, 4d, and 4e for the spray application method and 4i for the brush application method. Moreover, the coating that appears almost only on the surface of the wood for both application methods was shown in Fig. 4f, 4g, and 4h, respectively.

The coating consists of the solvent and dissolve substances. The solvent, commonly referred to as resin, plays a crucial role in ensuring even coating distribution by evaporating and allowing the active coating material to adhere to the wood surface and penetrate into it. The resins that were used in this research were oil-based and water-based. According to Fig. 4, the resin type had no significant effect on the coating penetration distance. However, in the case of the brush application method, light-oak oil-based (LOO) and light-oak water-based (LOW) coating showed different phenomena. The LOO coating mainly appeared on the surface of the wood, while the LOW coating showed the deep penetration. This phenomenon might have happened because of the dissolved substances of the coating chemical content and the viscosity of the coating material itself.

The Fig. 4 implied that the coating contained TiO_2 (BR, PK, and WH) was distributed uniformly to a certain degree throughout the veneer as shown as their peak is the highest, whereas the coating that contains TiO_2 with additional chemical (MW) or other than TiO_2 (LOW, LOO, GL, and SL) was mainly present on the surface. The brown, pink, and white coating (Fig. 4b, 4c, and 4e) have the same active chemical content that is TiO_2 but showed different penetration phenomenon. The deepest penetration amount of brown, pink, and white coating was 569 μm , 625 μm , and 730 μm makes the white coating has the deepest penetration compared to brown and pink coating. Moreover, the average penetration among these coatings (Fig. 5), showed 227 μm for brown coating, 169 μm for pink coating and 299 μm for white coating. This phenomenon could be caused by the chemical constituents' concentration in the coating materials which will affect the anchoring and penetration power of the coating itself. With the same active chemical content and addition of silica, matte white coating had shallower penetration amount at 384 μm . The average penetration amount of matte white coating was 153 μm . This result implied that the silica has some effect on penetration distance suggested further investigation needed to observe the interaction between silica and the wood chemicals that influence the penetration rate.

Besides TiO_2 , there are coatings that contain other chemicals such as copper and zinc (gold coating), and aluminum (silver coating). These two coatings mainly found on the surface (Fig. 4f and 4g). The deepest penetration amount of these coatings was 140 μm and 50 μm . Average penetration amount for these coatings was 36 μm and 2 μm . This phenomenon occurs probably because the chemicals used in both coatings have difficulty getting into the wooden vessel.

The LOO and LOW coating have Ferric as the main coating active chemical content with addition of amorphous silica and cobalt for LOO coating, whereas the LOW coating has amorphous silica and TiO_2 as additional active chemical. Even though both of them claimed to have the same color which was light-oak, the active chemical content and resin used are different. This caused a significant difference in penetration value shown in Fig. 4i and 4h. The LOO coating had shallower penetration with 60 μm as its deepest penetration value whereas LOW had 516 μm . The average penetration amount for these coatings was 0.78 μm and 142 μm . This phenomenon implied that the cobalt might has some effect on penetration distance which further investigation is suggested.

According to the results, the various phenomenon and three-dimensional penetration plot was successfully visualized and calculated using μXCT . The coating active materials had a significant effect on the coating penetration distance. In addition, further investigation was suggested for further understanding regarding silica and cobalt's interaction with the wood materials.

The coating penetration depth seems to depend on the nature of the substrate and its anatomy which is presented in Fig. 3. All images from X-ray microtomography are presented at the same scale. The appearance and disappearances of coating materials inside the certain vessels was successfully visualized. This phenomenon implied that the coating flows through the wood vessels. In addition, the coating in the vessel can be verified based on the intersection of the three section such as transverse, radial, and tangential as explained in Fig. 6. The blue, green, and red layers indicate the active 2D image

on the transverse section, radial section, and tangential section respectively. This discovery could be used as verification that clarify the penetration phenomena and coating quantitative analysis.

In conclusion, the use of μ XCT visualization images can be used to investigate the penetration coatings in the veneer and quantify its penetration amount resulting in easier quality assurance and more efficient in production line. This discovery is important for industrial aspects that might combine many wood species and coating products such as wood decking and tertiary wood products.

Conclusions

X-ray microtomography can be used to visualize coating penetration phenomenon into *Fagus crenata* veneer and quantified its penetration value by clearly distinguish between the wood vessels and the coating part three-dimensionally. The three-dimensional penetration depth plot shows unique phenomena of each coating based on its chemical content and concentration, with the highest is the white coating at 624 μ m. The active chemicals in the coating materials have implied to affect its ability to flow into the wood vessels suggested further investigation. The average penetration plots can quantitatively describe the coating thickness and penetration depth of coating materials to assess the quality related to the uniformity and heterogeneity of the distribution. The results described here were provided by semi non-destructive technique of visualization using X-ray microtomography, which is a promising technique to observe coating penetration into woody materials for quality assurance in industrial application.

Declarations

Author contributions statement

T.S.E. conceived the experiments; T.S.E., and T.T. conducted the experiments; T.S.E and T.T. analyzed the results; T.S.E. prepared and wrote the original draft; T.S.E. and T.T. wrote, reviewed, and edited the manuscript; T.T. supervised the research; and T.T. acquired the funding. All authors reviewed the manuscript.

Competing interests

The authors declare no competing interests.

Additional information

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Author Contribution

T.S.E. conceived the experiments; T.S.E., and T.T. conducted the experiments; T.S.E and T.T. analyzed the results; T.S.E. prepared and wrote the original draft; T.S.E. and T.T. wrote, reviewed, and edited the manuscript; T.T. supervised the research; and T.T. acquired the funding. All authors reviewed the manuscript.

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Data availability

The datasets used and/or analyzed during the current study available from the corresponding author on reasonable request.

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Figures

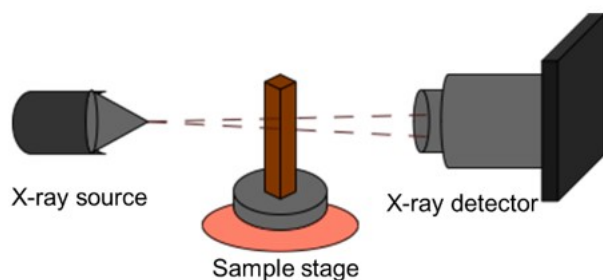


Figure 1

Schematic of the X-ray microtomography apparatus

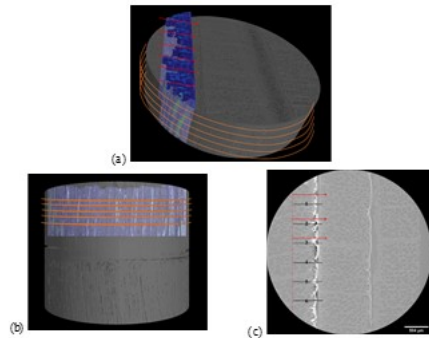


Figure 2

The mechanisms of partition of the wood-coated samples (a) The determination of axis; red arrow indicates the longitudinal axis and orange line indicates the tangential axis of wood-coated surface; (b) tangential cut; (c) longitudinal cut.

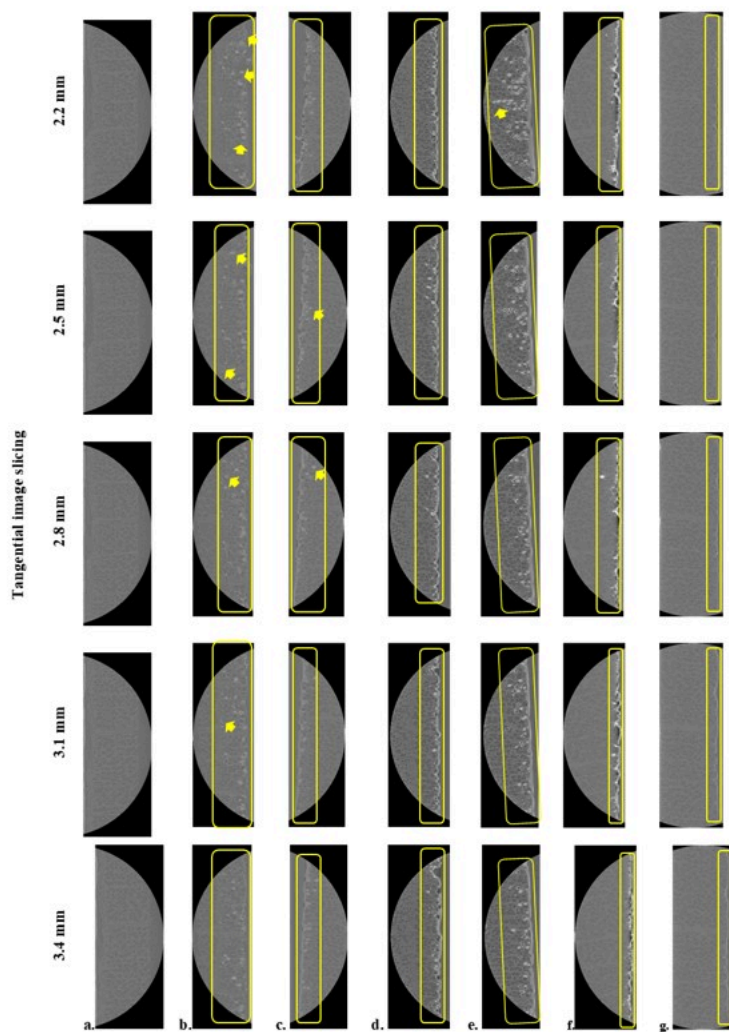


Figure 3

The 2D X-ray visualization of various coatings. (a) Control (no treatment), (b) Brown coating, (c) Pink coating, (d) Matte white coating, (e) White coating, (f) Gold coating, (g) Silver coating; yellow arrow indicates the coating and yellow line indicates the coating area.

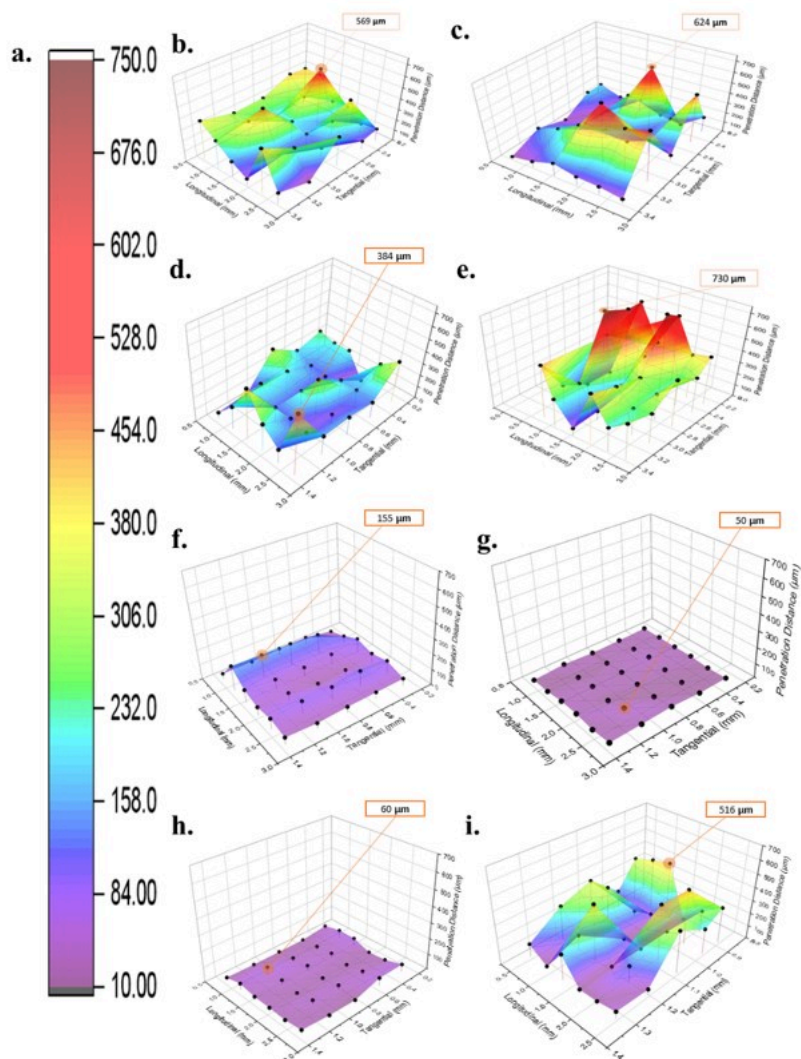


Figure 4
Three-dimensional coating penetration plot of Japanese commercial coating. (a) Color chart level of penetration depth, (b) Brown coating, (c) Pink coating, (d) Matte white coating, (e) White coating, (f) Gold coating, (g) Silver Coating, (h) Light-oak oil-based coating, (i) Light-oak water-based coating.

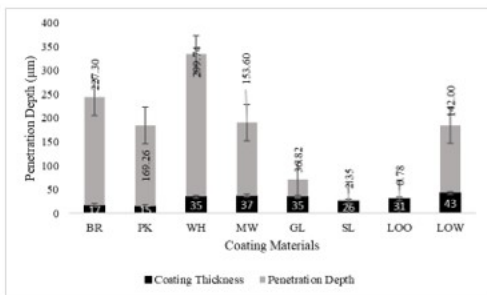


Figure 5
Average coating thickness and penetration depth

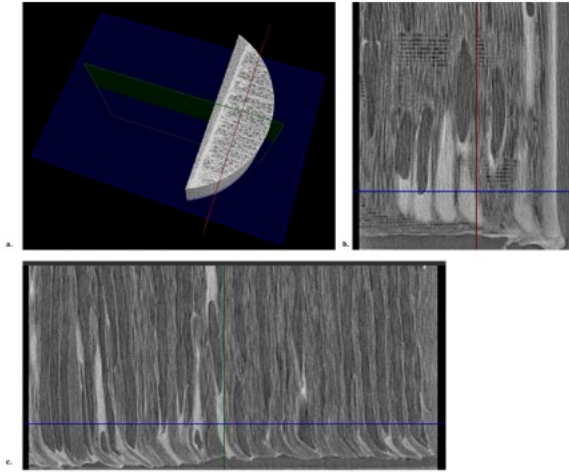


Figure 6
A 3D X-ray observation of coating penetration. (a) A 3D image stack of veneer with white coating, (b) 2D representative image of a radial section were obtained from the green cut section of the 3D image, (c) 2D representative image of a tangential section were obtained from the red cut section of the 3D image.