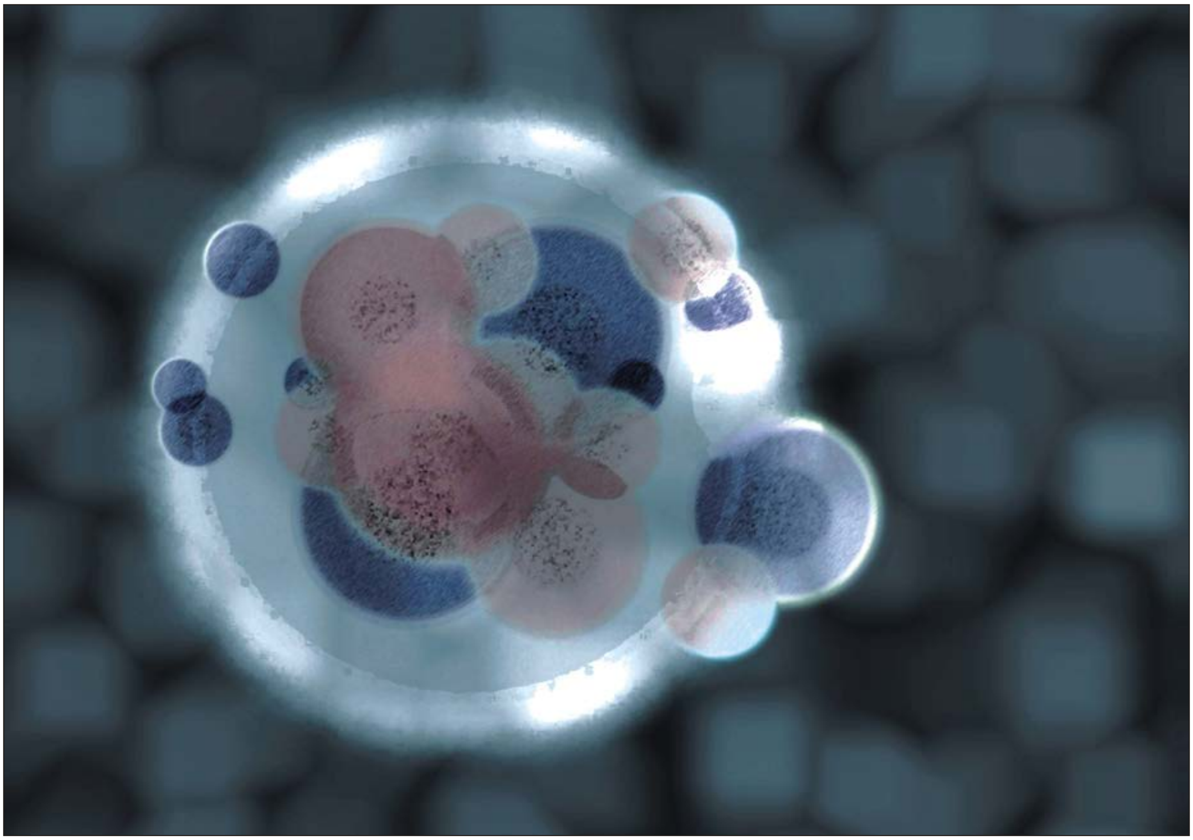

Journal of Biological Sciences

ISSN 1727-3048

OPEN ACCESS
Journal of Biological Sciences
ISSN 1727-3048
DOI: 10.3923/jbs.2018.201.207

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Research Article
Effect of Chemical Modification of Jabon Wood (*Anthocephalus cadamba* Miq.) on Morphological Structure and Dimensional Stability

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Abstract
Background and Objective: The chemical modification of wood was a chemical reaction between some reactive parts of wood components and a simple chemical reagent to form a covalent bond between both wood and chemical reagent. The utilization of jabon wood has long been done by the people of Indonesia. Jabon wood (*Anthocephalus cadamba* Miq.) is used for building materials, having quality of wood is not good such as dimensional stability. This study was to find out the effect of treatment of wood with styrene monomer using methyl methacrylamide in change in morphology structure and dimensional change. **Materials and Methods:** Jabon wood was impregnated with a styrene and methyl methacrylate to a strain of 50%. The changes in cell structure were analyzed to determine the effects of the treatment. Scanning Electron Microscopy (SEM) was used to determine the change in microfibril angle (MFA), preferred orientation of fiber (PO) and wood crystallinity (WC). Samples between the tangential sections were used for density measurement, as determined by the American Standard Testing and Material (ASTM) D2395 method. Furthermore, dimensional stability of the samples were calculated according to standard. **Results:** The wood density and volumetric swelling coefficient (SC) after modification of jabon wood showed a range of variation. The value of MFA in modified wood decreased significantly compared with the control. Wood modification results in an increase in the crystallinity. Cellulose crystallinity is reduced after impregnation, because the monomer groups interact with the groups in the volume of wood and occupy a larger space in the wood. **Conclusion:** The impregnation increased the dimensional stability of jabon wood (density and specific gravity).

Key words: Impregnation, density, dimensional stability, morphology structure, *Anthocephalus cadamba* Miq.

Received: February 19, 2018 Accepted: April 02, 2018 Published: April 15, 2018

Citation: Anne Hadiyane, Rudi Dungani, Susana Paulina Dewi and AlfiRumidatul, 2018. Effect of chemical modification of jabon wood (*Anthocephalus cadamba* Miq.) on morphological structure and dimensional stability. J. Biol. Sci., 18: 201-207.

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Competing Interest: The authors have declared that no competing interest exists.

Data Availability: All relevant data are within the paper and its supporting information files.

INTRODUCTION

The wood from plantation forests both industrial plantations (HTI) and community forests are expected to dominate the timber market in the future, following the declining productivity and quality of existing natural forest productions. This is reflected in the growing demand tends timber species is less well known that during the last 5 years. Meanwhile, the wood will require certain treatments before they are used for different characteristics when compared to natural forest wood. Wood from plantations forest is generally inferior in particular in terms of strength and dimensional stability[1].

To develop higher value applications of fast-growing trees, chemical modification of wood which leads to an increased dimensional stability have been done to improve wood quality. In chemical modification of wood, a chemical reagent will reaction with reactive part of wood or the chemical reagent with or without catalyst[2]. Many chemical agents have been used to increase dimensional stability of wood, such as dimethyl sulfate,[3] carboxylic acid,[4] acetic anhydride[5] and epoxides[6,7]. Jabon wood (*Anthocephalus cadamba* Miq.) has low dimensional stability due to its low wood density. Densifying by impregnation is chemical modification to improve its properties on jabon wood. In the impregnation process, wood cavity structure is filled with substances such as phenol formaldehyde resins, vinyl solution, liquefied natural resin, wax, sulfur and light weight metal so that the wood becomes more dense[8-11].

Several studies have been conducted on densifying wood by impregnation using the monomers styrene (ST), methyl methacrylate (MMA) and glycidyl methacrylate (GMA). This method can improve the mechanical properties, biological durability, dimensional stability, hardness and UV-stability of wood[12-15]. Furthermore, according to Abdul Khalil *et al.*[7], modifications of *Acacia mangium* carried out with propionic anhydride and succinic anhydride in the presence of the catalyst sodium formate provided resistance to microbial attack.

To prevent densified wood from recovering its original shape and size, wood can be treated with the water repellent materials[16]. Inoue *et al.*[16] found that compressed formaldehyde in Sugi (*Cryptomena japonica*) was able to produce cross linking among the wood components through the process of polymerization. Densified wood remained stable despite being exposed to steam for 1 h[16]. Compressive deformation was well fixed by this method without any special post-treatment. There were limitations, considering formaldehyde pollution causes risks from the free formaldehyde remaining in the treated products.

Chemical modification or impregnation is an effective method of permanent fixation of compressed deformation of wood. There are few studies conducted to determine the effect of the combination of chemical impregnation and compression. Therefore, the present study was conducted to investigate the effect of chemical modification on the dimensional stability and morphology structure of jabon wood.

MATERIALS AND METHODS

This study was conducted from July-October, 2017, divided into two parts, i.e. materials and methodology. In material section, it consists of material preparation, starting from trees selection to the specimen manufacturing, whilst the methodology comprises the detail procedures to conduct the experiments.

Materials: Jabon lumber was prepared from mature (5 years old) jabon trees collected from a community forest in West Java, Indonesia. Styrene, methyl methacrylamide (65/28 w/w%), benzoyl peroxide (2%) and divinyl benzene (5%) of Sigma-Aldrich (USA) specification were procured from local chemical vendor.

The jabon trees were sawn using the polygon sawing method to obtain the dense homogenous lumber of jabon wood. Wood was sawed radially at a thickness of 35 mm to obtain samples. The samples were initially dried for 24 h at 50°C in an oven to reach 15% moisture content (MC) and a specific gravity between 0.25 and 0.45 before impregnation and finally cut to samples with dimensions of 10 mm (L)$\times$20 mm (T)$\times$20 mm (R). The samples wood was then steamed at 180°C for 8 min according to the method of Inoue *et al.*[17]. The steam treatment is effective method for fixing the compressed deformation of wood for permanent fixation.

Methods

Chemical impregnation treatment: A monomer solution of styrene (ST) and methyl methacrylamide (MMA) (65/28 w/w%) was mixed with a 2% benzoyl peroxide catalyst (polymerization) and a 5% divinyl benzene cross-linker was prepared at 40% of monomer solution with 60% benzene based weight/weight. The mixture was perfectly incorporated into a chamber to begin the process of impregnation. The wood was impregnated with the monomer solution by vacuum-pressure, with an initial vacuum of 3 bars for 30 min, pressure at 7 bars for 60 min and subsequently at 3 bars for 10 min. The wood specimens were impregnated with ST/MMA to produce wood polymer composites (WPCs).

Morphological structure analysis: Scanning Electron Microscopy (SEM) (LEO Supra 50 Vp, Germany) was used to characterize the morphology of the wood samples. The samples were attached to an aluminium stub, sputter-coated with gold (Polaron SC515, Fisons Instruments; UK) and analyzed at an accelerating voltage of 5 kV. X-ray diffraction (XRD) analysis, carried out on a Philips PW 1050 X-pert (Netherlands), was used to determine the change in microfibril angle (MFA), preferred orientation of fiber (PO) and wood crystallinity (WC).

Density and dimensional stability properties: Samples between the tangential sections were used for density measurement, as determined by the American Standard Testing and Material (ASTM) D2395 (2002) method. Dimensional parameters such as volumetric swelling were calculated by a reported method[18]. The samples were submerged in distilled water evacuated in vacuum desiccators. Excess water was drained and the volume of the samples were determined. The soaking process was continued until samples attained constant volume.

The volumetric swelling coefficient (SC) was calculated by Eq. 1:

$$S_{u.t}\ (\%) = \frac{(V_w - V_{u.t})}{V_{u.t}} \times 100 \quad (1)$$

Where:

V_w = Swollen volume of wood sample after treatment (cm^3)

$V_{u,t}$ = Oven-dried volume of either treated or untreated samples (cm^3)

Statistical analysis: All the specimen in the testing used five replicates. The mean of the samples was used to show the changes in properties of the WPCs, while standard deviation was used to show the significant difference between the treatments. This was analyzed by F-test or probability analysis.

RESULTS AND DISCUSSION

Properties of modified wood: The wood density and volumetric swelling coefficient (SC) after modification of jabon wood showed a range of variation (Table 1). Compared with the untreated (control), the density of modified jabon wood each was increased by 26.87%. The impregnation treatment made a contribution to the density increase. Under normal conditions for the modified wood, a higher density causes dimensional stability.

Table 1: Density and swelling coefficient (SC) of modified jabon wood

	Control (untreated)	Modified jabon wood
Density (kg cm^{-2})	0.49 (0.04)	0.67 (0.03)
SC (%)	7.17 (0.20)	2.18 (0.03)

Data reported as the mean ± standard deviation

Table 2: Change in micro structure of modified jabon wood

Percentage	Control (untreated)	Modified jabon wood
MFA	19.67	11.47
PO	38.80	70.40
GC	49.36	37.47

The increase of density due to compression occurs because the wood cell cavities and cell walls become denser and contain low cellulose in the primary wall and middle lamella. Jabon wood impregnated with monomers to fill the void space by press-vacuum treatment led to an increase in density. Polymer-filled wood yielded higher density compared to control because co-polymerization of the cell wall in the wood produced permanent fixation by the presence of ST/MMA, as shown by Rahman *et al.*[11], reported permanent fixation of the wood matrix due to ST/MMA cross linking with the wood cell wall.

The rate of dimension change of the WPCs was within the range of 0.012-0.015% (Table 1). The rate of change in the volumetric swelling of the WPCs is very low due to the presence of monomers *in situ* copolymerization in the cell cavities. Therefore, the increase in the polymer material used increased stable dimensions of the wood[9,19].

Morphological structure: The change in morphology can be seen by comparing the SEM cross-section of the fracture surface of the control and modified jabon wood. The bulk of hardwood consists of short narrow hollow cells, or tracheids, that fit closely together (Fig. 1a). Figure 1b showed that the monomer has filled the cavity so that the cavity cells shrink even closed. It appeared that the cavities in the parenchyma tissue are full, so impregnation has occurred. The high density of the pores in the wood after impregnation with monomer showed that the mixture of monomers used has been symmetrically and achieve homogeneity resulting in improved mechanical and physical properties[20].

Changes of microstructure: The chemical modification of jabon wood had significant effects on the change in microstructure relevant to the microfibril angle (MFA), preferred orientation of fiber (PO) and gradient crystallinity (GC) as shown in Table 2.

Table 2, untreated jabon wood has a higher MFA than modified jabon wood. This suggested that the impregnation process of 50% decreased the microfibril angle to improve the properties of jabon wood. The MFA curves of modified jabon

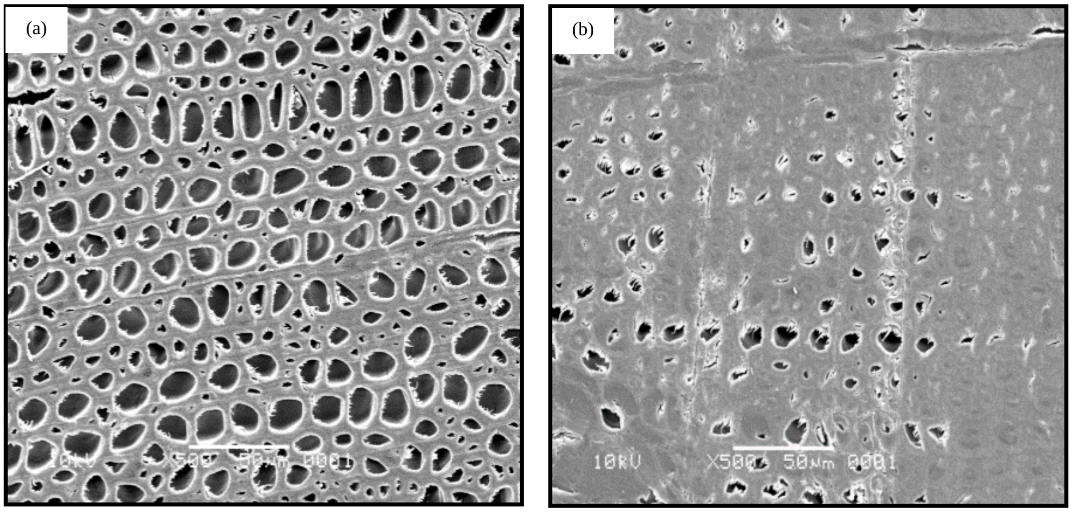

Fig. 1(a-b): SEM micrographs showing morphology changes in modified jabon wood. (a) Untreated (control) and (b) Wood polymer composite (WPCs)

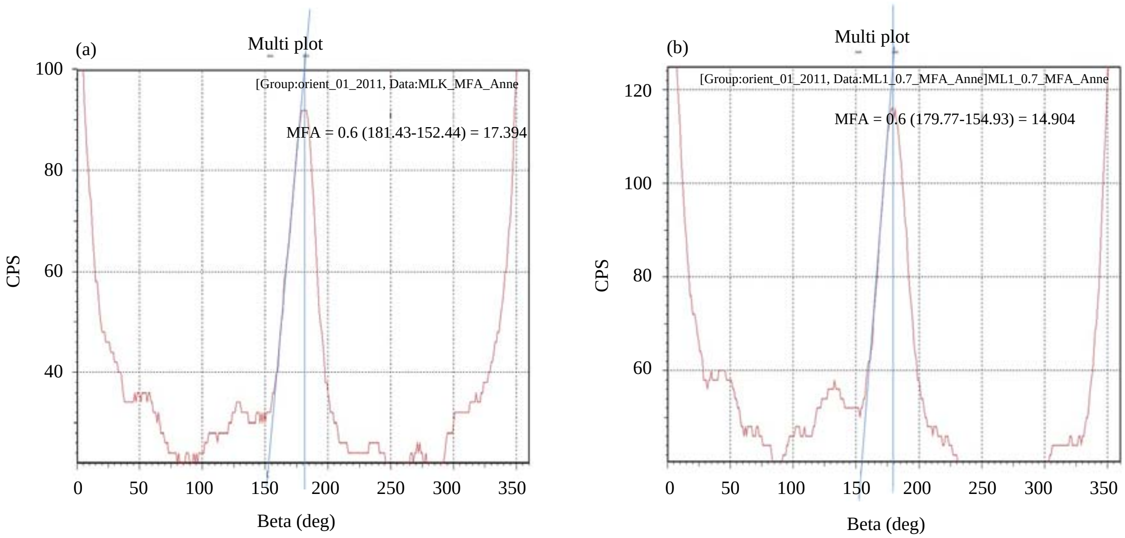

Fig. 2(a-b): MFA curves of modified jabon wood. (a) Untreated (control) and (b) Wood polymer composite (WPCs)

wood was shown in Fig. 2. The value of MFA in modified wood (Fig. 2b) decreased significantly compared with the untreated jabon wood (control) (Fig. 2a). The MFA showed a variable relationship with wood density[21] and dominant parameters that affect shrinkage and shrinkage anisotropy[22]. The dominant parameters, as defined was key importance on major effects stiffness and shrinkage, such as orientation and crystallinity of cellulose microfibrils in cell wall along the fiber axis[23]. The obtained results revealed that the increased dimensional stability and strength of treated wood with minor values of MFA of WPCs.

Impregnation causes irregular fibers. The phenomenon that occurs in wood impregnated showed that the inner layer (inner part of sample) is more regular than the outside fiber (surface of sample) (Fig. 3). Changes in the value of preferred orientation were shown in Table 2. Chemical modification is the process to change from hydrophobic to hydrophilic in the wood. There will be changes in the shape regularity structure, so flexibility will be lower because wood density increases. This phenomenon showed that cells in wood filled with chemical agents changed the preferred orientation of wood by replacement of hydroxyl groups with monomers that polymerize with wood[24].

Wood modification results in an increase in the crystallinity of modified jabon wood, as shown by X-ray diffraction (Fig. 4). The primary component of wood is

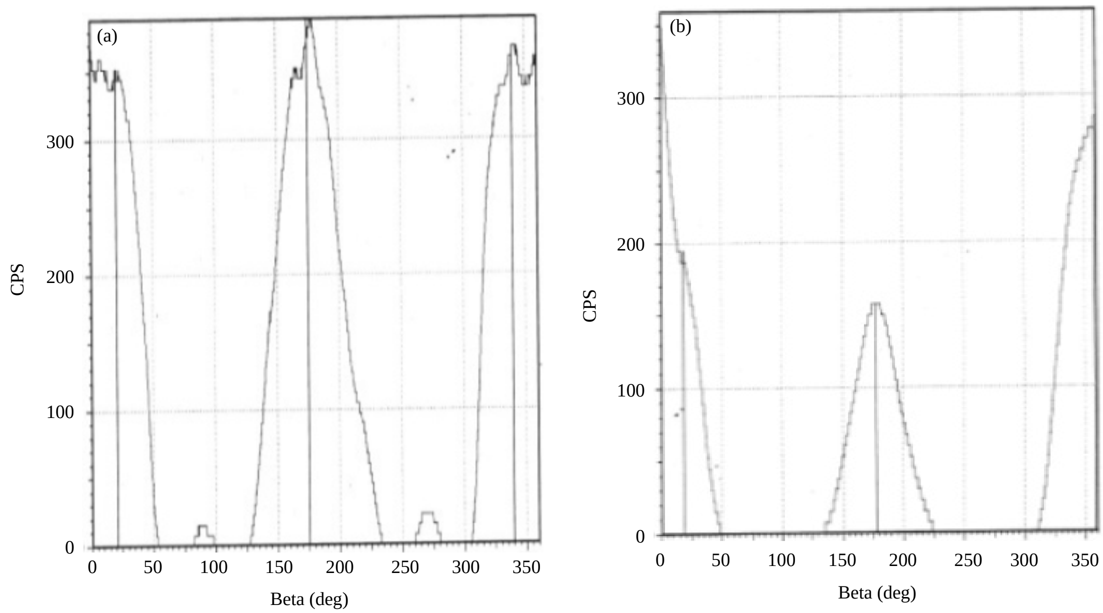

Fig. 3(a-b): PO curves of jabon modified wood. (a) Untreated control and (b) Wood polymer composite (WPC)

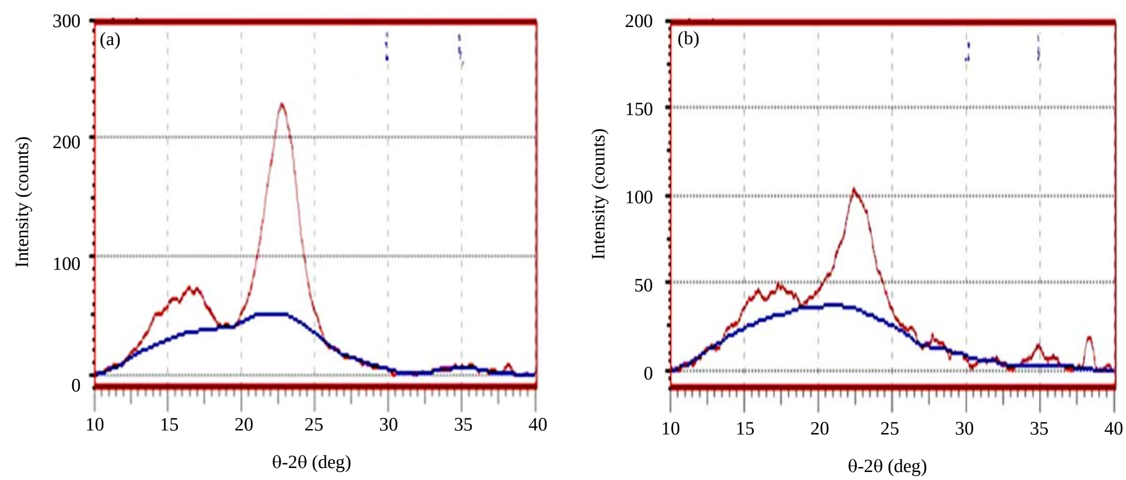

Fig. 4(a-b): GC curves of modified jabon wood: (a) Untreated control and (b) Wood polymer composite (WPC)

cellulose and generally cellulose is in a crystalline form. Cellulose crystallinity is reduced after impregnation, because the monomer groups interact with the groups in the volume of wood and occupy a larger space in the wood. This process causes a change to the arrangement of wood fibrils, to cause a reduction in crystallinity so fibrils become more rigid.

CONCLUSION

Modifications to the impregnation process successfully improved the physical properties of jabon wood. At 50% impregnation, an increase of the gradient density and a decrease in the volumetric swelling in jabon wood was observed. The impregnation also increased the dimensional stability of the treated wood (density and specific gravity). Meanwhile, process modification by impregnation resulted in a small increase in the value of microfibril angle (MFA). Densifying the wood by impregnation resulted in decrease in the crystallinity of wood and tended to result in a reduction of cell wall chemistry.

SIGNIFICANCE STATEMENT

This study discovers the properties of wood modified based on impregnation process by styrene (ST) and methyl methacrylamide (MMA) in connection with quality enhancement of jabon wood. The findings of this study can be beneficial as an alternative to wood and wood-based composites for structural purposes. Thus, a new theory on the effect of chemical modification of jabon wood (*Anthocephalus cadamba* Miq.) on the morphological structure and dimensional stability.

ACKNOWLEDGMENTS

The authors would like to thank Institute of Teknologi Bandung (ITB), for providing Research Grants of Research, Community Services and Innovation Program (P3MI)-ITB. The authors would also like to thank Forestry Products Research and Development Center, Ministry of Environment and Forestry Indonesia, Bogor, for providing the necessary facilities.

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