EFFECTIVENESS OF MICROWAVE-SOAKING ASSISTED IMPREGNATION OF TEAK WOOD (Tectona grandis Linn. f) WITH SULFUR-CONTAINING AMMONIUM SALT AS FIRE RETARDANT

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Abstract

The objective of this study is to investigate the use of microwave-soaking combination methods to improve the impregnation efficiency of Ammonium Sulfate (AS) as a fire-retardant into the teak wood. Various methods were compared by testing the (WPG). Experiments were performed for different impregnation conditions such as microwave power, radiation time and soaking time. The results showed the optimum condition for maximum WPG with microwave was to impregnate with AS solution using microwave power of 300 W, with a radiation time of 10 min, and then soaking at 75°C for 6 hours. The effect on the properties of the treated wood before and after microwave-soaking treatment was also investigated by Fourier Transform Infrared Spectroscopy and compression strength parallel to the grain. Furthermore, this work evaluated the effect of AS contents having a concentration of 0-25 wt.% impregnated into teak on flammability and leachability. Flammability was tested using the Limiting Oxygen Index (LOI) and, leachability was carried out according to JIS K 1571. The test indicated that the LOI values increased from 21.24% for untreated wood to 38.5 for 25 wt.% AS as a self-extinguishable wood, and the leaching of AS from wood was in the range 1-2.5%, indicating the good fixation of AS in teak.

Keywords: Ammonium sulfate, Compression strength, Fire retardancy, Leachability, Limiting oxygen index, Teak wood.

1. Introduction

Teak (Tectona grandis Linn. f.) has a reputation of being the "king of timbers" due to its beautiful and uniform colour (Golden yellow, brown coloured heartwood), good machining properties, strength, high natural durability, pleasant appearance, and weather resistant [1, 2]. These features contribute to their high performance in various applications such as furniture, construction, floor finishes, shipbuilding, etc. [2]. In modern society, teak furniture or indoor decorative materials shows elegant values on classic furniture and a high-value timber [3]. It is necessary, therefore, to test the timber according to a variety of uses to find the most durable form. One of the limitations of teak is that it is a flammable material. It can, therefore, pose a serious fire risk under the right conditions, such as when it is hot enough [4]. Many studies have been carried out to investigate ways to reduce the ignitability, heat release and the spread of flames [5, 6]. Substances have been developed to meet the safety requirement as fire-retardants for wood, but none has reported on products for teak wood.

Fire-Retardant Chemicals (FR) loaded into the wood structure can modify the reaction mechanism and the pyrolysis product during burning [7, 8]. This reaction is able to delay or even extinguish combustion [9]. The effect in FR treated wood will depend on the type of chemicals, the add-on of the chemicals, the species of the wood and the impregnation processes [6, 7]. Ammonium Sulfate is one of the most effective fire retardants and a well-known component of fertilizer that is non-toxic and inexpensive. Researchers have previously found that AS has a good effect as FR for cotton fabrics [10], fir wood [5], oak [11], sugar-cane bagasse, cellulose, pinus halepensis needles and their main components (cellulose, lignin and extractives) [12]. In all instances, AS are applied to delay ignition and flame spread as well as to lower the rate of heat release.

The effectiveness of chemical impregnation depends on the chemical formulation, the concentration of the chemical, method of impregnation, kinds of wood species, the moisture content of the wood, amount of chemical retention and distribution [6-8]. A method whereby a substance could be impregnated into a wood structure is the main point of this study. Much of the literature also involves improvement of impregnation of chemicals in wood such as soaking, vacuum, pressure, steaming, microwave and plasma [5, 12-14]. The conventional method involves vacuum and pressure impregnation such as Bethel process, Lowery process and Rueping process [6, 15]. This method causes a deep penetration of chemicals, but at high capital costs and a long treatment time. Another technique is soaking at atmosphere or at high temperature that results not deep impregnation [16]. To solve these problems, the microwave method has also become of interest as an alternative approach for the conventional methods.

Microwaves (MW) are a series of waves that consist of electromagnetic energy (frequency band between 300 MHz and 300 GHz and wavelengths ranging from 1.0 meters to 0.1 centimetres) [12]. MW interact with polar molecules and ions in a material to obtain quickly high temperature and then generates a steam pressure of substances within structure increasing the solubility of the FR and rupture the structures of thin-walled cells and pit membranes to form pathways for the easy transport of liquids and vapours [12]. Its advantage is good permeability, fast heating, energy saving, ease of control, compactness, and good uniform heating. The study of Poonia et al. [17] reveals that the

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preservative retention was 5.11 kg m⁻³ of Eucalyptus tereticornis wood treated with Acid Copper Chrome by using a 2.45 GHz frequency microwave with MW heating for 5 min and MW intensity for 143.35 W/cm². Domeny et al. [18] confirmed microwave has an effect on the fluid permeability of beech false heartwood. The permeability was increased as a function of the MW time consumption applied and the WPG increased to 33.84% at 20 seconds intervals (20 seconds treatment, 30 seconds relaxation and 20 seconds treatment). Vinden et al. [19] studied the effect of MW process parameters (preservative, sleeper weight, sleeper density, MW energy, and MW power) of Pinus radiata railway sleepers for preservative treatment on preservative distribution and uptake in sleepers and found that dramatic increase in wood permeability and solution uptake after MW modification. Hong-Hai et al. [20] reported that the larch wood was treated by water by microwave irradiation under different radiant intensity (500-900 kW) and treating duration (20-50 seconds). The CS values of microwave-treated specimens decreased slightly compared to the untreated specimen but not remarkably. In this study introduces a new method as a combination of microwave and soaking method to enhance fixation efficiency by increasing the WPG and by shorting the time of impregnation of FR into the wood. It has also come of interest as an alternative to conventional methods.

The main objective in the first part of this study was to investigate the effect of the AS impregnation methods (microwave/soaking treatment and with/without soaking temperature into the sample) on WPG. Secondly, the effect of parameters of microwave combined soaking methods, such as microwave power and radiation time, and the effect of soaking time of AS was determined on the WPG, FTIR, and compression strength. Lastly, we specifically determined the effect of AS with various concentrations on the flammability properties (LOI) and the leachability of AS. This study, possibly, provides a useful understanding on a potential scale-up from a laboratory experiment to an industrial technique.

2. Materials and Methods

2.1. Materials

The teak lumber was obtained for this experiment from commercially produced flat sawn lumber supplied by a local sawmill in Phitsanulok province, Thailand. Samples were carefully chosen for having the same annual ring. The teak timbers were kiln dried at 60°C and thereafter conditioned to stable moisture content and then it was machined into samples with the dimensions of $100 \times 10 \times 7 \text{ mm}^3$ and $8 \times 2 \times 2 \text{ cm}^3$ that were selected by the criteria of similar weight (±0.001 g) and average density 743 kg/m³. Before impregnation, the teaks were dried in an oven at 105°C for 24 hours, then keep into desiccator for two weeks, and weighed. Ammonium Sulfate ((NH₄)₂SO₄, >98% purity) was purchased from Ajax Finechem, Australia. AS solutions were diluted in distilled water before use at 2.5%, 5%, 10%, 15% and 25% concentrations, respectively [5].

2.2. Impregnation of teak samples

For the experiment, a commercial microwave MS20220LG (Microwave, Thailand), was basically a rectangular container ($455 \times 330 \times 260 \text{ mm}^3$). The frequency microwave and power outputs were 2450 MHz and 700 W [21]. The impregnation of teak was performed using the microwave method followed by the

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soaking method, respectively with the volume ratio of the specimen to aqueous solutions at 1:10. The wood in solution was microwaved for 2 minutes, then taken out and allowed to soak on heat at 30°C (room temperature) and 75°C for 2 hours. Afterwards, the wood sample was removed from the chamber and shaken off lightly to remove excess solution from the surface of the wood. After the treatment, all the teak were dried until the weight stabilised at 60°C. The AS impregnation of the teak were divided into seven groups to study the effect of impregnation methods shown in Fig. 1. It was found through the use of the Conductivity Meter, that AS does not decompose when exposed to microwaves. All the experiments of each condition in this study were performed using 10 samples.



Fig. 1. Schematic diagram of impregnation methods applied to teak.

The efficiency of FR-fixation of wood is measured by WPG that this value has to be considered to be a fixed solid (FR) in the wood matrix with monitoring the weight before and after the treatment process. WPG of each condition was measured according to Eq. (1) as previously mentioned in other studies [22]:

$$WPG\% = (W_2 - W_1) / W_1 \times 100 \tag{1}$$

where W_1 is the oven-dried weight of the wood sample before impregnation (g) and W_2 is the oven-dried weight of the wood sample after impregnation (g).

2.3. FTIR analyses

Fourier Transform Infrared Spectroscopy (FITR) spectroscopy was performed in Attenuated Total Reflectance (ATR) mode using a Spectrum GX FTIR spectrometer (Perkin Elmer Instruments GmbH, Überlingen, GER) within a wavenumber range from 400 cm⁻¹ to 4000 cm⁻¹ at a spectral resolution of 4 cm⁻¹ (64 scans) using a diamond crystal (GladiATR Vision, Pike Technologies, Madison, USA).

2.4. Compression strength test

The CS value is the maximum crushing strength in compression parallel to grain and is the frequently used method for research purposes. CS was conducted using the Tecnotest Macchina Tipo KL200. Samples had dimensions of 8 (axial) $\times 2 \times 2$ cm³ and the load was applied in the axial direction. Samples were conditioned for 2 weeks at 20 $\pm 2^{\circ}$ C and 65 $\pm 5\%$ relative humidity before testing [23].

2.5. Leaching of treated wood specimens

The Procedure Leaching Test was conducted according to (JIS) K 1571 (JIS, 2004) and Chinese National Standard 6717 (2000) [24]. The procedure involves immersing the five specimens per run in a 1000 ml beaker containing 400 ml distilled water added in a ratio of 10 volumes of water to 1 volume of wood, stirring with a shaker follower (400-450 rpm) at 27°C for 8 hours, followed by drying for 16 hours and then teak was stored in a desiccator. The percentages of leaching the AS impregnated samples (%L) according to the following Eq. (2):

$$\% L = (W_2 - W_3) / W_2 \times 100 \tag{2}$$

where W_2 is the oven-dried weight of teak after impregnation (before leaching) (g) and W_3 is the oven-dried weight of impregnated teak after leaching (g).

2.6. Limited oxygen index (LOI) measurements

The LOI value was defined fire performance of wood samples as the minimum percentage of oxygen for retaining flaming combustion of each sample under test conditions. LOI measurements of samples were carried out by using a Stanton Red Croft instrument, which is a vertical burning test. It was applied for evaluation of LOI in accordance with ASTM D2863-10 [25]. The sample is placed with the holder in a gas mixture of oxygen/nitrogen and adjusting the amount of O_2 until flaming combustion of the sample for 5.08 centimetres (2 inches) or 3 minutes after the removal ignition source. The five replicate measurements were observed for each value, with R.S.D. lower than 1%.

3. Results and Discussion

3.1. Comparison of impregnation methods of AS into teak wood

From the data in Fig. 2, Comparing WPG between groups treated without a microwave (I, II) and with microwave (III-VII) found that WPG of groups III-VII more than I, II. This indicates that the impregnated AS depends on the method and the temperature, which was the MAI and the soaking temperature, significantly affect the WPG of wood samples. In the case of using microwave irradiation without solution before AS soaking (groups III and IV) had WPG of samples more than groups I and II. It is reasonable to suppose that the microwave energy adsorption in the wood may also cause a changing of the matrix from the centre to the surface layer or gradient shrinkage between regions during the impregnation processes, with the result, the fluid can diffuse into the wood [26]. Especially, the substantial difference in uptake caused by wood immersed in solution under microwave processes was higher for V, VI and VII than for III and IV. The reason for the high permeability may be due to microwave energy heated solution generating steam pressure from within the bulk, which creates the micro-cracks of

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wood cell walls, pit membranes, ray cells and the partial rupture of tyloses in vessels to produce more and more pathways, leading to for an increased mass transfer of substances between boundaries [27]. Microscopic studies by He et al. [28] revealed the tyloses in vessel and pit membranes of the Eucalyptus were ruptured after the MV treatment is the factors for the increased permeability. Ramezanpour et al. [21] pointed out that the effect of MW on preservative penetration can also be attributed to a radial wood permeability improvement.



Fig. 2. Comparison of impregnation methods of AS into teak wood on WPG.

Furthermore, compared to treatment by soaking after microwave (V, VI, VII), soaking has a major effect on the impregnation of AS in wood and WPG of VII less than V and VI. This indicated that soaking increases the time duration of mass transfer of AS and the solubilization of AS to enter the wood structure. The result here presented a clear advantage of MAI in enhancing the permeability of AS. The main parameters play an important role on AS impregnation of teak. Lastly, the combined action of microwave and soaking into solution at a high temperature in VI proved previous conclusions. Microwave combined with soaking could greatly increase the WPG, which was the highest WPG at 3.078% (the retention was 5.772 kg m⁻³). It was improved by almost 12 times compared to that for the soaked samples.

3.2. Effect of microwave power and radiation time

Figure 3 shows the impregnation profiles for the WPG of 2.5 and 25% AS impregnation obtained using Microwave-Assisted Impregnation combined soaking (75°C, 2 hours) with various radiation times and microwave power of microwave.

Figure 3 demonstrates the increase of WPG of the impregnation during microwave treatments of teak woods. The WPG increased with microwave power, radiation time, and AS solution concentration. The effect of AS concentration levels into the teak wood on WPG. As expected, the WPG in wood samples treated with AS increases with the increment of AS concentration levels from 2.5 to 25%.

It can be noted that increasing the concentration of the AS increases significantly the impregnation of wood.





When compared with the microwave powers, the increment of WPG depending on the radiation powers was observed. The WPG reached a relatively high value (7.78%) when the treating power increased to 700 W for 10 minutes of 25%AS. This is because of the intensity of the vibrations of the molecules inside the wooden structure of higher wattages [29]. The high amount of microwave energy absorbed generated AS steam within the wood and damaged the cell structure to form microholes, increasing pathways for transportation of fluid [20]. In this paper, it was found that a slight difference in the value of the WPG for samples MW-radiated at 300 W and 700 W for 10 minutes was observed. It can also be stated that both microwave powers have influence closely on the movement capability of fluid as AS solution.

In order to directly compare the radiation time ranged from 2 to 10 minutes, the WPG increased with the same microwave power. It was observed indeed that at a high microwave power level with increasing radiation time the WPG profile of the specimens continuously increases more than that in the case of low microwave power level. Especially, there was a sharp increase in WPG for specimens with power at 300 W, from 3.25 to 4.84 (49%) of 2.5% AS and 5.84 to 7.42 (27%) of 25% AS whereas, at 700 W the WPG increased slightly nearly 14.32% (2.5% AS) and 25.44% (25% AS) with increase in MW treatment time. This result indicated that the duration of radiation causes a positive effect on a fluid add-on to enhance the diffusion and fixation of AS within the lumen cells/voids. The observed data cannot be compared with literature due to the research articles has not been reported before dealing with the add-on of fire retardant into the teak wood. The previous works by Ramezanpour et al. [21] found that the microwave radiation in pre-treatment of fir wood (Abies alba L.) at a frequency of 2.45 GHz from 10 to 16 minutes could increase the retention of acid copper chromate.

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3.3. Effect of soaking time

The effect of 2.5% AS impregnation using MAI (300 W, 10 minutes) combined after soaking at various soaking times with a sample was taken and checked every 15 minutes. The sample was removed from the solution, dried and examined. Figure 4 shows that the results of the longer the soaking time increased the impregnation rate only in the first 6 hours and WPG up to 4.5-6.0, thereafter, the rate of impregnation and WPG flattens out, leading to no further gains. Increasing the soaking time from 15 minutes to 1.15 hours results in dramatic increases in the WPG efficiency due to the capacity of the wood pores, which have spare space allowing AS to be adsorbed fully and quickly, thereafter, the capacity diminishes until no further absorption is possible.



Fig. 4. Effect of soaking time at 75°C on WPG of teak wood impregnated with 2.5% AS; microwave power, 300 W; and radiation time, 10 minutes.

3.4. Fourier Transform Infrared Spectroscopy analysis

The FTIR spectra of untreated and treated teak with AS solution containing 2.5% and 25% concentration with microwave power levels varying from 150 to 700 W for 10 minutes, and then fixed soaking condition at 75°C for 6 hours exposure are represented in Fig. 5. In the untreated teak wood, the dominant peaks of the *O*-*H* (3334 cm⁻¹), *C*-*H* (2800-2990, 1368 cm⁻¹), *C*=*O* (1734 cm⁻¹), *C*=*C* (1595, 1507 cm⁻¹), *C*-*O* (1240 cm⁻¹) and *C*-*O*-*C* (1024 cm⁻¹) are observed in overall spectra, which indicate mainly derivative components of natural polymer (wood) such as cellulose, hemicelluloses, and lignin [30, 31]. Similar results have been reported previously [32, 33].

After impregnation with AS (2.5% and 25%) at power 300 W, it can clearly be observed that there is a significant difference between the curves treated and untreated wood. The FTIR spectrum of wood treated with sulphur fire retardant shows the new peak at 1120 cm⁻¹, attributed to the stretching vibration of *S*-*O* [34, 35]. It can prove that sulphur fire retardant is present in the wood. Their intensity increases with increasing the AS concentrations. The decrease in intensity of the bands at 896-895 cm⁻¹ shows the changes in *C*-*H* out-of-plane distortion in cellulose and hemicelluloses [36]. The band at 1024 cm⁻¹, which was assigned to the *C*-*O* stretching vibration of *Ph-O-C* coupled with aromatic ring vibration in lignin, *C*-*O* stretching vibration in

xyloglucan [37], gradually decreased as AS impregnated. The decrease in the *C-O* stretch with exposure AS concentration resulted in a decrease in the vibration of this band. The peaks at 1595 and 1507 cm⁻¹ peaks are associated with the aromatic skeletal stretch of lignin [38], which was reduced on AS treatment.



Fig. 5. FT-IR spectra of untreated and microwave-treated teak wood.

A significant reduction was observed the peak located at 1730-1740 cm⁻¹, corresponding to C=O stretching vibrations of acetyl, carbonyl and carboxyl groups 38]. This change suggests that the cleavage of acetyl side chains from hemicelluloses after impregnation. These results indicate that the addition of AS enhances the hemicelluloses degradation. Interestingly, the bands at around 2900 cm⁻¹ *C-H* stretching peaks) was enhanced and divided into two peaks at 2920 cm⁻¹ (CH₂ asymmetrical) and 2850 cm⁻¹ (symmetrical stretching) [39]. Their intensity increased with increasing concentration of AS. This may be due to a change in lignin or cellulose of sp^3 and sp^2 hybridized *C-H* stretch by AS solution. A similar finding was reported by Cheng et al. [40].

The peak of 3000-3500 cm⁻¹, which, corresponds to the stretching vibration of O-H of treated wood is different than that of untreated wood and decreases with higher concentrations of AS [30]. A possible explanation might be that the sulphur fire retardant may bond with wood cellulose by hydrogen bonds. This result is in agreement with our previous findings. Other peaks, i.e., 1315, 1368, 1420 and 1460 cm⁻¹ peaks [38], were decreased slightly after treatment. There might have occurred the degradation of lignin and hemicellulose.

The effect of MW irradiation power on FTIR spectra of 25%AS-treated wood are shown in Fig. 5. With the increase in the MW irradiation power from 150 W to 700 W, the transmittance of the *O-H*, *C-H*, *C=O*, *C-O-C*, *S-O* and *C-O* slightly decline, however, the changes are not significant [33, 37]. These observations can be attributed to the physical and chemical effects of microwave treatment. Especially, the intensity of the *C=O* stretching vibrations of carboxyl groups at

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1730 cm⁻¹ showed a decrease of 4% at 700 W compared at 150 W. This result indicates that hemicellulose component was more effective by the microwave treatment. The intensity of both 2920 cm⁻¹ (CH₂ asymmetrical) and 2850 cm⁻¹ (symmetrical stretching) display a slight increased with increasing microwave powers. However, these results indicate that the cellulose molecular was changed a little and amount of hemicellulose still exist in teak wood structures in the case of various microwave treatment powers. Zhu et al. [41] pointed out that the thermochemical pretreatment for Miscanthus biomass by using the microwave with H₂SO₄ (0.2 kmol/m³) had little influence on lignin and more effect on hemicellulose, the degradation might occur in partial components with increasing the H₂SO₄ concentrations. The changes in functional groups affected the mechanical property of wood, as will be discussed in detail in the following sections.

3.5. Compressive strength analysis

In order to understand better the effect of microwave on the strength properties, the compression strength parallels to grain was also performed. Figure 6 shows the CS profile of untreated and AS-treated wood (2.5 and 25%AS) with respect to microwave irradiation times for different microwave power levels with microwave and fixed soaking condition at 75°C for 6 hours.

According to Fig. 6, The CS value in average obtained for untreated teak (59 MPa) and AS-treated teak in all cases (>50 MPa). Generally, the CS values of AS-treated specimens were reduced slightly compared to the untreated specimens. This value is within the range reported in the literature (44.4-65.9 N/mm²), which is classified as being a hardwood [42]. This may be due to the impregnation effect of AS, which causes degradation of the main substance of wood polymers, the cumulative effect resulting in pit damage and microchannels. Nurmi et al. [43] reported the fire-retardant treatment decreases the mechanical strength.





As can be seen in the Fig. 6, CS values of all samples after treatment decreased as a function of increasing power level, radiation time and chemical concentration. As expected, the compressive strength values decreased with increasing of flame

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retardant concentration, which was contrasting to the tendency of WPG. It was obvious that the high content of flame retardant in the wood specimens, the lower CS values of treated specimens. The highest decrease in CS, 15.44%, as compared to control specimens, was determined to occur at 700 W for 10 minutes of 25% AS. The percentages of CS decreases for both concentrations were found to be in the range of 2.72-7.19% (at 150 W), 4.23-8.81% (at 300 W), and 9.90-15.44% (at 700 W) for 2 to 10 minutes, when compared with the control specimens. This data indicates that a high microwave power level (700 W) the CS profile of the specimens continuously decreases sharply more than that low microwave power levels. These results are consistent with those reported by Hong-Hai et al. (2005) [20]. The reason might due to the amount of the energy applied to samples during the process, which can cause changes in the chemical structure of the wood. This is involved in the degradation of hemicelluloses is less resistant to the vibratory energy into heating than cellulose and lignin, which cause the reduction in mechanical properties [44].

Concerning low microwave intensity, there seemed to be no significant difference between CS of 150 W and of 300 W. This indicates that the microwave radiation may provide the tiny cracks on the cell walls inside the wood and did not connect each other to form macroscopic cracks, which have great influence on the mechanical properties of the wood [20]. In the other literature, Šefc et al. [23] reported that the CS had a little effect with a decrease of 5% for fir wood and the consistency for beech of microwave treatment by citric acid with 750 W power. Hermoso and Vega [45] determined the different strength properties (CS) of dried Eucalyptus globulus wood reduced ranging from 72.4-47.3 MPa due to microwave treatment (290-430 MJ m⁻³) before the conventional process (vacuum conditions of 30 kPa and pressure of 900 kPa for 120 minutes) with TANALITH E solution.

3.6. Flame retardancy and leachability analysis

The WPG for each concentration of wood impregnation by microwave (300 W, 10 minutes) combined and soaking at 75°C for 6 hours are reported in Fig. 7. The WPG in wood samples increases linearly with the increment of AS concentration. The highest increment of WPG depending on the concentrations was observed on AS treated wood samples as follows: the WPG was founded 7.422 at 25 wt% of AS solution. This indicates that the impregnated AS was fixed in the wood matrix because of the pressure differences between the wood structures and AS solution. Thus, the high concentration obtains the adsorption into the cell lumen and cell walls of wood increased, and as a result, WPG increased [42].

Figure 7 shows the percentages of leaching the AS impregnated samples (%L) along with standard deviations. %L of all treated samples after the leaching test was found in the range 0.1-2.2%, which means there is a small mass loss inside the wood sample during the test due to the AS flame retardants being a water-soluble salt. The amount of leaching increased slightly in treated samples as the loading of AS fire-retardants inside wood increased likely as results of better AS fixation, when the wood was treated with higher concentrations of fire-retardants. A possible explanation might be that the mass loss of treated wood could be related to the removal of excess AS fire-retardants or temporary fixed AS fire-retardant [24]. However, it can be observed in Fig. 7 that most of the AS were able to be fixed into the wood structure, which confirmed that AS was an effective flame retardant for wood samples under humid conditions.

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Fig. 7. WPG and leachability of non-impregnated and impregnated teak wood with AS.

The fire performance of AS-impregnated wood samples was evaluated based on LOI measurements. The results are presented in Fig. 8, the LOI value of untreated teak specimens was found to be 21.24% that can be categorized as slow burning in air with the classification of material according to ISO 4589 [46]. These LOI values tended to be raised with increasing WPG of AS in the sample, and all treated samples showed higher LOI levels than controls [47]. It was indicated that the higher the LOI value could increase the flame retardancy of the impregnated woods. The WPG of fire-retardant wood can reach 5.219%, while the LOI value could be up to 31.55%. Especially, all treated teak samples are classified as self-extinguishing (LOI >28) that means the wood samples will stop combustion after the removal of the ignition source [48]. The previous publication by Qu et al. [49] claimed the effect of fire retardants and concentration levels on various fire characteristics of wood.



Fig. 8. LOI of non-impregnated and impregnated teak wood with AS.

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4. Conclusions

The AS impregnation under soaking after being treated by microwave gives the highest WPGs while requiring the shortest impregnation times comparing with the other methods. The maximum WPG of the teak impregnated with 2.5%AS into the wood structure was 4.43%, that occurred while being treated with microwaves (300 W, 10 minutes) and soaking at 75°C for 6 hours. In addition, the highest increment of WPG depending on the concentrations was observed on AS treated wood samples as follows the WPG was founded 7.422 at 25 wt.% of AS solution. All treated teak samples with AS (5-25%) are classified as self-extinguishing (LOI >28), meaning the wood samples will stop combustion after the removal if the ignition source. The leaching of the AS from wood was in the range 1-2.5%, showing that the AS is firmly fixed in the wood structure. These results demonstrate the potential for alternative methods to impregnate fire retardant into the teak wood.

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Abbreviations	
AS	Ammonium Sulfate
CS	Compression Strength Parallel to Grain
FR	Fire-Retardant Chemicals
FTIR	Fourier Transform Infrared Spectroscopy
L	Leachability, %
LOI	Limiting Oxygen Index
MAI	Microwave-Assisted Impregnation
MW	Microwave
RPM	Revolutions Per Minute
RSD.	Relative Standard Deviation
WPG	Weight Percent Gain

References

- Kaewkrom, P.; Gajaseni, J.; Jordan, C.F.; and Gajaseni, N. (2005). Floristic 1. regeneration in five types of teak plantations in Thailand. Forest Ecology and Management, 210, 351-361.
- 2. Dungani, R.; Bhat, I; Khalil, H. A.; Naif, A.; and Hermawan, D. (2012). Evaluation of antitermitic activity of different extracts obtained from Indonesian teakwood (Tectona grandis Lf). BioResources, 7(2), 1452-1461.
- Kongprasert, N. (2012). Emotional design approach to design teak wood 3. furniture emotional design approach to design teak wood furniture. Proceedings of the Asia Pacific Industrial Engineering and Management Systems Conference. Phuket, Thailand, 805-812.
- 4. Lee, H.-L.; Chen, G.C.; and Rowell, R.M. (2004). Thermal properties of wood reacted with a phosphorus pentoxide-amine system. Journal of Applied Polymer Science, 91, 2465-2481.

Journal of Engineering Science and Technology

- 5. Di Blasi, C.; Branca, C.; and Galgano, A. (2008). Thermal and catalytic decomposition of wood impregnated with sulphur and phosphorus containing ammonium salts. *Polymer Degradation and Stability*, 93(2), 335-346.
- Lahtela, V.; Hämäläinen, K.; and Kärki, T. (2013). The effects of preservatives on the properties of wood after modification (Review paper). *Baltic Forestry*, 20(1), 189-203.
- 7. Lowden, L.A.; and Hull, T.R. (2013). Flammability behaviour of wood and a review of the methods for its reduction. *Fire Science Reviews*, 2:4, 19 pages.
- 8. Tomak, E.D.; Baysal, E.; and Peker, H. (2012). The effect of some wood preservatives on the thermal degradation of Scots pine. *Thermochimica Acta*, 547, 76-82.
- Fernandes Jr., V.J.; Fernandes, N.S.; Fonseca, V.M.; Araujo, A.S.; and Silva, D.R. (2002). Kinetic evaluation of decabromodiphenil oxide as a flame retardant for unsaturated polyester. *Thermochimica Acta*, 388(1-2), 283-288.
- 10. Mostashari, S.M.; and Mostashari, S.Z. (2008). Combustion pathway of cotton fabrics treated by ammonium sulfate as a flame-retardant studied by TG. *Journal of Thermal Analysis and Calorimetry*, 91(2), 437-441.
- 11. Kol, H.S.; Uysal, B.; Kurt, Ş.; and Ozcan, C. (2010). Thermal conductivity of oak impregnated with some chemicals and finished. *BioResources*, 5(2), 545-555.
- 12. Athina, P.; Mikedi, K.; Tzamtzis, N.; and Statheropoulos, M. (2006). TG-MS analysis for studying the effects of fire retardants on the pyrolysis of pine-needles and their components. *Journal of Thermal Analysis and Calorimetry*, 84(3), 655-661.
- 13. Caldeira, F. (2010). Boron in wood preservation a review in its physicochemical aspects. *Silva Lusitana*, 18(2), 179-196.
- 14. Pabeliña, K.G.; Lumban, C.O.; and Ramos, H.J. (2012). Plasma impregnation of wood with fire retardants. *Nuclear Instruments and Methods in Physics Research B*, 272, 365-369.
- 15. Salamah, S.; and Mohd. Dahlan, J. (2008). Vacuum-pressure treatment of rubberwood (hevea brasiliensis) using boron-based preservative. *Journal of Tropical Forest Science*, 20(1), 1-7.
- 16. Harada, T.; Uesugi, S.; and Taniuchi, H. (2003). Evaluation of fire-retardant wood treated with poly-phosphatic carbamate using a cone calorimeter. *Forest Products Journal*, 53(6), 81-85.
- Poonia, P.K.; Hom, S.K.; Sihag, K.; and Tripathi, S. (2016). Effect of microwave treatment on longitudinal air permeability and preservative uptake characteristics of chir pine wood. *Maderas Ciencia Y Tecnología*, 18(1), 125-132.
- Dömény, J.; Koiš, V.; and Dejmal, A. (2013). Microwave radiation effect on axial fluid permeability in false heartwood of beech (Fagus sylvatica L.). *BioResources*, 9(1), 372-380.
- 19. Vinden, P.; Torgovnikov, G.; and Hann, J. (2011). Microwave modification of radiata pine railway sleepers for preservative treatment. *European Journal of Wood and Wood Products*, 69(2), 271-279.

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- Hong-Hai, L.; Qing-Wen, W.; Lin, Y.; Tao, J.; and Ying-Chun, C. (2005). Modification of larch wood by intensive microwave irradiation. *Journal of Forestry Research*, 16(3), 237-240.
- Ramezanpour, M.; Tarmian, A.; and Taghiyari, H.R. (2015). Improving impregnation properties of fir wood to acid copper chromate (ACC) with microwave pre-treatment. *iForest-Biogeosciences and Forestry*, 8, 89-94.
- Mattos, B.D.; Serrano, L.; Gatto, D.A.; Magalhães, W.L.E.; and Labidi, J. (2014). Thermochemical and hygroscopicity properties of pinewood treated by in situ copolymerisation with methacrylate monomers. *Thermochimica Acta*, 596, 70-78.
- 23. Šefc, B.; Trajković, J.; Sinković, T.; Hasan, M.; and Ištok, I. (2012). Compression strength of fir and beech wood modified by citric acid. *Drvna Industrija*, 63(1), 45-50.
- Kartal, S.N.; Yoshimura, T.; and Imamura, Y. (2009). Modification of wood with Si compounds to limit boron leaching from treated wood and to increase termite and decay resistance. *International Biodeterioration and Biodegradation*, 63(2), 187-190.
- 25. ASTM D2863-10 (2000). Standard test method for measuring the minimum oxygen concentration to support candle-like combustion of plastics (oxygen index). *ASTM International*, West Conshohocken, USA.
- 26. Miura, M.; Kaga, H.; Sakurai, A.; Kakuchi, T.; and Takahashi, K. (2004). Rapid pyrolysis of wood block by microwave heating. *Journal of Analytical and Applied Pyrolysis*, 71(1), 187-199.
- 27. Torgovnikov, G.; and Vinden, P. (2009). High-intensity microwave wood modification for increasing permeability. *Forest Products Journal*, 59(4), 84-92.
- He, X.; Xiong, X.; Xie, J.; Li, Y.; Wei, Y.; Quan, P.; and Li, X. (2017). Effect of microwave pretreatment on permeability and drying properties of wood. *BioResources*, 12(2), 3850-3863.
- 29. Younis, A.A. (2016). Evaluation of the flammability and thermal properties of a new flame retardant coating applied on polyester fabric. *Egyptian Journal of Petroleum*, 25(2), 161-169.
- 30. Rowell, R.M. (2012). *Handbook of wood chemistry and wood composites* (2nd *ed.*). Boca Raton, Florida: CRC Press.
- Agarwal, U.P. (1999). An overview of Raman spectroscopy as applied to lignocellulosic materials. *Advances in Lignocellulosics Characterization*, Chapter 9, 201-225.
- Méndez-Mejías, L.D.; and Rogue, R.M. (2016). Effects on density, shrinking, colour changing and chemical surface analysis through FTIR of Tectona grandis thermo-treated: Density, shrinking, colour changing and chemical analysis by FTIR of the thermally-treated teak wood. *Scientia Forestalis*, 44(112), 811-820.
- Li, M.-Y.; Cheng, S.-C.; Li, D.; Wang, S.-N.; Huang, A.-M.; and Sun, S.-Q. (2015). Structural characterization of steam-heat treated Tectona grandis wood analysed by FT-IR and 2D-IR correlation spectroscopy. *Chinese Chemical Letters*, 26(2), 221-225.
- Wang, X.; Pan, Z.; Shen, X.; and Liu, W. (2016). Stability and decomposition mechanism of ettringite in presence of ammonium sulfate solution. *Construction and Building Materials*, 124, 786-793.

- 35. Fortin, T.J.; Shilling, J.E.; and Tolbert, M.A. (2002). Infrared spectroscopic study of the low-temperature phase behaviour of ammonium sulfate. *Journal of Geophysical Research*, 107(D10), 1-9.
- 36. Chen, W.; Yu, H.; Liu, Y.; Chen, P.; Zhang, M.; and Hai, Y. (2011). Individualization of cellulose nanofibers from wood using high-intensity ultrasonication combined with chemical pretreatments. *Carbohydrate Polymers*, 83(4), 1804-1811.
- Lin, B.-J.; Colin, B.; Chen, W.-H.; Pétrissans, A.; Rousset, P.; and Pétrissans, M. (2018). Thermal degradation and compositional changes of wood treated in a semi-industrial scale reactor in vacuum. *Journal of Analytical and Applied Pyrolysis*, 130, 8-18.
- Durmaz, S.; Özgenç, Ö.; Boyaci, I.H.; Yildiz, U.C.; and Erisir, E. (2016). Examination of the chemical changes in spruce wood degraded by brown-rot fungi using FT-IR and FT-Raman spectroscopy. *Vibrational Spectroscopy*, 85, 202-207.
- Urruzola, I.; Andrés, M.A.; Serrano, L.; and Labidi, J. (2012). Adsorption of toluene onto bleached eucalyptus pulp treated with ultrasound. *BioResources*, 7(3), 4067-4084.
- 40. Cheng, D.; Gu, J.; Xu, B.; and Li, Y. (2015). Effect of (NH₄)₂ SO₄ Concentration on pyrolisis properties of rayon fiber from bamboo. *BioResources*, 10(4), 8352-8363.
- Zhu, Z.; Simister, R.; Bird, S.; McQueen-Mason, S.J.; Gomez, L.D.; and Macquarrie, D.J. (2015). Microwave assisted acid and alkali pretreatment of miscanthus biomass for biorefineries. *AIMS Bioengineering*, 2(4), 449-468.
- 42. Baillères, H.; and Durand, P.Y. (2000). Non-destructive techniques for wood quality assessment of plantation-grown teak. *Bois et Forets Des Tropiques*, Reference 16, 2000(263), 17-29.
- 43. Nurmi, A.; Hakkarainen, T.; and Kevarinmäki, A. (2010). This is how to cite an internet reference. Retrieved June 4, 2018, from https://www.vtt.fi/inf/pdf/workingpapers/2010/W146.pdf.
- 44. Kučerová, V.; Lagaňa, R.; Výbohová, E.; and Hýrošová, T. (2016). The effect of chemical changes during heat treatment on the colour and mechanical properties of fir wood. *BioResources*, 11(4), 9079-9094.
- 45. Hermoso, E.; and Vega, A. (2016). Effect of microwave treatment on the impregnability and mechanical properties of Eucalyptus globulus wood. *Maderas. Ciencia Y Tecnología*, 18(1), 55-64.
- 46. ISO 4589 (1996). The International Organization for Standardization. *Plastics-determination of burning behaviour by oxygen index: Part 1: Guidance.* New York.
- 47. Wu, G.; Lang, Q.; Chen, H.; and Pu, J. (2012). Physical and chemical performance of eucalyptus wood with impregnated chemicals. *BioResources*, 7(1), 816-826.
- 48. Tomak, E.D.; and Cavdar, A.D. (2013). Limited oxygen index levels of impregnated scots pine wood. *Thermochimica Acta*, 573, 181-185.
- 49. Qu, H.; Wu, W.; Wu, H.; Xie, J.; and Xu, J. (2011). Study on the effects of flame retardants on the thermal decomposition of wood by TG-MS. *Journal of Thermal Analysis and Calorimetry*, 103(3), 935-942.