# EFFECT OF OLEORESIN HEAT TREATMENT ON MECHANICAL AND PHYSICAL PROPERTIES OF THE BAMBOO *Thyrsostachys siamensis*

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**SUMMARY**
In this study, *Thyrsostachys siamensis* was treated oleoresin heat under different temperatures (120°C to 140°C) and durations (60 min to 120 min). The oleoresin used in this study is extracted from *Dipterocarpus alatus*. Response surface methodology (RSM) models for treatment temperature and treatment duration of oleoresin heat treated *T. siamensis* were developed. *T. siamensis* samples were treated oleoresin heat with different treatment temperatures (116°C to 145°C) and times (48 min to 133 min). The water absorption (WA), thickness swelling (TS), anti-swelling efficiency (ASE), modulus of rupture (MOR) and modulus of elasticity (MOE) of the treated samples were determined. The results are demonstrated that the dimensional stability of *T. siamensis* was enhanced by oleoresin heat treatment and improved along with increasing temperature. However, there was a reduction in strength properties as shown by decreased MOR and MOE. The proper temperature and time applied at 140°C for 60 min resulting the WA 28.65%, TS 3.23%, MOR 160.26 MPa and MOE 5.07 GPa. Better dimensional stability could be observed in the samples treated at higher temperature even when exposed to a shorter time. Applying an optimal treatment condition of the temperature 140°C for 120 min, the ASE achieve a high value of 50.8%.
**Keywords: heat treated bamboo, mechanical property, oleoresin, physical property.**

## 1. INTRODUCTION

Bamboo is one of the important vegetative resources after plantation wood and is a major raw material for the forest product industry. In Vietnam, bamboo has become the main material for industrial manufacturing of round and laminated bamboo furniture and parquet. The bamboo *Thyrsostachys siamensis* with its Vietnamese name “Tam Vong” is one of the most common species growing mainly as a forest and also largely cultivated in the provinces Binh Thuan, Gia Lai, Kong Tum, Lam Dong and Tay Ninh. The culms are the main raw material of many bamboo companies in South Vietnam for furniture for exportation.

Bamboo has low natural durability against fungi and insects compared with wood. Heat treatment is one of the treatments to improve the durability and dimensional stability of bamboo. Several studies of the heat treatment of wood and bamboo have been carried out in Asia, Europe (Jones *et al.* 2019) and America. In Europe, the heat treatment process has long been used for timber treatment and in Asia it has been used for bamboo and rattan. These methods were commercialized in response to the increased environmental awareness that drives the industry towards reducing the use of chemicals. Oil heat treatment is another alternative way in treating bamboo without use of preservatives. This process is considered as eco-friendly treatment. Thermal wood/bamboo treatment involves temperatures of 100 - 300°C (Jones *et al.* 2019). Investigations on bamboo treated with heat oil of Leithoff and Peek (2001), Manalo and Acda (2009), Salim *et al.* (2010) and Hao *et al.* (2021) showed that the temperature was effective to improve durability of bamboo and dimensional stability of bamboo.

Natural resin extracted from dipterocarp is considered one of the most important non-timber forest products of forest-adjacent communities in Southeast Asia (Evans *et al.* 2003, Baird 2009, Luu and Pinto 2007, Baird 2010, Lægaard 2010). The resin from dipterocarps can be classified into two categories: oleoresin (liquid resin) and solid resin. Liquid resin is extracted mostly from *Dipterocarpus* species in Laos, Thailand, Vietnam and Cambodia, with *D. alatus* being the main source (Luu and Pinto 2007). In Vietnam liquid resin is collected mostly from *D. alatus* (Luu and Pinto 2007). Resin is used domestically for lighting and sealing boats, and commercially for paints, varnishes and perfume fixatives (Dao 2004, Evans *et al.* 2003, Orwa *et al.* 2009).

In this investigation, *T. siamensis* was treated oleoresin (from *D. alatus*) heat under different temperatures and durations. The changes in physical and mechanical properties of bamboo culms after oleoresin heat treatment were studied to prove the feasibility of the thermal modification of bamboo culms using *D. alatus* oleoresin.

## 2. RESEARCH METHODOLOGY

### 2.1. Response Surface Methodology (RSM) and Central Composite Design

Central composite design (CCD) using RSM was used in the present study to investigate the effects of treatment variables on the physical and mechanical properties of wood and bamboo. Two independent variables, namely, treatment temperature (°C) and treatment time (min) were selected and the response variables were water absorption (WA), thickness swelling (TS), anti-swelling efficiency (ASE), modulus of rupture (MOR) and modulus of elasticity (MOE). The CCD was conducted using JMP 10. A 9-run CCD using RSM was developed and the ranges of the variables are shown in Table 1. Each of the independent variables was coded by five different levels as shown in Table 1, where the treatment temperature and treatment times ranged from 116°C to 145°C and 48 to 133 min, respectively.

**Table 1. Range and level of the variables**

| Range and level of actual and coded values | Temperature T (°C) $X_1$ | Time t (min) $X_2$ |
|---|---|---|
| $+\alpha$ | 145 | 133 |
| +1 | 140 | 120 |
| 0 | 130 | 90 |
| -1 | 120 | 60 |
| $\alpha$ | 116 | 48 |

### 2.2. Bamboo samples

Mature 3-year-old bamboo culms of *T. siamensis* were collected from a factory of Bamboo Nature Company in Binh Duong Province. From bamboo culms, samples of about 260 mm length were taken between the internodes. All samples with average moisture content of 65% were used for heat treatment within three days of collection. The bamboo samples were divided into 9 treatment experiments in addition to one group set as control (untreated) samples. Five replicates of each experiment were carried out and the average results were computed and analysed.

**2.3. Oleoresin heat treatment**

The bamboo samples were treated in hot oleoresin using a fabricated oil curing apparatus. The apparatus consisted of stainless-steel cylindrical vessel with 300 mm diameter and 400 mm length heated by electric plates connected to a thermocouple and digital temperature controller. Oleoresin extracted from *D. alatus* with viscosity 2.3 cps obtained from the Department of Chemical Engineering and Processing, Nong Lam University of HCMC.

Bamboo samples were completely submerged in the heated oleoresin. The treatment schedules were investigated with different temperature (120°C, 130°C and 140°C) for various time (60 min, 90 min and 120 min) as suggested by RSM modes (Table 1).

Untreated bamboo samples conditioned for eight weeks at 21°C and 65% relative humidity were used as controls.

**2.4. Determining the physical and mechanical properties**

The treated bamboo samples and controls were dried in the oven at 103 ± 2°C for 18 h prior to dimensional stability evaluation. Water absorption (WA) and thickness swelling (TS) of treated samples were tested based on 24 h submersion in distilled water. The weights and thickness before and after the soaking process were determined. Subsequently, the anti-swelling efficiency (ASE) was calculated.

The treated bamboo samples and controls were conditioned at ambient temperature and 65% relative equilibrium moisture content. The mechanical properties were tested based on ASTM D 143-94 (ASTM 1995).

The specimens of 250 × 27 × 11 mm in size for MOR and MOE testing and the specimens of 27 × 27 × 11 mm for WA and TS were applied. Three replications for each run were done.

**3. RESULTS AND DISCUSSION**

**3.1 Effect of the treatment temperature and the treatment time on physical properties of bamboo treated with heat oleoresin**

The results of water the absorption (WA), the thickness swelling (TS) and the anti-swelling efficiency (ASE) of bamboo samples treated are presented in Table 2. The bamboo *T. siamensis* treated with heat oleoresin are improve the dimensional stability.

Oleoresin heat treatment from 116 to 145°C resulted in decreased WA for *T. siamensis* showed reduction in WA of 37% to 65.4%, respectively, with respect to untreated control (Table 2, Fig. 1 and 3). Treatment temperatures from 116 to 145°C had a highly significant effect on WA ($p < 0.001$). However, effect of treatment duration from 48 to 133 min on WA were not significant ($p > 0.05$). Interaction between temperature and duration of treatment was also not significant ($p > 0.05$).

Improvement in dimensional stability, as indicated by reduction in TS as well as increasing in ASE (Table 2, Fig. 1, 2, 4 and 5). When the *T. siamensis* samples were treated at 145°C for 90 min, the lowest TS of 1.6% were noted. Conversely, the highest TS of 6.5% was obtained with the samples treated at 116°C for 90 min. Temperature had significant effect on TS ($p < 0.001$) while duration only had moderate or little effect ($p < 0.05$). However, no significant and obvious interaction were evident ($p > 0.05$). The treated samples of *T. siamensis* indicated ASE ranging from 32.5 to 52% which was a significant improvement in dimensional stability. Effect of temperature on ASE was remarkable ($p < 0.001$) while treatment time only had noticeable or little effect ($p < 0.05$). Additionally, no considerable and obvious interaction were evident ($p > 0.05$). Similar improvements in dimensional stability were reported in oil heat treatment of wood (Rapp and Sailer 2001, Salim *et al.* 2010, Wang and Cooper 2005) and oil heat treatment of bamboo (Hao *et al.* 2021, Manalo and Acda 2009, Leithoff and Peek 2001).

**Table 2. WA, TS and ASE of oleoresin heat treated samples and untreated samples**

| Run | Temperature T (°C) $X_1$ | Time t (min) $X_2$ | Water absorption WA (%) | Thickness swelling TS (%) | Anti-swelling efficiency ASE (%) |
|---|---|---|---|---|---|
| 1 | 116 | 90 | 43.0 | 6.5 | 32.5 |
| 2 | 120 | 60 | 40.7 | 6.2 | 34.3 |
| 3 | 120 | 120 | 39.5 | 5.5 | 37.0 |
| 4 | 130 | 48 | 36.0 | 5.5 | 40.1 |
| 5 | 130 | 90 | 35.5 | 4.9 | 42.6 |
| 6 | 130 | 133 | 34.3 | 3.9 | 45.3 |
| 7 | 140 | 60 | 28.3 | 3.0 | 49.6 |
| 8 | 140 | 120 | 25.3 | 1.9 | 51.1 |
| 9 | 145 | 90 | 23.6 | 1.6 | 52.0 |
| Control | - | - | 68.2 | 11.4 | - |

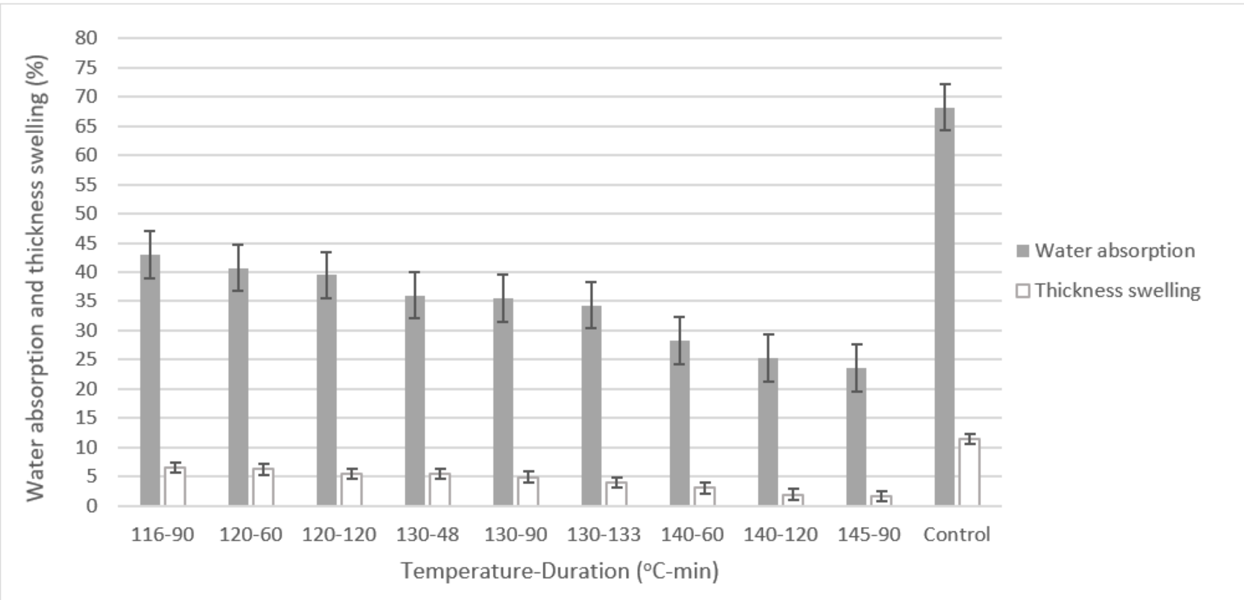


**Fig. 1. WA and TS of oleoresin heat treated samples and untreated samples**

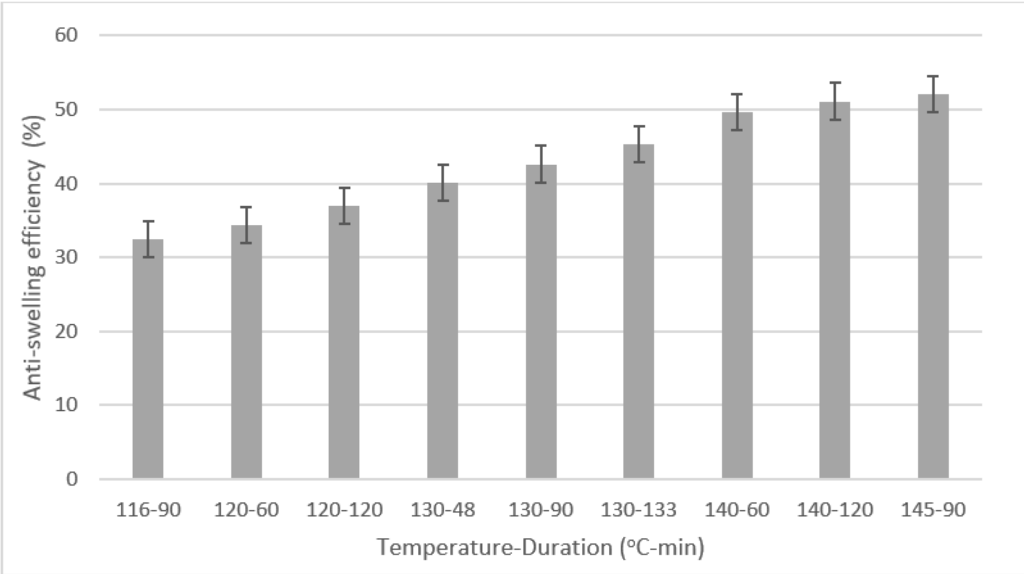


**Fig. 2. ASE of oleoresin heat treated samples**

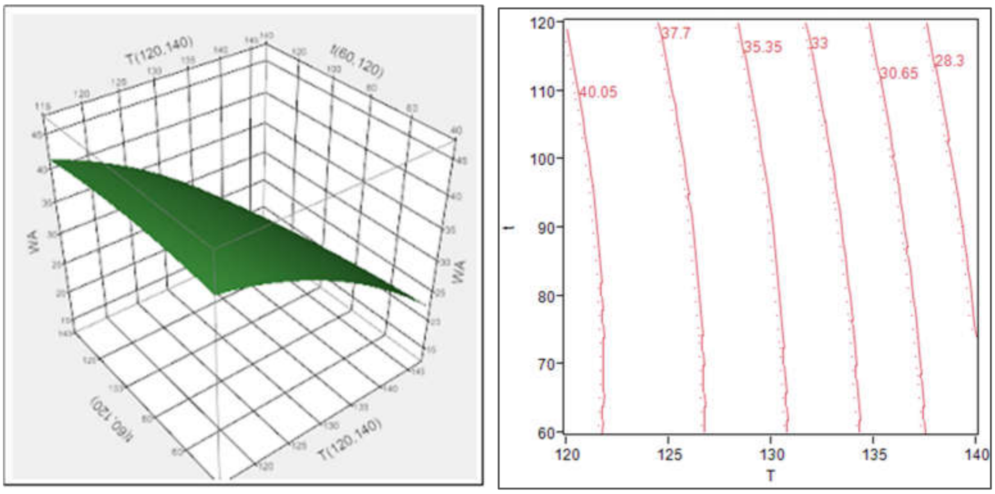


**Fig. 3. The 3D-surface plot of WA as function of time and temperature**

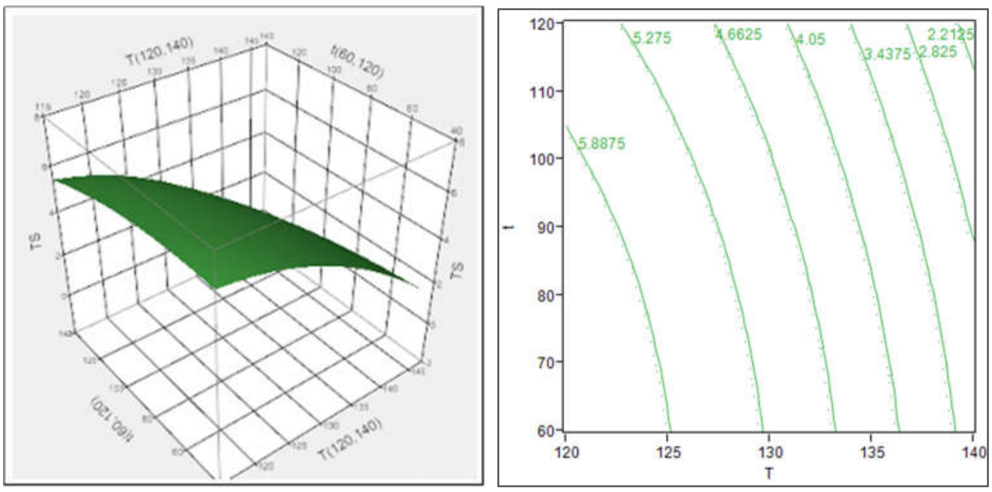


**Fig. 4. The 3D-surface plot of TS as function of time and temperature**

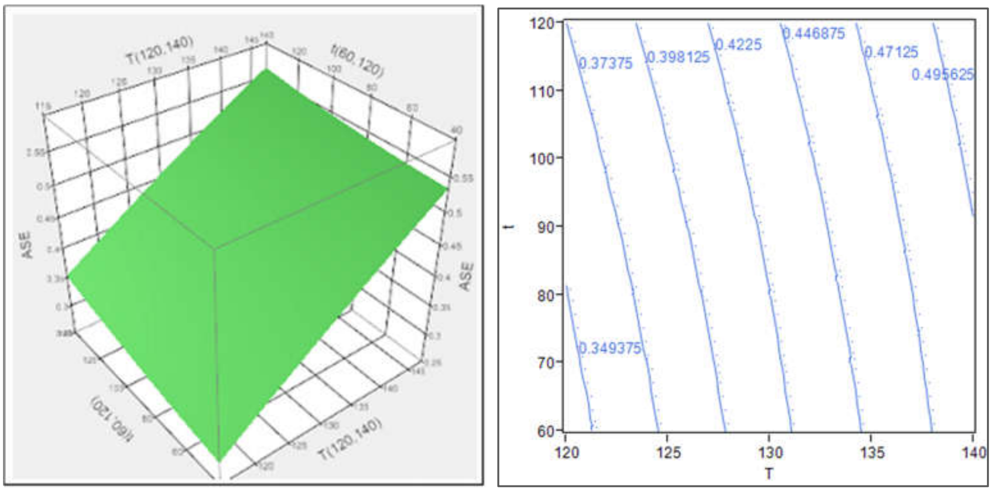


**Fig. 5. The 3D-surface plot of ASE as function of time and temperature**

### 3.2. Effect of the treatment temperature and the treatment time on mechanical properties of bamboo treated with heat oleoresin

The mechanical properties of treated bamboo were also affected by the temperature and duration of oleoresin heat treatment. The modulus of rupture (MOR) and modulus of elasticity (MOE) of bamboo samples before and after oleoresin heat treatment at different times and temperatures is shown in Table 3, Fig. 6, 7, 8 and 9. MOR showed a marked reduction of 9.8 - 25.4% with respect to untreated sample at 145°C (Fig. 6 and 8). Increasing temperatures from 116 to 145°C had a significant effect on MOR ($p < 0.001$). However, duration of treatment had little or no effect in MOR ($p > 0.05$). MOE values for *T. siamensis* showed a 20.5 - 34.5% reduction in stiffness with increasing treatment temperature compared with untreated sample (Fig. 7 and 9). Duration of treatment from 48 to 133 min have noticeable effect ($p < 0.05$) while its interaction with temperature seemed to have little or no effects on MOE ($p > 0.05$). Considerably, results of MOR and MOE in this investigation are similar to the previous studies of Wahab *et al.* (2004, 2005), Hao *et al.* (2021), Manalo and Acda (2009), Leithoff and Peek (2001).

**Table 3. MOR and MOE of the oleoresin heat treated samples and untreated samples**

| Run | Temperature T (°C) $X_1$ | Time t (min) $X_2$ | Modulus of rupture MOR (MPa) | Modulus of elasticity MOE (GPa) |
|---|---|---|---|---|
| 1 | 116 | 90 | 185.3 | 5.7 |
| 2 | 120 | 60 | 182.2 | 5.6 |
| 3 | 120 | 120 | 175.6 | 5.5 |
| 4 | 130 | 48 | 169.9 | 5.4 |
| 5 | 130 | 90 | 166.7 | 5.2 |
| 6 | 130 | 133 | 164.4 | 5.1 |
| 7 | 140 | 60 | 161.6 | 5.1 |
| 8 | 140 | 120 | 155.9 | 4.8 |
| 9 | 145 | 90 | 153.3 | 4.7 |
| Control | - | - | 205.5 | 7.2 |

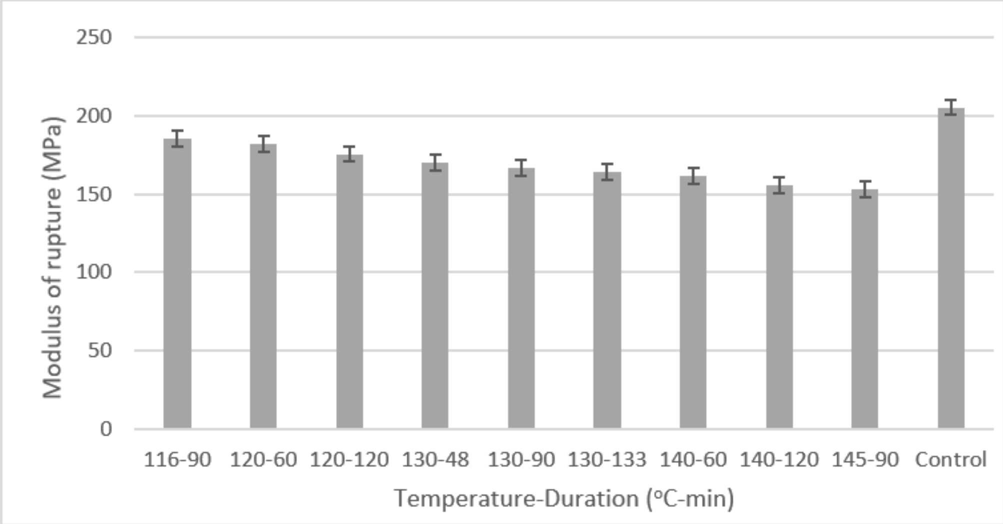


**Fig. 6. MOR of the oleoresin heat treated samples and the untreated sample**

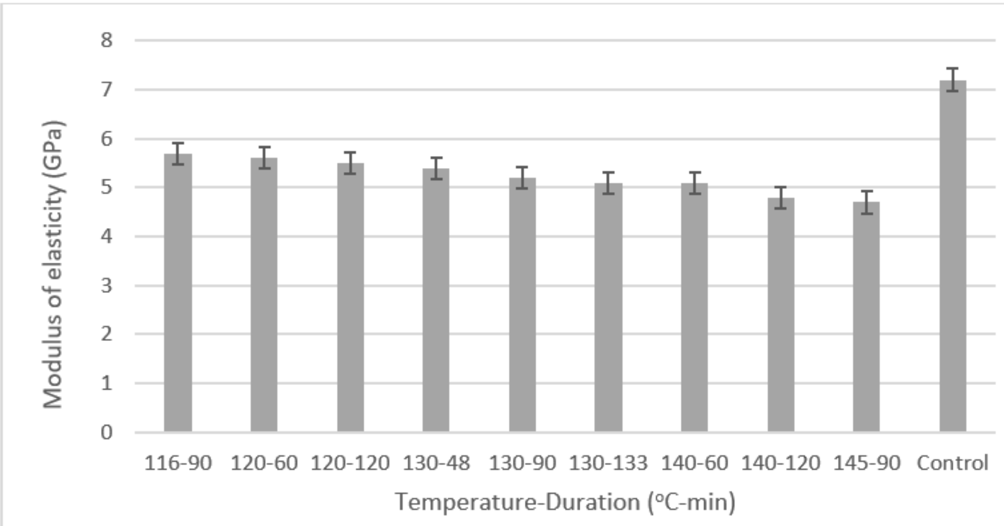


Fig. 7. MOE of the oleoresin heat treated samples and the untreated samples

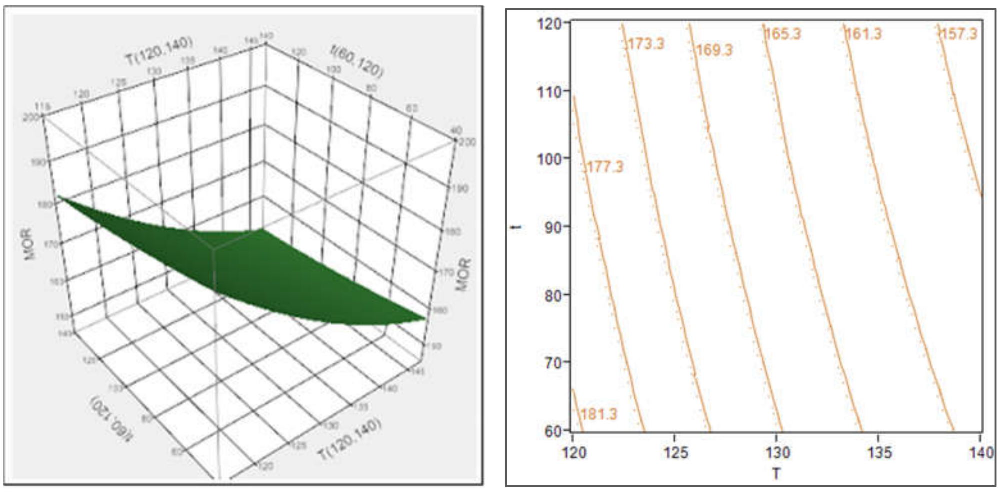


**Fig. 8. The 3D-surface plot of MOR as function of time and temperature**

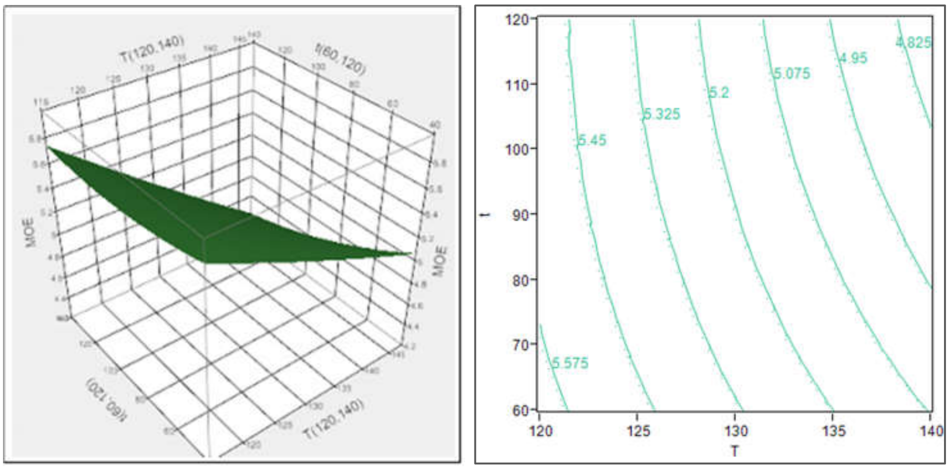


**Fig. 9. The 3D-surface plot of MOE as function of time and temperature**

### 3.3. Regression and adequacy of the model and optimal condition

***Regression***

To ensure the fitted model gave a sufficient approximation of the results obtained in the experimental conditions, the adequacy of the model was evaluated. The fit of the model was evaluated using coefficient of multiple regression ($r^2$) and adjusted $r^2$ was used for confirmation of the model adequacy. Based on the analysis, $r^2$ values of 0.9935, 0.9924, 0.993, 0.9945 and 0.9948 for the WA, TS, ASE, MOR and MOE respectively, indicated high fitness of the model. Describing the functional relation of the independent variables (T: treatment temperature and t: treatment time) and the response variable using regression analysis obtain five models. The final equations in terms of actual factors are shown below:

WA (%) = 121.34 – 0.66T; TS (%) = 28.12 – 0.17T – 0.02t; ASE (%) = – 0.53 + 0.007T + 0.0005t

MOR (MPa) = 312.73 – 1.06T – 0.08t; MOE (GPa) = 9.72 – 0.03T – 0.003t

***Optimal values of oleoresin heat treatment parameters***

The optimal condition was computed by the RSM, resulting shown in Fig. 10. The optimal condition is the treatment temperature 140ºC for the treatment time 60 min, the adequate values of WA, TS, ASE, MOR and MOE are 28.65%, 3.23%, 48.5%, 160.26 MPa and 5.07 GPa, respectively.

To maximize ASE, the treatment is applied at 140ºC for 120 min, resulting the high value of ASE is 50.8% (Fig. 11).

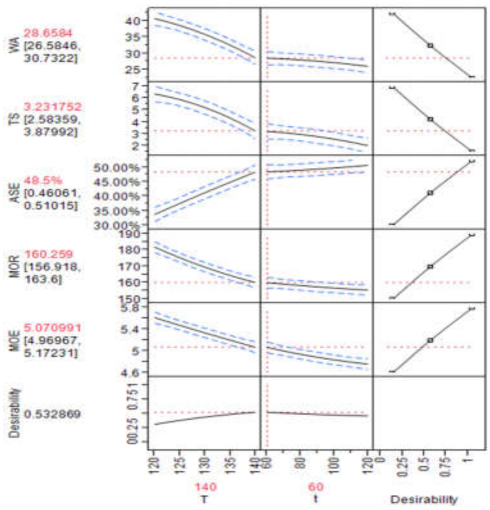


**Fig. 10. The cross-sectional surface meets the optimum point for the adequate values of**

**WA, TS, ASE, MOR and MOE**

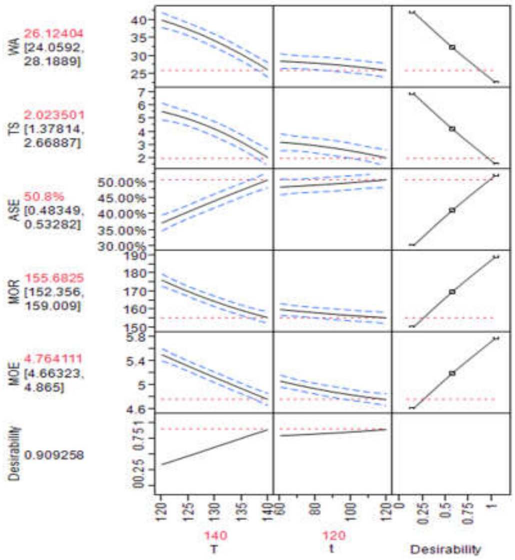

**Fig. 11. The cross-sectional surface meets the optimum point for the highest value of ASE**

## 4. CONCLUSIONS

Results show that it is possible to implement the thermal modification of bamboo culms using *D. alatus* oleoresin. The dimensional stability of *T. siamensis* was improved by oleoresin heat treatment. However, the strength as MOR and MOE of the treated bamboo were decreased. The proper temperature and time applied at 140ºC for 60 min reaching the WA 28.65%, TS 3.23%, MOR 160.26 MPa and MOE 5.07 GPa. Better dimensional stability could be observed in the samples treated at higher temperature even conducting a shorter time. Obtaining with the optimal treatment condition of the temperature 140ºC for 120 min, the ASE achieve a high value of 50.8%.

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# ẢNH HƯỞNG CỦA CHẾ ĐỘ XỬ LÝ NHIỆT NHỰA DẦU TỰ NHIÊN ĐẾN TÍNH CHẤT CƠ LÝ CỦA TRE TẦM VÔNG *(Thyrsostachys siamensis)*

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## TÓM TẮT

Trong nghiên cứu này, tre Tầm vông *Thyrsostachys siamensis* được xử lý bằng nhiệt nhựa dầu tự nhiên với các điểm nhiệt độ (120ºC đến 140ºC) và thời gian (60 phút đến 120 phút) khác nhau. Nhựa dầu tự nhiên sử dụng trong nghiên cứu được chiết xuất từ cây dầu rái *Dipterocarpus alatus.* Sử dụng phương pháp bề mặt đáp ứng (RSM) để bố trí thí nghiệm với các thông số nghiên cứu nhiệt độ (116ºC đến 145ºC) và thời gian (48 phút đến 133 phút). Kết quả cho thấy sự ổn định kích thước của tre Tầm vông *T. siamensis* được cải thiện rõ rệt qua xử lý nhiệt nhựa dầu với áp dụng chế độ nhiệt cao. Tuy nhiên, độ bền cơ học về cường độ uốn tỉnh (MOR) và modul đàn hồi (MOE) của tre xử lý nhiệt nhựa dầu rái bị giảm. Nhiệt độ và thời gian xử lý thích hợp ở 140ºC và 60 phút đạt được độ hút nước (WA) 28,65%, độ trương nở (TS) 3,23%, MOR 160,26 MPa và MOE 5,07 GPa. Độ ổn định kích thước tốt hơn ở các mẫu được khi áp dụng nhiệt độ xử lý cao hơn ngay khi áp dụng với thời gian ngắn hơn. Chế độ xử lý tối ưu với nhiệt độ 140ºC và 120 phút sẽ đạt được hệ số chống trương nở (ASE) 50,8%.

**Từ khóa: nhựa dầu rái, tính chất cơ học, tính chất vật lý, tre xử lý nhiệt.**

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|---|---|
| **Received** | **: 19/10/2021** |
| **Revised** | **: 25/11/2021** |
| **Accepted** | **: 07/12/2021** |